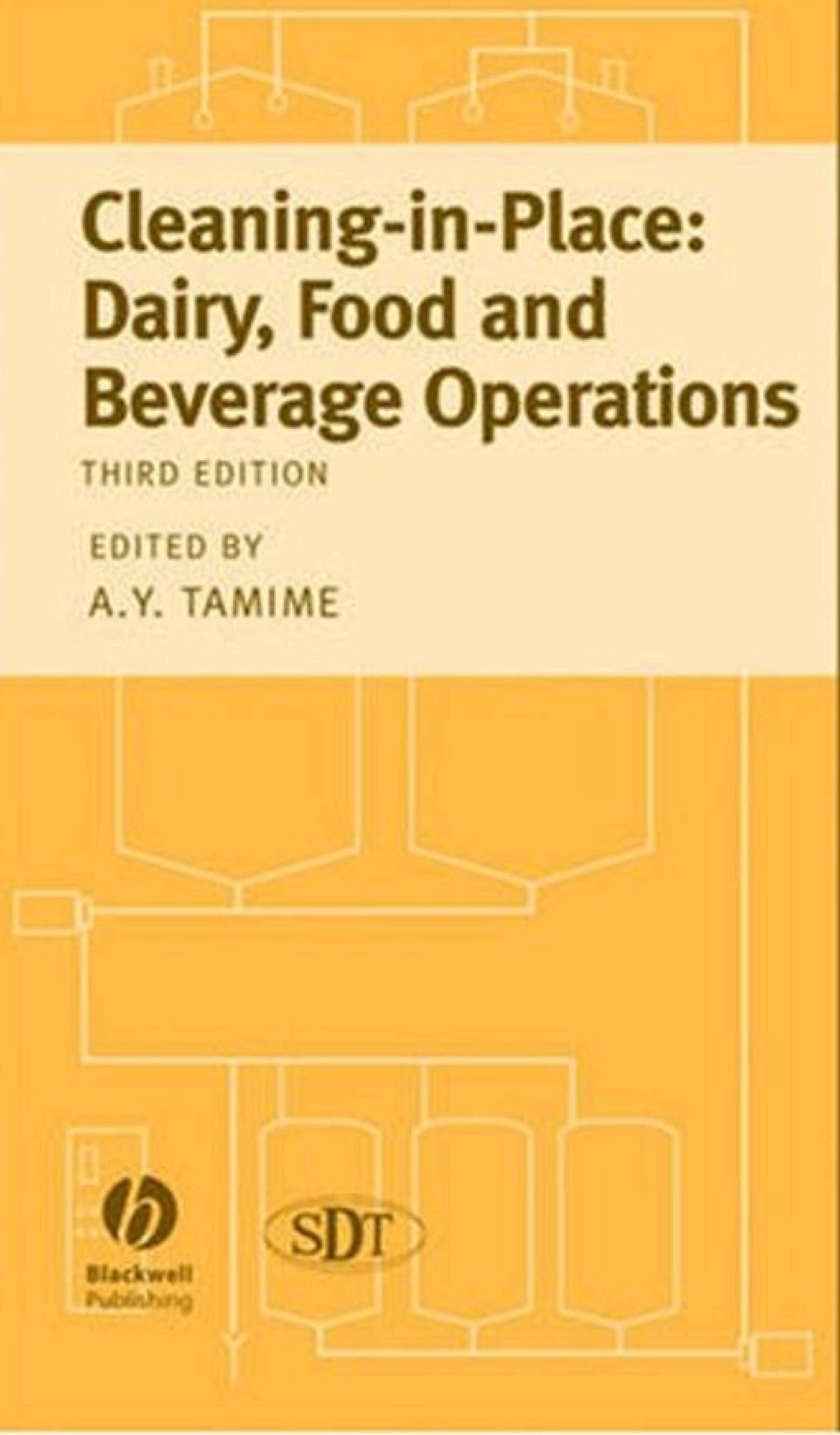

Cleaning-in-Place: Dairy, Food and Beverage Operations

THIRD EDITION

EDITED BY

A.Y. TAMIME

SOCIETY OF DAIRY TECHNOLOGY

Blackwell Publishing

SDT

Cleaning-in-Place

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Cleaning-in-Place

Dairy, Food and Beverage Operations

Third Edition

Edited by

Dr Adnan Tamime
Dairy Science and Technology Consultant
Ayr, UK

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Preface to Technical Series

For more than 60 years, the Society of Dairy Technology (SDT) has sought to provide education and training in the dairy field, disseminating knowledge and fostering personal development through symposia, conferences, residential courses, publications, and its journal, the *International Journal of Dairy Technology* (previously known as the *Journal of the Society of Dairy Technology*).

In recent years there have been significant advances in our understanding of milk systems, probably the most complex natural food available to man. Improvements in process technology have been accompanied by massive changes in the scale of many milk-processing operations, and the manufacture of a wide range of dairy and other related products.

The Society has now embarked on a project with Wiley-Blackwell to produce a Technical Series of dairy-related books to provide an invaluable source of information for practising dairy scientists and technologists, covering the range from small enterprises to modern large-scale operation. This fifth volume in the series, the third edition of *Cleaning-in-Place: Dairy, Food and Beverage Operations*, now under the editorship of Dr Adnan Tamime, provides a timely and comprehensive update on the principles and practice of the cleaning-in-place of process equipment. Thanks to the perishability of milk and many milk products, the dairy industry has been in the vanguard of the development of cleaning techniques and associated hygiene requirements. These are equally valid for other sectors of the food and bioprocessing industries, and this book will provide a valuable resource for food and dairy technologists.

Andrew Wilbey
Chairman of the Publications Committee, SDT

Preface to Third Edition

The first edition of this book was published in 1959 by the Society of Dairy Technology (SDT), and was entitled *Cleaning-In-Place (CIP) of Dairy Equipment*. An updated second edition, edited by A.J.D. Romney, was published in 1990.

Although the original title of the publication was orientated towards the dairy industry, the technical aspects of cleaning-in-place allow a broadening of the target audience towards readers concerned with food and beverage operations. The processed food industry has seen a major shift towards CIP over the past 10–15 years, and the beverage industry, which has been broadly in line with dairy industry technology, has seen increased demands from customers with regard to CIP verification and validation, and the attendant improvements in plant hygiene and related efficiency.

The book has been extensively revised and updated in this new edition. The two chapters on Chemistry of Detergents and Chemistry of Disinfectants have been combined into one chapter, and sections on Fluid Flow Dynamics and Laboratory Test Methods now appear as separate chapters. One new chapter on the subject of Membrane Cleaning has been added. This is a relatively new area and requires specialised cleaning products and procedures.

Authors have been selected from within the industry, allied suppliers and academia to provide a balanced and leading-edge assessment of the subject matter. Whilst the second edition has been a very popular publication, it is now rather outdated, and this revision is timely. The book will be a valuable addition to the SDT's Technical Series, offering the latest information on CIP to readers within the dairy, food and beverage processing industries internationally.

A.Y. Tamime

Preface to Second Edition

Following the highly successful rewriting of the Society's *Pasteurizing Plant Manual* in 1983, a need was identified to update the manual on *In-Place-Cleaning of Dairy Equipment*, published in 1959 and out of print for some years.

To this end, a decision was taken by the Council to reconstitute the Dairy Equipment and Standardisation Committee disbanded in 1974; this committee was re-formed in 1985 under the new title of the Dairy Equipment Advisory Committee (DEAC), part of its brief to progress this task.

A listing of the proposed chapter headings and possible authors was drawn up, and I was invited to take on the role of both coordinating and editing the new work.

To all those who have contributed to the text and provided the illustrations for this project I extend most hearty thanks, both on my own behalf and on that of the Society. My gratitude goes also to my good friend and mentor, Tom Ashton, both for the Foreword to this edition and for his guidance and support in the past.

It is the hope of the Council, of all the members of the DEAC and of myself that this work will prove of value, to dairy managers and quality assurance staff as well as to students entering our industry.

A.J.D. Romney
1990

Preface to First Edition

In 1953, the Society of Dairy Technology published the *Pasteurizing Plant Manual*. The success of that venture encouraged the Dairy Equipment and Standardisation Committee to consider what could be done further in this new field of the Society's activities. Once again the inspiration, and much of the preliminary work, came from the late J.R. Cuttell. In producing this book, the Drafting Committee has been guided by the inspiration and has endeavoured to achieve a result worthy of the original conception.

The text has been written by Dr T.R. Ashton, Mr G.H. Botham, Dr L.F.L. Clegg, Mr H.C. Cooper, the late Mr J.R. Cuttell, Mr H.S. Hall, Mr H.C. Hillman, Mr P.A. Lincoln, Dr R.J. MacWalter and Mr W.W. Ritchie assisted by their colleagues on the Drafting Committee, Mr T.A. Hole, Mr E.L. Jarvis, Mr J.R. Rowling, Mr W. Rushton and Mr G.E. Taylor. The task of editing has again been taken by Dr J.G. Davis.

The Drafting Committee wishes to acknowledge gratefully the substantial contributions to its work by Mr P. O'Niell, who has acted as Secretary, and Miss E.G. Dunworth, who has undertaken the typing and duplicating work. The Committee greatly appreciates the facilities provided by the National Dairymen's Association, in whose offices all the meetings have been held.

Illustration material has kindly been provided by the APV Co. Ltd, Clarke-Built Ltd, CP Equipment Ltd, Dairy Pipe Lines Ltd and Talbot Stead Tube Co. Ltd. Mr H.C. Cooper has designed the cleaning circuit illustrations.

It is the hope of the Drafting Committee that this book will serve as an introduction to what is a comparatively new subject and so pave the way to the wider use of modern techniques.

H.S. Hall
1959

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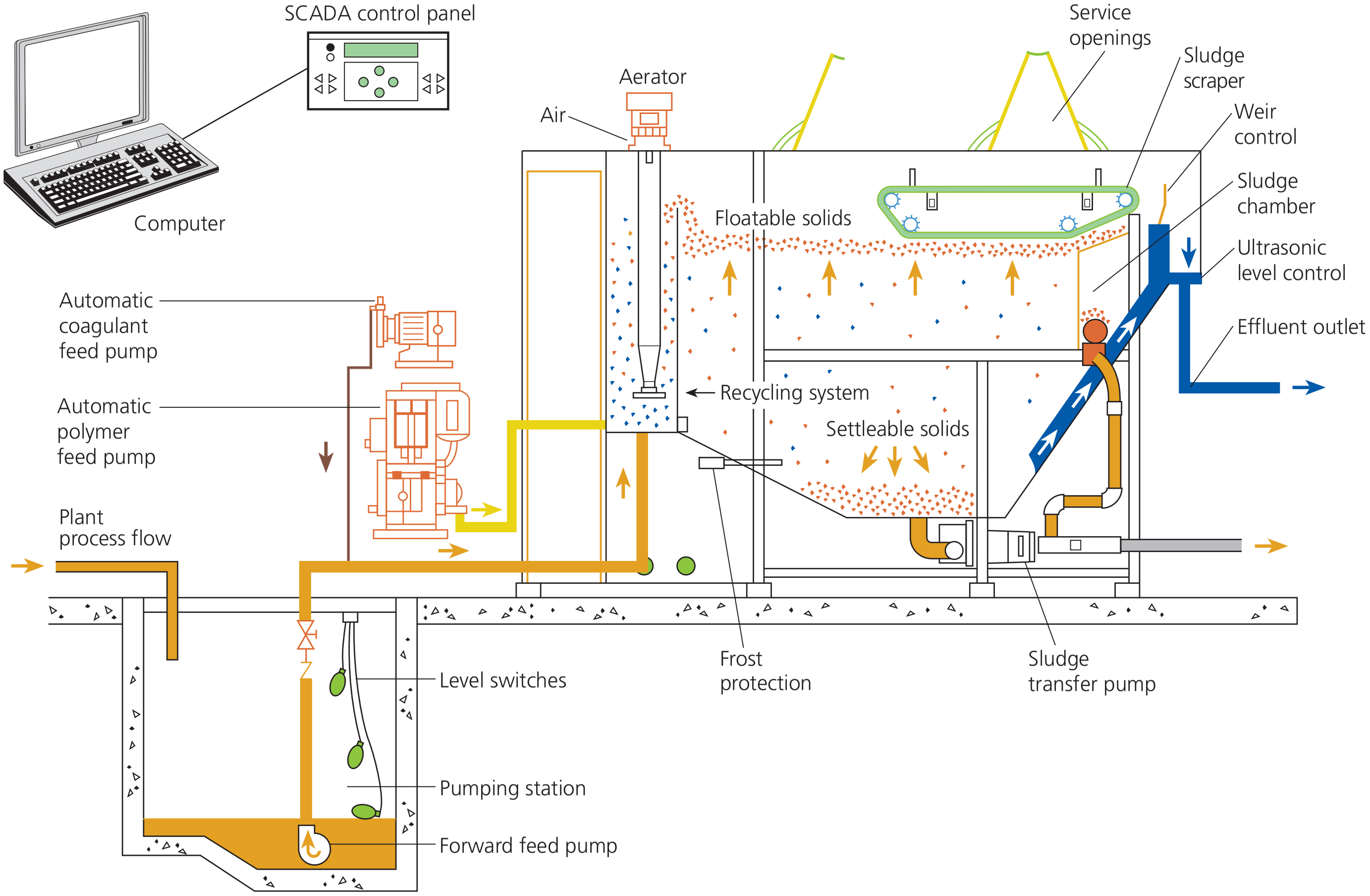

Plate 1 Cross-section of SCADA air filtration (SCAF®) unit for pretreatment of wastewater, showing how solids are separated. Reproduced with permission from Hydro International, Navan, Ireland.

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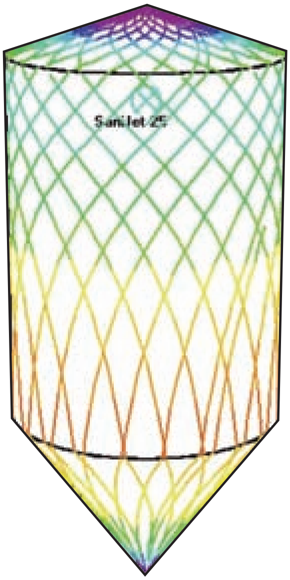
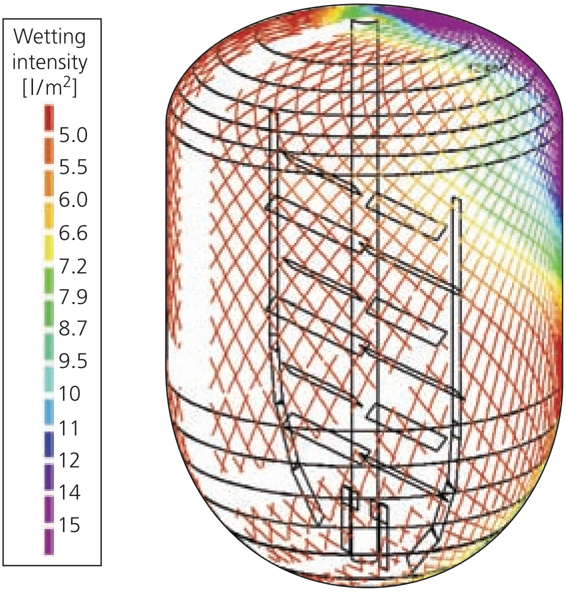


Plate 2 SaniJet 25 – pattern of cleaning liquid distribution build-up – first cycle. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

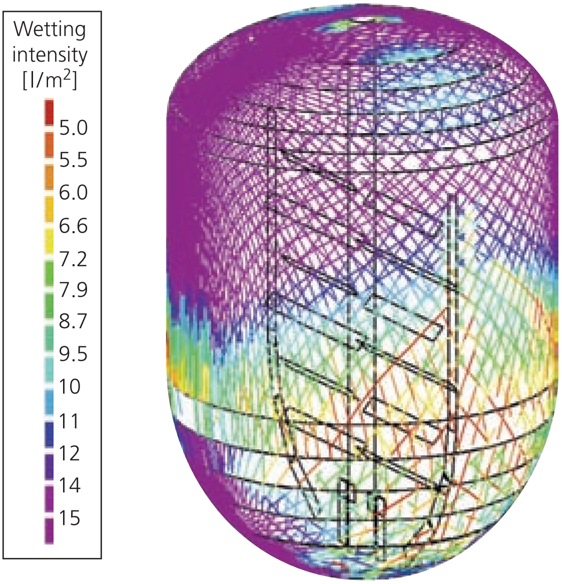
Plate 3 SaniJet 25 – full pattern of cleaning liquid distribution build-up. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.



Mixing vessel 1 × TZ-89 2 × 2.5 mm

Time = 3.4 min water used = 104 L

Plate 4 Illustration of shadows created by agitator with in-tank cleaning devices. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.



Mixing vessel 2 × TZ-89 2 × 2.5 mm

Time = 3.4 min water used = 207 L

Plate 5 Illustration of shadows from agitator eliminated with two cleaning devices. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

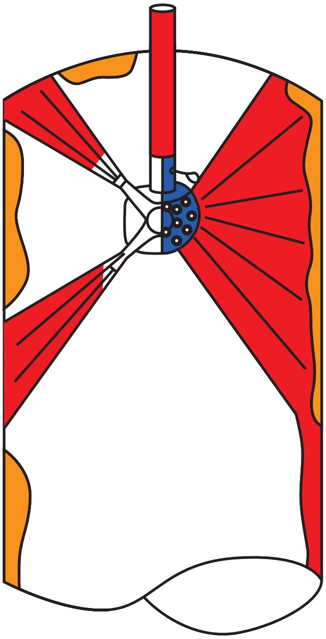


Plate 6 The flow behaviour of the spray heads of the rotating jets as influenced by pressure.

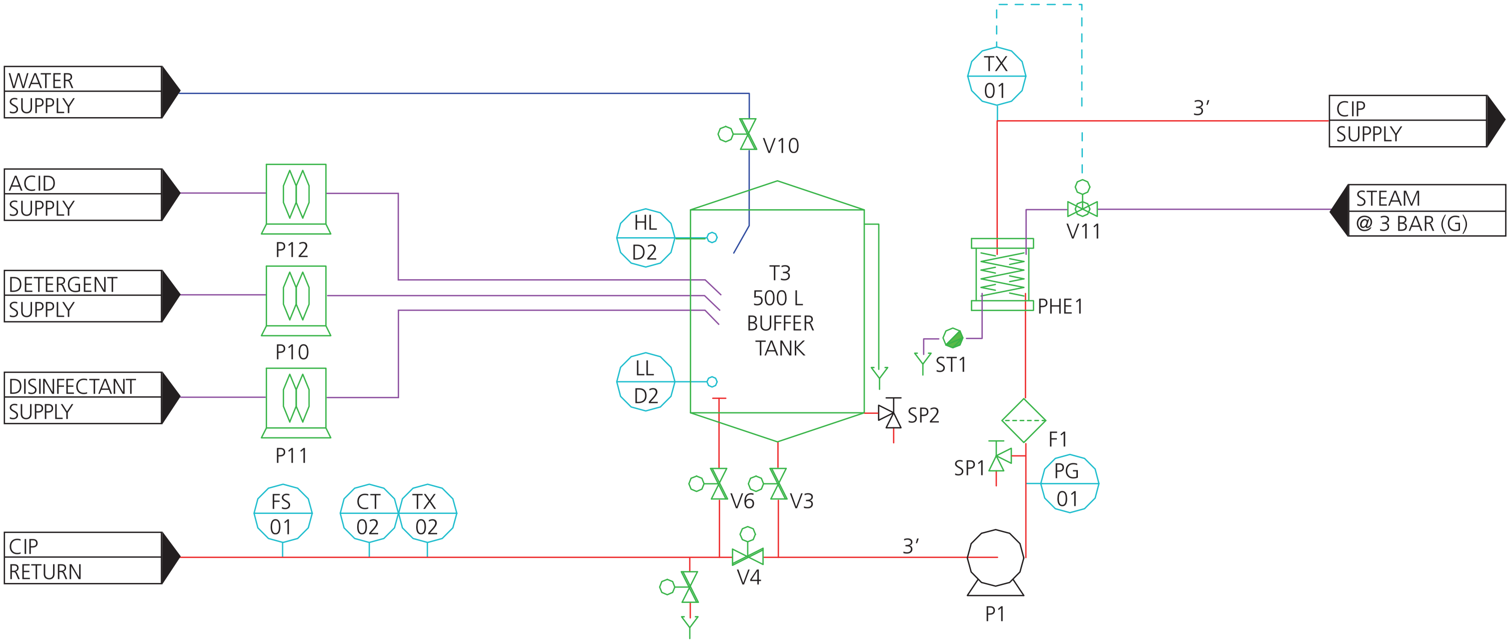


Plate 7 Single-use CIP system.

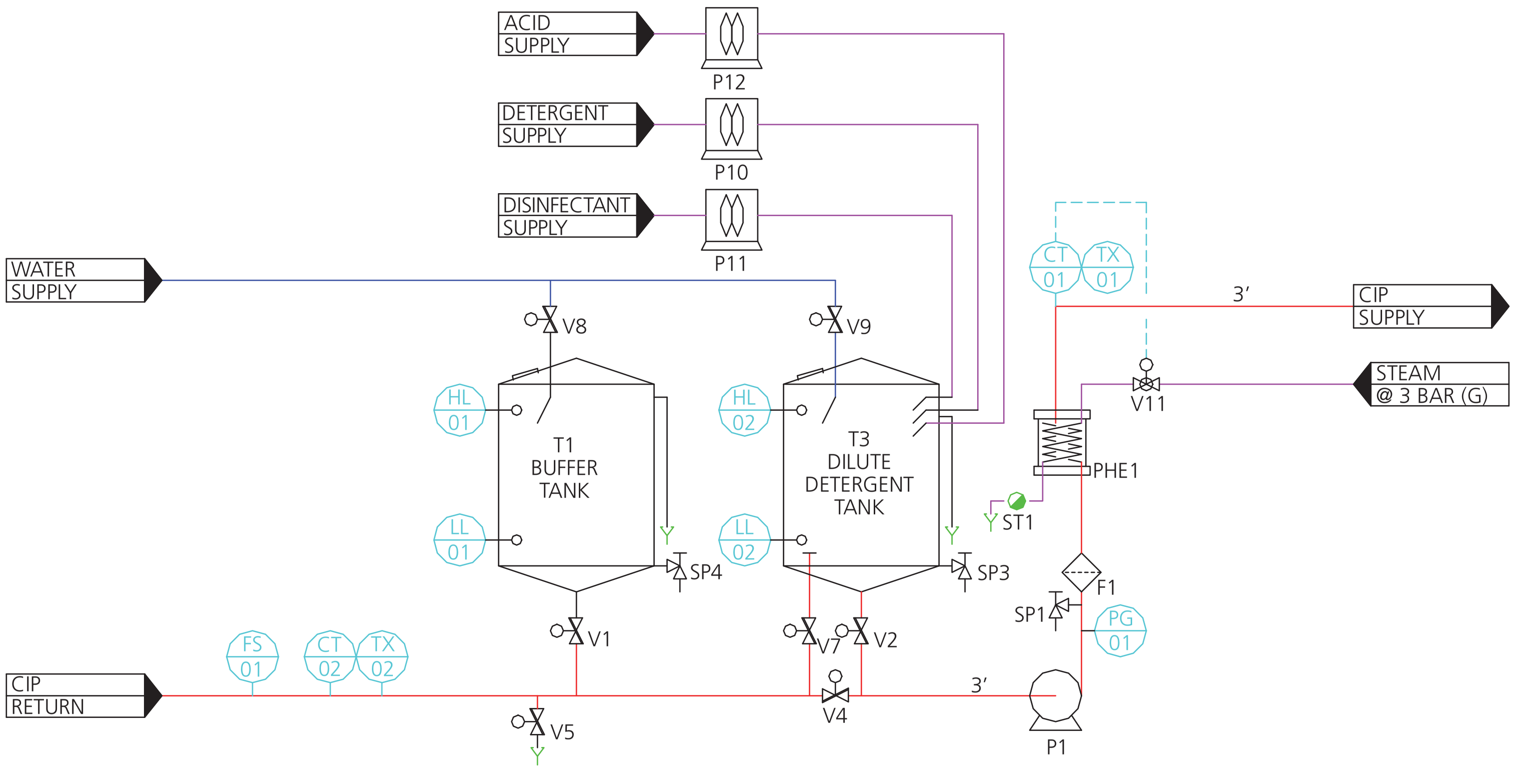


Plate 8 Partial recovery CIP system.

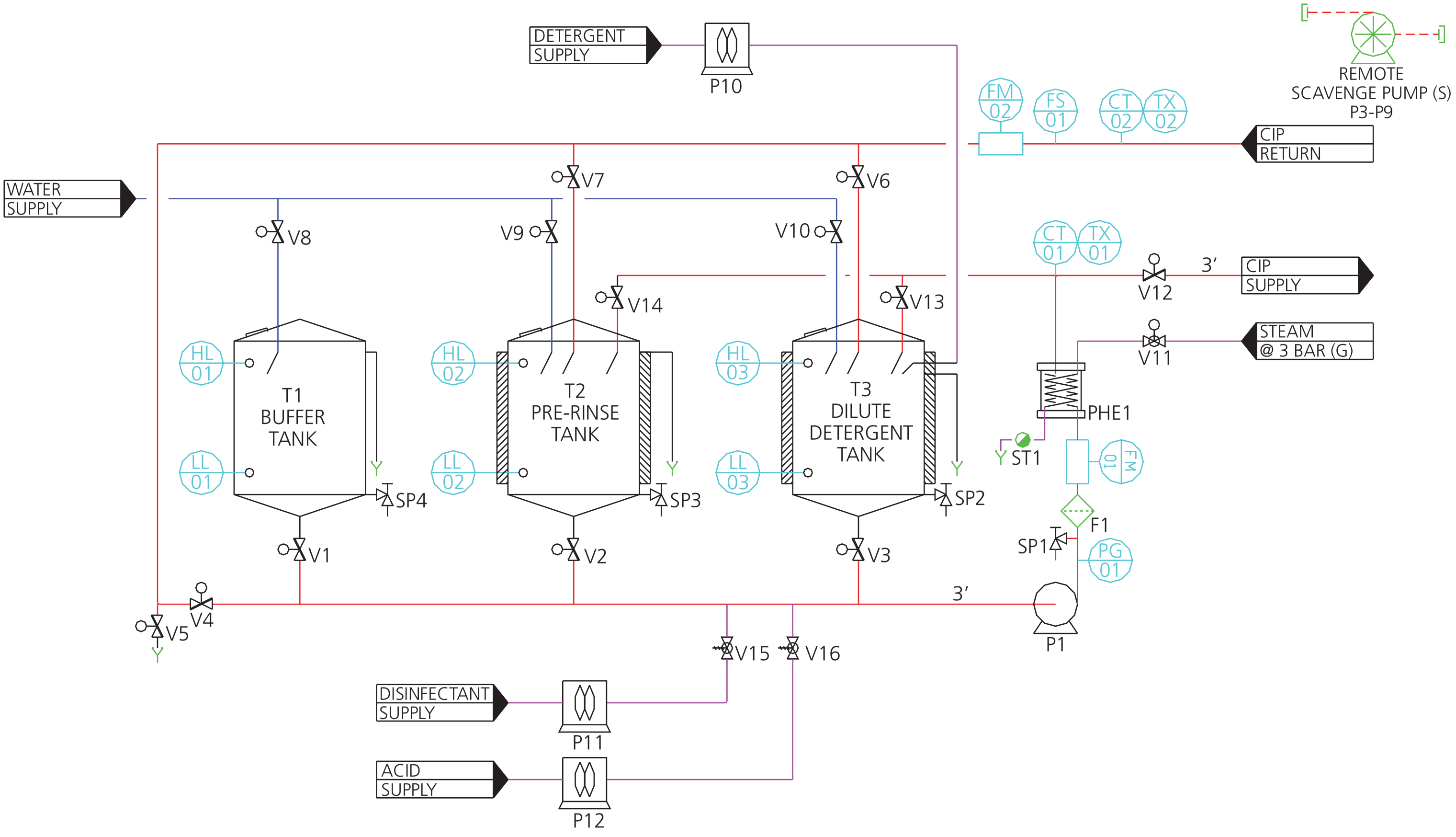


Plate 9 Full recovery CIP system.

1 Principles of Cleaning-in-Place (CIP)

M. Walton

1.1 Introduction

Cleaning-in-place (CIP) is now a commonplace activity in almost all dairy, beverage and processed-food production plants. The processed food industry has seen a major shift towards CIP over the past 10–15 years, and the beverage industry, which has been broadly in line with the dairy industry technology, has seen increased demands from customers in terms of CIP verification and validation to provide improvements in plant hygiene, finished product quality, and related shelf-life and microbiological considerations.

The highest standards of plant hygiene are an essential prerequisite for the production of any high-quality product being produced for human consumption. The cleaning and subsequent disinfection or sterilisation of any item of processing plant or equipment must be carried out with the utmost care and attention if the final product quality is to be fully assured. In earlier days, cleaning tended to be a manual process; indeed, it still is today in many small-scale operations, especially in the processed food sector, where a combination of manual strip-down clean and rebuild is common. Where manual cleaning is still practised, it is vital that there is meticulous attention to detail, because – for reasons of the health and safety of the operative – only mild and comparatively cool chemical solutions, detergents and disinfectants can be used, and strict adherence to cleaning procedures is critical. In larger-scale operations, and where more complex plant and equipment may be involved, the most usual approach today is to employ CIP, and it is to this aspect of cleaning technology that this book is primarily devoted, with a view to providing an understanding of the concepts and application of CIP in the processed food, pharmaceutical, dairy and beverage sectors.

1.2 Cleaning-in-place (CIP): definition

In the 1990 edition of the Society of Dairy Technology manual *CIP: Cleaning in Place*, CIP was defined as:

> *The cleaning of complete items of plant or pipeline circuits without dismantling or opening of the equipment and with little or no manual involvement on the part of the operator. The process involves the jetting or spraying of surfaces or circulation of cleaning solutions through the plant under conditions of increased turbulence and flow velocity.*

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This was taken from the National Dairyman's Association (NDA) Chemical Safety Code, which was published in 1985; although the NDA has been superseded, their definition of CIP is still felt to be quite appropriate.

1.3 CIP systems: hardware

CIP units comprise vessels for storage and recovery of cleaning solutions, along with valves, pumps, pipelines and field instrumentation to allow cleaning to take place, usually automatically. They vary in complexity and degree of automation, and hence their efficiency and cost-effectiveness are also variable. For example, the single-use CIP units tend to be very expensive to operate (detergent, water and energy requirements are high), but can be much more hygienic as the chance of cross-contamination and potential spore formation is greatly reduced. Full recovery systems with large detergent storage tanks are usually multifunctional and tend to be relatively economic in operation, but need to be closely monitored to prevent the build-up of soil residues in the dilute detergent or recovered rinse tanks due either to the inherent recovery efficiency of the set or perhaps to poor pre-rinsing. It is therefore very important to refresh cleaning solutions on a regular basis.

1.4 The processes of cleaning

The cleaning processes, whether manual or automated and throughout all industry sectors, tend to follow similar principles, and will usually consist of a series of discrete stages or cycles, generally including:

- removal of gross debris (product recovery)
- pre-rinse
- detergent recirculation
- intermediate rinse
- second detergent recirculation (optional)
- intermediate rinse
- disinfection
- final rinse

1.4.1 *Removal of gross debris (product recovery)*

In manual cleaning operations, this tends to refer to removal of any residual product by mechanical means prior to introduction of a water rinse. In CIP applications, removal of gross debris generally involves draining product from the system to be cleaned under gravity, or physically displacing the product using various media, such as compressed air, water or a mechanical pigging device. This stage is often incorporated into the pre-rinse cycle of the cleaning programme with the addition of a divert valve system to facilitate product recovery into a suitable vessel or direct routeing to drain. Control of this feature is quite often via automated valve and timer, but it is also possible to use more sophisticated methods, such as turbidity or conductivity sensors in the return line. It is important to include an override timer into these systems as a ‘failsafe’ in order to avoid filling a product recovery tank with pre-rinse water if the system fails to activate the divert valve: this is not an uncommon situation, with probe and controller maintenance being a critical aspect of successful operation. Product recovery systems are becoming more sophisticated with the introduction of membrane plants that are designed to remove high levels of water from the effluent stream – often termed ‘white water’ in the dairy sector – to allow the recovered solids to be sold on for re-processing: these plants are effective at reducing effluent loading, and can form part of site pollution prevention and control (PPC) systems (e.g. The Environmental Protection Act; Anonymous, 1990).

1.4.2 *Pre-rinse*

Pre-rinse cycles often utilise recovered ‘water’ from the intermediate rinse stage (see Section 1.4.4). This serves two purposes: first, to reduce total water consumption (and effluent generation); and second, to utilise any heat energy and possible residual detergent solution carried into the recovered rinse tank during the rinse recovery stage. It is not uncommon to find heated pre-rinse systems in certain applications, such as cream production, where the hot pre-rinse solution provides a greatly enhanced method of product residue removal. The pre-rinse stage is important because it is not desirable to introduce excessive soiling into the dilute detergent tank. This stage is generally controlled via a timer, sometimes split between product recovery and drain, and these timers are often set at excessive levels to ensure maximum product removal. However, this may not be cost-effective in circumstances where water and effluent costs are high. In general, the pre-rinse cycle for tanks, silos or vessels consists of several ‘burst’ or ‘pulsed’ rinses, as this both improves rinsing efficiency and can reduce water consumption significantly.

1.4.3 *Detergent circulation*

This is where the main task of cleaning takes place, resulting in the soil being lifted from the plant surface and held suspended or dissolved in the detergent solution; for the selection of suitable detergents see Section 1.5.5, but an important attribute of the detergent should be the ability to prevent any soil from being redeposited during recirculation. Recirculation timings need to be assessed by experimentation and a degree of experience, with timing generally varying from 15 min up to 1 h, where exceptionally large and complex circuits are being cleaned. Contact times can be reduced by offsetting the potentially reduced cleaning effectiveness with higher temperatures, higher concentrations, or the use of more sophisticated (and expensive) detergent formulations. Cycle timers are often set to start counting down once the temperature set point has been reached in the return leg: this can lead to excessive cleaning times if the efficiency of the heating system is inadequate. It is important, for example, to ensure that tanks incorporating a water-cooling jacket have the jacket drained prior to CIP. Depending on detergent formulation, foaming can sometimes be a problem, and it is often associated with product contamination. It can also be caused by many other factors, including air entrainment via leaking pump seals; the use of totally softened water supplies can also be a contributory factor. It is also possible to utilise an acidic detergent for the main cleaning step: this is quite common in both the dairy and beverage sectors, where milk residues in ‘cold/raw’ milk areas respond well to acidic detergents, and in the brewing sector, where acidic detergents have significant advantages over alkaline detergents in their ability to clean under CO_2 environments without loss of activity. Combined detergent/disinfectant chemical blends may be used in the cleaning cycle itself, though this approach has comparatively limited application, as they can be adversely affected by high soil loading, and the ratio of detergent to disinfectant can become imbalanced.

1.4.4 *Intermediate rinse*

The intermediate rinse serves to remove all traces of detergent and entrained soil from the plant being cleaned and, in a partial recovery situation, to recover as much detergent (and thermal energy) back to the dilute detergent tank as possible; it also may need to be sufficient to cool the plant down ready for disinfection and/or refilling. The intermediate rinse should use potable water, and is normally cold, although – if a warm secondary detergent step is being incorporated – it may be desirable to use hot water (if available from sources such as recovered and suitably treated condensate). The intermediate rinse is often recovered and reused as the pre-rinse for the next cleaning cycle.

1.4.5 *Second detergent circulation (optional)*

Some systems utilise a secondary detergent cycle, often an acidic detergent to follow an alkaline product in the first detergent stage. This is common practice where built detergents are not being used (sodium hydroxide liquor followed by nitric acid was once very common), and also where there are high levels of process-generated soils, such as in heat exchangers and cheese vats.

1.4.6 *Second intermediate rinse*

This second intermediate rinse will almost always use cold potable water. The quality of this water is critical, if there is to be no disinfection stage. Some sites that do not use a discrete disinfection stage in the CIP cycle ensure the quality of their potable water by treating it with chlorine dioxide.

1.4.7 *Disinfection*

The disinfection cycle is usually undertaken cold, and often uses an oxidising biocide, such as sodium hypochlorite or peracetic acid solution (equilibrium mixture of acetic acid and hydrogen peroxide). Some non-oxidising biocides are also available, but they must be low foaming and fast acting in cold water in order to be effective for CIP. It is also possible to use hot water at the disinfection stage rather than a chemical agent; this is also very effective, but requires a high thermal energy input, which can prove costly.

1.4.8 *Final rinse*

The final rinse stage should be undertaken using cold potable water. Again, the quality of this water is critical, as it can lead to post-disinfection contamination and product spoilage.

1.5 Planning a cleaning project

Above all else, the paramount consideration in the planning of any cleaning project must be safety – not only of the plant and personnel involved, but of the product which that plant is required to process. The mid-1980s saw a dramatic reappraisal of many of the standards and practices previously regarded as acceptable within the dairy industry, following incidents – both at home and overseas – of contamination of products by micro-organisms rarely ever encountered as presenting problems of any significance, other than in raw milk supplies, to the average United Kingdom dairyman. Problems of *Salmonella* spp., *Listeria* spp. and *Yersinia* spp. contamination in finished product have all played their part in accentuating the need for stringent food hazard assessment in every field of activity; cleaning technology is not least among these. The interconnection of 'raw' and 'processed' side plant and pipelines into a single cleaning circuit, or the separate cleaning of 'raw' and 'processed' side equipment from a common CIP set – frequently encountered in the days when the fashion was for large, multi-purpose, centralised cleaning systems – is now generally considered to present unacceptable product risks. The trend is now strongly towards the use of smaller units, specifically dedicated to either raw or finished products, or to the cleaning requirements of individual circuits and plant equipment items. The total separation of the 'raw' and 'processed' sides of a factory – the only point at which the two ever come together being the flow diversion valve of the processing plant – should be the basic design objective of every process engineer. This approach may, in some installations, carry a capital cost penalty, but the advantages in quality assurance and generally lower revenue operating costs weigh heavily on the benefit side. Such an approach need not, of course, preclude the use of a common centralised control system; the need for programme safety interlocks between the individual systems is vital to such an approach.

Before embarking on any cleaning project, however, a considerable number of questions have to be answered regarding the actual equipment to be cleaned and the standards of cleanliness to be achieved.

1.5.1 *What is the physical nature of the plant or equipment to be cleaned?*

Any food manufacturing or processing plant will comprise many different items of equipment: for example, dairies and breweries will have plate heat exchangers, storage tanks, vats, pumps, valves, and interconnecting pipework, as well as specialised items, such as bottle and carton fillers or – on the manufacturing side – cheese plant, evaporators, spray dryers and continuous butter-makers. Each of these will have its own cleaning requirements, and pose its own individual cleaning problems. Food processing plants are probably the most diverse sector in terms of equipment design and cleaning requirements, and full consideration needs to be given to the design of this equipment with respect to CIP. Materials of construction must be considered, not only regarding any metal parts, but also items such as gaskets and similar rubber components, and plastic mouldings, to ensure their compatibility with the cleaning chemicals proposed regarding corrosion or degradation. Questions as to temperature and pressure or vacuum limitations of the equipment must be considered, all aimed at answering the overriding question: 'Can the plant be cleaned safely and effectively by in-place methods, achieving acceptable standards of cleanliness without damage to the plant itself?'

1.5.2 *What standards of cleaning are required?*

It is important to understand that various degrees of cleanliness may be appropriate in different circumstances. It is vital that this is clearly recognised, and the target level of cleanliness defined when considering any cleaning project. Levels of cleaning that might be considered are as follows.

- *Physically clean*: This primarily addresses the aesthetic aspect. The surface appears clean, but chemical residues, often deliberately left to achieve a particular desired effect, may have been allowed to remain. Disinfection of the surface has not been considered.
- *Chemically clean*: The surface is rendered totally free from any trace of chemical residues.
- *Microbiologically clean*: This addresses the degree of microbiological contamination remaining on the surface, and may range from plant that has been 'disinfected' – that is, the number of bacteria on the surface of the equipment has been reduced to a level consistent with acceptable quality control and hygienic standards – to surfaces rendered totally sterile, as is essential in ultra-high-temperature (UHT) and similar aseptic operations.

One can thus reach a situation where the surface involved has been physically cleaned and has, perhaps, been rendered microbiologically clean by chemical disinfection, but traces of substantive disinfectant chemical have been deliberately left on the surface to reduce the risk of subsequent microbiological contamination, and the surface is therefore still chemically 'contaminated'.

1.5.3 *What is the nature of the soil to be removed?*

Soil can be considered as the product residues, scale and any other unwanted deposits of foreign matter that have to be removed from the plant surfaces during the cleaning process. Within the manufacturing or processing dairy, such soil may include fat, protein (both denatured and un-denatured: see IDF, 1997), sugar (possibly caramelised), minerals (both from product and from the water supply), fruit cells and various manufacturing ingredients including gums, starches, stabilisers and emulsifiers – all of which will present different and often complex cleaning problems to the detergent chemist. In the dairy context, soil can be divided into two broad general headings: organic soil, which is mainly of plant or animal origin, and is generally most susceptible to attack by alkaline detergents; and inorganic soil, which is mainly of mineral origin, and is usually most effectively attacked by acidic detergents.

Most soils are, however, a combination of both organic and inorganic deposits. 'Milkstone', for example, is primarily a combination of calcium caseinate and calcium phosphate. The degree of heat denaturation of the soil can also dramatically change its physical condition, and call for widely differing cleaning techniques and chemicals.

1.5.4 *When is the cleaning to be undertaken?*

Within any processing or manufacturing site, there will be a wide variety of plant and equipment, some of which may become available or may have to be cleaned during the day while other processing is still under way. Other plant may not be available or accessible for cleaning until the day's production run has been completed. Where any cleaning operations are undertaken during the production day, it is vital that all other plant and product are totally safeguarded from contamination. This is usually addressed by mechanical safety breaks in pipework. Swinging bend systems provide a physical break between CIP circuits and production pipe runs; they can be fitted with proximity switches to help ensure that the loop has been installed in the correct position prior to CIP, and can provide an electrical interlock that prevents the CIP circuit from being initiated if the proximity switch is not activated. Another method of ensuring the separation of CIP fluid and the process is to utilise either 'block and bleed' valves or 'double seated' valves: these provide extra security within the design, with any leakage past a seal being clearly evident at the valve location. These valves need to be installed in an area that can be easily seen, to avoid potential product or CIP fluid losses in the event of a leak.

1.5.5 *The selection of detergents*

In addition to the points already enumerated, water quality will be a major factor in detergent selection. This, together with a detailed discussion of detergent chemistry, is reviewed in Chapter 4, but the following general points should be considered.

The attributes of detergents

A CIP detergent will ideally exhibit the following attributes:

- organic dissolving power – to solubilise the proteins and fats;
- dispersing and suspending power – to bring insoluble soils into suspension and prevent their redeposition on cleaned surfaces;
- emulsifying power – to hold oils and fats dispersed within the cleaning solution;
- sequestering power – the ability to combine with calcium and magnesium salts, as found, for example, in hard water, to form water-soluble compounds, and to aid detergency and rinseability;
- wetting power – to reduce surface tension, and thus aid soil penetration; and
- rinsing power – the ability to rinse away clearly and completely without leaving any trace of soil or the detergent chemical on the cleaned surface.

The mechanisms of soil removal

During the cleaning cycle, energy is applied to the soil in three basic forms:

- kinetic energy in the form of solution turbulence;
- thermal energy in the form of solution temperature; and
- chemical energy – that is, the chemical reactions between the detergent components and the soil.

A deficiency in one of these energy components may be partially compensated for by an increase in the others, but all three are vital to the total operation. For example, the change from laminar to turbulent flow occurs in pipelines at certain critical flowrates, dependent on the pipe diameter, solution viscosity and temperature conditions. Successful cleaning of pipelines is generally associated only with turbulent flow conditions, 1.5 m s^{-1} being regarded as the accepted target flowrate for pipeline CIP. Where pipelines, plate heat exchangers and similar items of plant can be completely filled with detergent and circulated, this is referred to as a *closed CIP circuit*. Where large items of plant, such as storage tanks, have to be cleaned, it would be impractical to make a closed circuit by filling the vessel. The usual approach is to spray cleaning fluids onto the vessel walls via a spray device and pick up the detergent at the vessel outlet for return to the CIP set via a recovery or scavenge pump. Such a system is referred to as an *open CIP circuit*. These concepts are developed further in Chapter 7. It is generally accepted that temperatures above the melting point of butterfat are necessary where milk-based soils are being addressed. Thus temperatures below 60°C are less likely to yield satisfactory standards. However, as a generalisation, an increase of 10°C will increase the rate of chemical reaction by a factor of between 1.5 and 2.0, and there is a ‘pay-off’ between thermal and chemical energy input (IDF, 1979). In the beverage sector, CIP is very often carried out at ambient temperatures, but generally at much higher caustic alkalinity levels (2–3 g 100 g^{-1} caustic soda) than those employed in the dairy sector (1.0 g 100 g^{-1} caustic soda). In the brewery sector, consideration must be given to the effects of CO_2 on the cleaning process in terms of its reaction with caustic soda to form carbonate, which exhibits much poorer cleaning performance.

1.6 Conclusions

In summary, the basic principles of cleaning are as follows.

- Consider the physical nature and construction of the equipment to be cleaned.
- Assess the nature of the soil to be removed.
- Select a detergent appropriate to the removal of that soil.
- Bring the soil and the detergent together: that is,
  (a) at the right temperature
  (b) under the right conditions of flow and turbulence
  (c) at the right chemical concentration
  (d) for the right period of time.

- Rinse away all traces of detergent and soil, with the objective of achieving the standard of cleanliness appropriate to the duty for which the equipment is destined to be used.
- Always undertake cleaning as soon as possible after completion of the production operation.
- Where necessary, undertake a disinfection or sterilisation process immediately before the equipment is returned to processing or production duties in order to reduce the level of microbiological contamination to one consistent with the hygienic standard required for that duty.

References

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2 Fluid Flow Dynamics

M.J. Lewis

2.1 Introduction

An understanding of the principles of fluid flow is important for efficient cleaning of food processing equipment, as well as for storage tanks for raw milk and finished milk products. Fouling deposits will form during processing and also during storage, and therefore the effective removal of these deposits, thereby leaving the surface free of chemical residues and micro-organisms, is essential for ensuring food safety and quality.

This chapter will be devoted mainly to the cleaning of pipelines, heat exchangers, evaporators and membrane processing equipment (see also Chapter 10). This processing equipment is essentially enclosed, and some is probably operating under pressure. In these situations, fouling is likely to be more severe, especially where heat has been applied. It is important to remember that other vessels, such as storage tanks and road tankers, will also need cleaning. This involves the use of spray nozzles, spray balls and other devices to contact the detergent with the surface. Cleaning is best achieved by pumping detergent at high velocity over the surface. Temperature and detergent strength are also important. There is a need to provide both kinetic and thermal energy; usually cleaning flow velocities are much higher than normal processing flows, and different pumps may be used for the cleaning and disinfecting. Energy needs to be supplied to the pump to overcome frictional losses. This pumping energy is also converted largely to heat. The aim of this chapter is to provide an overview of the basic principles involved to make you feel more comfortable with them, but without covering the complexities of theoretical and computational fluid dynamics.

One important contribution towards making cleaning more efficient and reducing the costs of cleaning is to cut down the amount of fouling deposit that accumulates in the first place. In this sense, measurements of pressures and flowrates can be beneficially used to monitor fouling, and to also monitor the cleaning process. One final interesting question relates to knowing when cleaning has been properly completed.

2.2 Some background principles

Cleaning-in-place (CIP) is about contacting liquid detergents and sanitising fluids with soiled food processing surfaces, and involves pumping the liquid across the surface. Therefore it is important to understand the principles of fluid statics and, especially, fluid dynamics.

Fluid statics deals with fluids at rest, and fluid dynamics deals with fluids in motion. In any cleaning operation there will be a relationship between the volumetric flowrate Q through the equipment and the pressure drop, or head loss, ΔP. It is important to understand

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the factors affecting this relationship. Key to successful cleaning is the ability to pump the detergent, and to provide the correct flow velocities and adequate turbulence. It is also very important to understand the characteristics and behaviour of different pumps.

2.3 Some background information

Pressure is defined as force per unit area. Thus:

$$\text{Pressure} = \frac{\text{force}}{\text{area}} = \text{N m}^{-2}\ (\text{Pa})$$

The SI unit of pressure is the *pascal* (Pa), which is a very small unit. For convenience, the *bar* is often used, where 1 bar = 10^5 Pa. One bar is approximately equal to 1 atmosphere. Also used are kilopascals (kPa) and megapascals (MPa), whereas the Imperial units are pounds force per square inch (psi).

The most common gauge for measuring pressure is a Bourdon-type gauge. For hygienic applications, a diaphragm gauge would be required. Pressure can be measured and recorded either as an absolute pressure or as a gauge pressure. Absolute pressure is measured above an absolute vacuum (or zero absolute pressure), and zero absolute pressure is the lowest pressure attainable.

Gauge pressure is measured above or below atmospheric pressure (see Figure 2.1). Gauges measure pressure either above atmospheric pressure (pressure gauges) or below atmospheric pressure (vacuum gauges). However, some equipment, such as evaporators, may operate below atmospheric pressure when concentrating milk, but above atmospheric pressure when being cleaned and sterilised. In this case, a compound gauge may be more appropriate.

Atmospheric pressure itself changes continuously, and is measured accurately by a barometer. When listening to the weather forecast, we are all familiar with regions of high pressure and regions of low pressure! Some values for one standard atmosphere in different units are given in Section 2.9.1.

The relationship is: absolute pressure is gauge pressure plus atmospheric pressure (i.e. in consistent units), i.e.

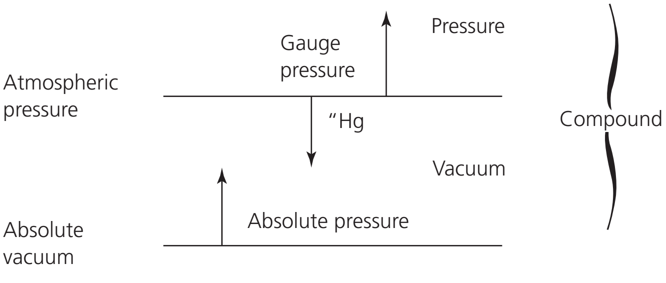


Fig. 2.1 The relationship between absolute pressure and gauge pressure. Reproduced from Lewis (1990) with permission from Woodhead Publishing.

$$P_{\text{absolute}} = P_{\text{gauge}} + P_{\text{atmospheric}}$$

This distinction is important, because tables – such as many thermodynamic tables (e.g. steam tables), for example – usually quote pressures in absolute terms; also, equations involving the gas laws (e.g. $PV = RT$) require the substitution of absolute pressures. Some conversion factors are given in Section 2.9.7.

When a fluid flows along a pipe or passes through a spray nozzle, there is a drop in pressure, also known as a *pressure differential*, ΔP. Similarly, the function of a pump is to increase the pressure of a fluid in order to overcome the frictional losses (see Section 2.5).

Differential pressure is thus

$$\Delta P = P_2 - P_1$$

Pressures and differential pressures can also be expressed as a height or *head* of fluid. The relationship between them is given by the following equation:

$$P \text{ or } \Delta P = \rho g h$$

where P (ΔP) is pressure (or differential pressure) (N m^{-2}), ρ is fluid density (kg m^{-3}), h is head (m), and g is the acceleration due to gravity (9.91 m s^{-2}). This relationship is useful in a number of different circumstances.

For example, consider a pressure drop of 0.5 MPa, or a pump that develops a pressure of 0.5 MPa. In both cases, if the fluid is milk (ρ = 1030 kg m^{-3}), then the head is

$$h = \frac{\Delta P}{\rho g} = \frac{5 \times 10^5}{1030 \times 9.81} = 49.5 \text{ m}$$

For pumps, knowledge of the head developed by a pump is more immediate: in this example, the pump is capable of pumping milk to a height of 49.5 m.

Following from this, the pressure at any depth in a tank will increase with the depth of immersion (h in Figure 2.2):

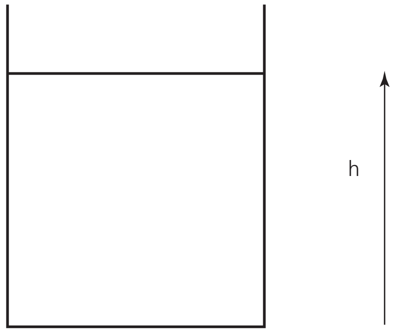


Fig. 2.2 Pressure increases with depth of immersion.

$$P = P_{\text{surface}} + \rho g h$$

If a pressure gauge is inserted in the base of the tank, the pressure will increase as the height of the liquid increases. This principle can be used to monitor the height of liquid, and hence the volume in the tank: that is, its status (full, half-full or empty). Other factors that have to be considered are as follows.

2.3.1 *Temperature*

Temperature is a very important property, because it will affect viscosity and alter reaction rates, which is important for chemical cleaners. Temperature can simply be defined as the degree of hotness. Of more importance, it determines the direction of heat transfer: energy is transferred from a high to a low temperature, and the rate of heat transfer (J s^{-1} or W) is proportional to the temperature difference. Temperature control is important, and hotter is not always better. In this context, accurate temperature measurement and regular calibration of thermometers are important. More detail is provided in Section 2.9.

Energy is required in cleaning operations to bring detergents to the required temperature. The amount of energy Q can be calculated from the equation

$$Q = mc\Delta\theta$$

where m is mass (kg), c is specific heat (J kg^{-1} K^{-1}), and $\Delta\theta$ is temperature rise (K).

2.3.2 *Volumetric flowrate*

The SI unit of volumetric flowrate is m^3 s^{-1}; this is not a very practical unit, because 1000 L = 1 m^3. However, the Imperial unit is the gallon, where 1 gallon (Imperial) = 4.54 L. The American gallon is smaller than the Imperial gallon (see Section 2.9.2).

For example, 5000 L h^{-1} is equal to 5 m^3 h^{-1} or 5/3600 m^3 s^{-1} = 1.39×10^{-3} m^3 s^{-1}; some conversion factors are given in Section 2.9.2.

2.3.3 *Density*

Density is defined as the mass per unit volume, and the SI units are kg m^{-3}. Water has a density of 1000 kg m^{-3} or 1 g mL^{-1} or 62.3 lb ft^{-3}. Milk has a density usually between 1025 and 1035 kg m^{-3}. The theory of fluid dynamics is much simplified by assuming that fluids are incompressible – that is, the fluid density is not affected by moderate change in temperatures and pressures. This is a reasonable assumption to make, and leads to simplified solutions to fluid flow problems. This is in contrast to gases and vapours, which are highly compressible. However, in reality the density of most fluids is slightly temperature dependent. In fact, as fluids get hotter, they become less dense, and this provides the driving force behind natural convection. The addition of most solids, such as minerals, sugars or proteins, will increase the density, whereas oils and fats will decrease the density. The densities (kg m^{-3}) of solid constituents have been summarised by Peleg & Bagley (1983) and Walstra & Jenness (1984) as follows: lactose (1780), protein (1400), fat (900–950), salt (2160), and water (1000).

2.3.4 *Specific gravity*

Specific gravity is less susceptible to changes in temperature, compared with density. The relationship between the specific gravity and the density (ρ) of a material is given by

$$\mathrm{SG} = \frac{\rho}{\rho_{\mathrm{water}}}$$

Density and specific gravity could be useful for monitoring changes occurring during cleaning, as removal of soil from surfaces will change the density. When air is incorporated into a liquid, the density decreases. The amount of air incorporated is measured by the *overrun*, which is the increase in volume divided by the original volume (i.e. expressed as a percentage). This may be relevant where excessive foaming occurs, and may be a nuisance as it decreases density and may interfere with the performance of the pump.

2.3.5 *Viscosity (η) and rheology*

Rheology is the study of the deformation of materials subjected to applied forces. A distinction is usually made between fluids and solids: fluids will flow under the influence of forces, whereas solids will stretch, buckle or break. The viscosity of a fluid is defined as the internal friction within the fluid. Shear stress τ is defined as the shearing force F divided by the surface area A over which the force is applied. This gives rise to a velocity gradient or *rate of shear* ($\mathrm{d}v/\mathrm{d}y$) (Figure 2.3).

For Newtonian fluids there is a direct relationship between the shear stress and the rate of shear. The ratio of the shear stress to shear rate is known as the *dynamic viscosity* or *coefficient of viscosity*, η:

$$\eta = \frac{\text{shear stress}}{\text{shear rate}} = \frac{\tau}{\mathrm{d}v/\mathrm{d}y}$$

The viscosity of water at 20°C is 1.002×10^{-3} N s m^{-2} or 1.002 cP (note that the centipoise is still in common use). Viscosity is highly temperature dependent, with the viscosity of

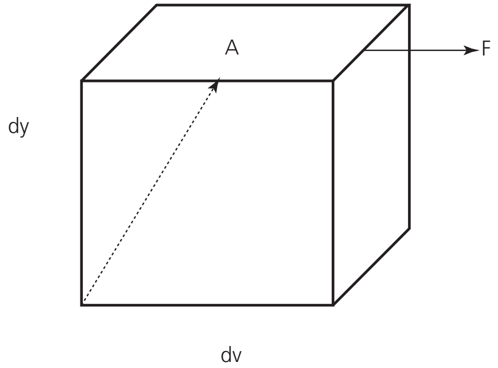


Fig. 2.3 Showing the concepts of shear stress and shear rate.

fluids decreasing with increasing temperature between 2% and 10% for each degree Celsius: therefore it is very important to control the temperature accurately when measuring the viscosity, and the temperatures should always be quoted with the results. The viscosity of some dairy fluids at different temperatures is given in Section 2.9.10.

Occasionally, it is more appropriate to use the term *kinematic viscosity*, defined as

$$kv = \frac{\mathrm{d}v}{\text{density}}$$

The units of kinematic viscosity are as follows:

	Dynamic viscosity	Density	Kinematic viscosity
cgs	poise	g mL^{-1}	cm^2 s^{-1} (Stoke)
SI	N s m^{-2} (Pa s)	kg m^{-3}	m^2 s^{-1}

Kinematic viscosity is measured directly by the Ostwald capillary flow-type viscometer. This is one of the most accurate instruments for detecting small changes in the viscosity of milk.

Milk, skimmed milk, cheese whey and whey permeate are usually considered to be Newtonian fluids, as are dilute detergent solutions. In more complex fluids there are considerable interactions, which result in non-linear relationships between shear stress and shear rate. Various types of non-Newtonian behaviour are recognisable. To detect non-Newtonian behaviour requires the use of variable-speed rotational viscometers. Thus, by altering the speed, it is possible to alter the shear rate: at each shear rate, the corresponding shear stress is measured. Non-Newtonian fluids are characterised by an *apparent viscosity* η_a, where

$$\eta_a = \frac{\tau}{\mathrm{d}v/\mathrm{d}y}$$

Many time-independent non-Newtonian fluids obey a power law equation:

$$\tau = k\left(\frac{\mathrm{d}v}{\mathrm{d}y}\right)^n$$

A straight-line relationship results when log τ is plotted against log(dv/dy). The consistency index k and the power law index n are often used to characterise the behaviour of such fluids.

Shear thinning or pseudoplastic behaviour is the most commonly encountered ($n < 1$), whereby the apparent viscosity reduces as the shear rate increases. Dilatant behaviour or *shear thickening* ($n > 1$) shows the opposite, and is not often encountered. Cleaning fluids are most likely to be Newtonian ($n = 1$) at the start of the cleaning cycle; their viscosity characteristics may change as they solubilise fouling material, but probably not by much.

2.3.6 *Continuity equations and energy balances*

For a fluid flowing through a circular aperture, the *average velocity* is given by

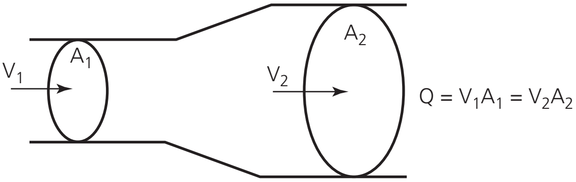


Fig. 2.4 Flow along a tube.

$$v_{\text{av}} = \frac{Q}{A} = \frac{4Q}{\pi D^2}$$

Sometimes this is known as the superficial or plug flow velocity (Figure 2.4). In reality, some elements of the fluid will move at higher velocities, and some at lower velocities. The velocity of a fluid at a pipe wall is often assumed to be zero.

An *average residence time* is based on the average velocity:

$$t_{\text{av}} = \frac{L}{v_{\text{av}}}$$

At any two points along a flow system, the volumetric flowrates have to be equal. As volumetric flowrate is equal to the product of average velocity v and cross-sectional area A, then

$$v_1 A_1 = v_2 A_2$$

The consequence of this is that fluid velocity will increase when a fluid enters a restriction. For example, if the pipe diameter is reduced by half, the average velocity will increase by four times. This will also lead to a pressure drop (see later in this section).

The principle of conservation of energy is represented in fluid dynamics in terms of *Bernoulli's equation*. This states that the total energy at any two locations along a tube (1 and 2) is equal. The total energy at any location is made up of *kinetic energy*, *potential energy* and *pressure energy*. There are several ways that these energy terms can be expressed; this author's preference is in terms of head (see Figure 2.5). The terms are the pressure head $P/\rho g$, velocity head $v^2/2g$ and potential head h.

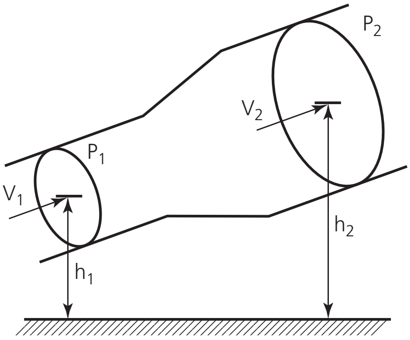


Fig. 2.5 Bernoulli's equation of motion (ideal situation).

Thus the ideal energy balance (i.e. assuming no frictional losses) is

$$\frac{P_1}{\rho g}+\frac{v_1^2}{2g}+h_1=\frac{P_2}{\rho g}+\frac{v_2^2}{2g}+h_2$$

If the tube is horizontal, then $h_1 = h_2$.

Thus

$$\frac{P_1-P_2}{\rho g}=\frac{v_2^2-v_1^2}{2g}$$

When frictional losses and the pumping energy (also expressed as a head, W) are taken into account (see Figure 2.6), the equation becomes

$$\frac{P_1}{\rho g}+\frac{v_1^2}{2g}+h_1+W=\frac{P_2}{\rho g}+\frac{v_2^2}{2g}+h_2+F$$

The following section will deal with factors affecting the frictional losses and the calculation of frictional losses, which in turn will lead to being able to determine W and select the correct pump for the application.

2.4 Streamline and turbulent flow

When a fluid flows thorough a pipe, the flow is one of two possible types: *streamline flow* (sometimes called laminar or viscous flow) or *turbulent flow*. For cleaning operations and efficient heat transfer, turbulent flow is usually required. The type of flow can be distinguished by a dimensionless group known as the *Reynolds number* (*Re*): this represents the ratio of the inertial forces to the viscous forces acting upon the liquid. When inertial forces predominate, the flow is turbulent, and when viscous forced predominate, the flow is streamline. For pipeline flow, the Reynolds number is

$$Re=\frac{vD\rho}{\eta}=\frac{4Q\rho}{\pi\eta D}$$

If the Reynolds number is less than 2000, the flow is streamline; if it is greater than 4100, the flow is turbulent. What is really interesting is that this applies whatever the dimensions

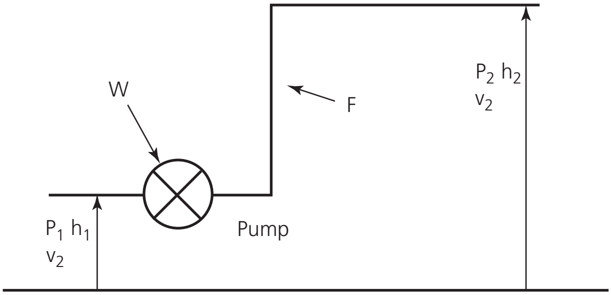


Fig. 2.6 Bernoulli's equation of motion (practical situation).

of the pipe and whatever the fluid. However, it is important to know the physical properties of the fluid already described. Knowing whether the flow is streamline or turbulent is extremely useful, and knowledge of the Reynolds number is required to determine the frictional losses (see Section 2.5).

Streamline flow is characterised by a parabolic velocity profile, with the maximum velocity being twice the average velocity:

$$v_{\text{max}} = 2v_{\text{av}}$$

The fluid can be imagined as existing as distinct layers, with the layer in contact with the wall having zero velocity, and the velocity increasing with increasing distance from the pipe wall (Figures 2.7 and 2.8). There is no bulk movement between layers, and the flow is smooth and not conducive to either mixing or heat transfer. In fact, the whole of the flow region behaves as a boundary layer. When streamline flow exists, there is also a wide spread of residence times.

When the flow is streamline, the relationship between pressure drop and flowrate can be predicted from *Poiseuille's equation*. For a fluid flowing along a tube of length *L* and diameter *D*:

$$Q = \frac{\Delta P \pi D^4}{128 L \eta}$$

This can be rearranged to give the head loss in terms of parameters that can be measured:

$$F = \frac{128 L Q \eta}{\pi D^4 \rho g}$$

This equation can be used to determine dynamic viscosity; it forms the basis behind capillary flow viscometers. If volumetric flowrate is plotted against the pressure drop, there is a linear relationship, and if plotted on log–log paper, the gradient would be 1.

If streamline flow conditions exist, increasing the flow velocity will eventually lead to a transition from streamline to turbulent flow. Turbulent flow involves random fluctuations in fluid velocity in an axial direction superimposed on the bulk flow along the tube. As a consequence, there is good radial mixing (Figure 2.8).

Where turbulent flow exists in a pipe, there are two distinct regions: first, a boundary layer adjacent to the pipe wall, which gets thinner as Reynolds number increases; and second, a bulk fluid region, which is well mixed. The velocity profile across the tube is much flatter, and the mixing across the tube is much in evidence. At the onset of turbu-

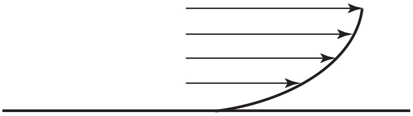


Fig. 2.7 Velocity profile across a fluid, streamline flow profile.

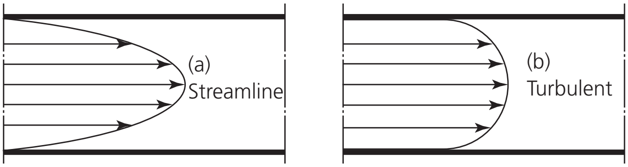


Fig. 2.8 (a) Streamline and (b) turbulent flow profiles. Reproduced with permission from Tetra Pak, Lund, Sweden.

lence there is a change in relationship between pressure drop and volumetric flowrate (Figure 2.9).

The relationship between the maximum velocity and average velocity is

$$\text{Maximum velocity } v_{\text{max}} = 1.2 v_{\text{av}}$$

As one might expect, frictional losses are higher when the flow is turbulent, and the distribution of residence times is narrower.

2.5 Calculation of frictional loss in a straight pipe

Calculating frictional losses is more complex when the flow is turbulent. Use is made of friction factors and their relationship with the wall shear stress τ_w. A force balance over the pipe (Lewis, 1990) gives rise to the following equation:

$$\tau_w = \frac{\Delta PD}{4L}$$

Thus the wall shear stress is dependent upon the pressure drop and the pipe dimensions. It gives a measure of the forces acting at the surface of the wall.

To calculate the pressure drop and head loss under turbulent flow conditions, a dimensionless *basic friction factor* ϕ has been introduced. It is defined as follows:

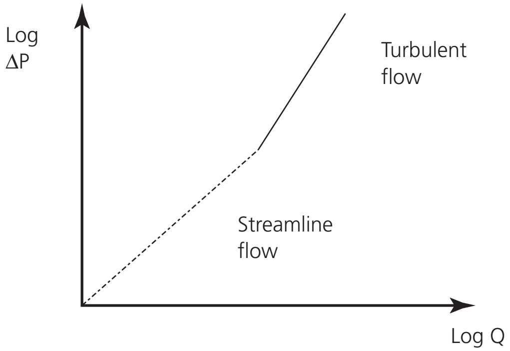


Fig. 2.9 The relationship between pressure drop and flowrate for streamline and turbulent flow.

$$\phi = \frac{\tau_w}{\rho v^2}$$

Experimentally it has been found that the basic friction factor depends upon the extent of turbulence and the roughness of the pipe. The results have been presented graphically in terms of basic friction factor plotted against the Reynolds number. A detailed chart (Figure 2.10) is provided in Coulson (1996). If charts are not available, the relationship between the basic friction factor and the Reynolds number for flow in a smooth circular tube under turbulent flow conditions is given by the *Blasius equation*:

$$\phi = 0.0396Re^{-0.25}$$

Once the friction factor has been determined, it can be substituted into the following equation to determine the head loss F:

$$F = 8\phi \frac{L}{D}\frac{v^2}{2g}$$

where L is the pipe length and D is the pipe diameter.

For example, consider the flow of water along a pipe of diameter 5 cm and 100 m long, at different flow velocities of 1 to 10 m s^{-1} (viscosity of water = 10^{-3} Pa s and density = 10^3 kg m^{-3}). The results for the frictional loss (head loss) are shown in Table 2.1. The cal-

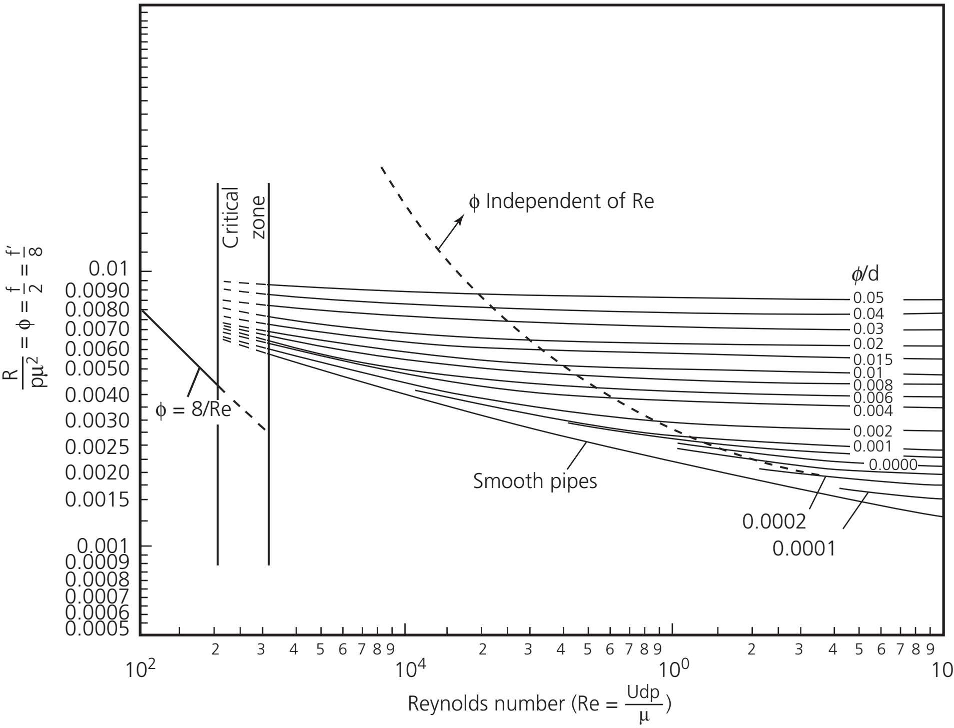


Fig. 2.10 Relationship between friction factor and Reynolds number. Reprinted from Coulson (1996), copyright 1996, with permission from Elsevier.

Table 2.1 Head loss in a smooth pipe for water determined by two methods.

Velocity (m s^{-1})	Reynolds number	Friction factor (chart)	Head loss (m) (chart)	Friction factor Blasius	Head loss (m) Blasius
1	50 000	0.0025	2.04	0.00265	2.16
2	100 000	0.0021	7.18	0.00223	7.26
5	250 000	0.0018	36.70	0.00177	36.10
10[a]	500 000	0.0016	130.50	0.00149	121.40

[a] This corresponds to a volumetric flowrate of 70.7 m^3 h^{-1}.

culations are for smooth pipes, using friction factors determined from the friction chart and from the Blasius equation.

The calculations are repeated for milk, with a viscosity of 0.002 Pa s and a density of 1035 kg m^{-3}, at the same flow velocities. It can be seen in Table 2.2 that the head loss for milk is slightly higher than that for water at the same flow velocity, caused by its slightly higher viscosity. The calculation is repeated for water at the same flowrate (70.7 m^3 h^{-1}) in wider-diameter tubes (Table 2.3).

Increasing the pipe diameter for the same flowrate significantly reduces the head loss and hence the pumping costs. However, it does increase the cost of the pipework. This leads to the concept of an economic pipe diameter: that is, the diameter of the pipe that gives the minimum overall cost when capital and energy costs are taken into account.

Note that all previous calculations were performed for smooth pipes. *Pipe roughness* can be taken into account by a dimensionless factor e/D, where e is the average height of roughness**.** Thus for a smooth pipe e/D equals zero; as the pipe surface gets rougher, the ratio e/D increases. Similar calculations can be done using the chart taking into account pipe roughness, given by e/D. The head loss for different e/D values is given in Table 2.4 and, for water at a flow velocity of 10 m s^{-1}, the e/D values are also shown in Table 2.4. It can be seen that frictional losses become much higher at higher e/D values. Also, surface roughness will be more influential in small-diameter pipes than in large pipes.

Frictional losses will also occur in other fittings, such as bends, exits, restrictions and enlargements, and valves. One commonly used procedure is to express this in terms of the *velocity head* ($v^2/2g$), where

Table 2.2 Head loss in a smooth pipe for milk determined by two methods.

Velocity (m s^{-1})	Reynolds number	Friction factor (chart)	Head loss (m) (chart)	Friction factor Blasius	Head loss (m) Blasius
1	25 750	0.0030	2.45	0.00313	2.55
2	51 500	0.0025	8.15	0.00263	8.57
5	128 750	0.0022	44.80	0.00209	42.60
10[a]	257 500	0.0018	146.80	0.00176	143.40

[a] This corresponds to a volumetric flowrate of 70.7 m^3 h^{-1}.

Table 2.3 Head loss for water at a constant flowrate (70.7 $m^3 h^{-1}$) in pipes of different diameter.

Velocity ($m s^{-1}$)	Diameter (cm)	Reynolds number	Friction factor (chart)	Head loss (m) (chart)	Friction factor Blasius	Head loss (m) Blasius
10	5	500 000	0.0016	130.5	0.00149	121.4
2.51	10	250 000	0.0018	4.59	0.00177	4.51
0.625	20	125 000	0.0021	0.167	0.00211	0.167

This calculation is also performed using the data presented in Figure 2.15. The head loss is about 4.9 m; these are more approximate values but are in good agreement with those calculated.

$$F = K\frac{v^2}{2g}$$

Values are given for some fittings in Table 2.5.

Another method is to express the losses in terms of an *equivalent pipe diameter*, *L*/*D*. Thus a bend is equivalent to 60 pipe diameters (whatever *D* is); this is further discussed by Coulson (1996) and Holland (1995). The procedures described here for determining frictional losses are in terms of a basic friction factor. A detailed plot of the basic friction factor against Reynolds number is given in Coulson (1996). Other friction factors that are encountered in the literature are the *Fanning friction factor* and the *Moody friction factor*. However, the procedures for calculating friction losses are similar.

The total frictional losses are determined by summing the head losses for all the individual components – that is, the straight pipe lengths, bends, section changes in fittings, valves and other components. A typical relationship between total head loss and volumetric flowrate is shown in Figure 2.11. The effects of a throttling valve closing are also shown in Figure 2.11. Change the characteristic from 1 to 2, and we see that the head loss increases for a fixed flowrate. Superimposed on this is the *pump characteristic curve* (see Section 2.6). Similar principles apply to head losses through nozzles and spray balls.

To summarise, it is important to know the relationship between flowrate and pressure drop for the system being cleaned, preferably at the operating conditions involved and for the detergents in question.

Table 2.4 Frictional losses at different *e*/*D* values.

Velocity ($m s^{-1}$)	Pipe roughness (*e*/*D*)	Friction factor (chart)	Head loss (chart)
10	Smooth	0.0016	130.5
10	0.00001	0.00163	132.9
10	0.0001	0.00175	142.7
10	0.001	0.0024	195.7
10	0.01	0.0046	375.1

Note: Reynolds number is 500 000.

Table 2.5 Loss coefficients and head loss for different fittings.

Fittings	Loss coefficient, *K*	Head loss (m)[a]
Bend (e.g. 90%)	0.4	2.04
Sharp-edged orifice	0.5	2.54
Branch (straight through)	0.9	4.59
Tee-piece (straight through)	0.4	1.53
Sudden increase	0.3 to 1.0	1.53 to 5.10
Sudden decrease	0.2 to 0.4	1.02 to 2.04
Gate valve[b]	0.2	1.02
Globe valve[b]	3.0	15.3
Butterfly valve[b]	0.2	1.2
Ballcock[b]	0.1	0.51

[a] Calculated at a flow velocity of 10 m s^{-1}.
[b] Valves fully open.

2.6 Pump characteristics

The other important component of any cleaning system is the pump. It provides the energy to overcome the frictional losses, and to deliver the cleaning fluid to the locations of interest. When specifying a pump for an application, again there are two important elements: the pressure head to be developed, and the flowrate. Normally a high volumetric flowrate is required in order to generate turbulence and high wall shear stress values.

The theoretical power requirement W can be calculated from

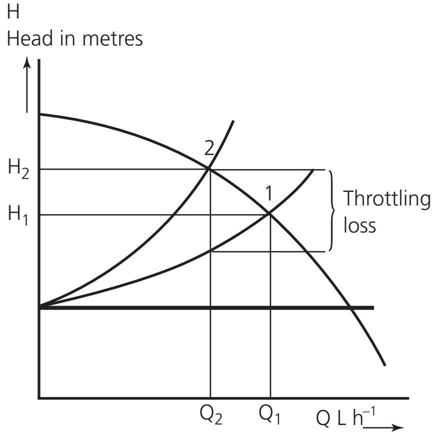


Fig. 2.11 The relationship between frictional loss and flowrate, showing the effects of throttling valve; also shows a pump characteristic and the corresponding operating points. Reproduced with permission from Tetra Pak, Lund, Sweden.

$$W = m'hg$$

where m' is mass flowrate (kg s^{-1}), h is head developed (m), and g is the acceleration due to gravity, 9.81 m s^{-2}. Since most of the pumping energy is converted to heat, this gives an indication of the heat input from the pump. As pumps are not 100% efficient, efficiency factors need to be known.

Pumps are broadly classified as either centrifugal or positive displacement, and each has its advantages and disadvantages. *Centrifugal pumps* are very widely used (Figure 2.12). They can deliver high volumetric flowrates at relatively high pressures. One of their most important characteristics is that the flowrate developed is dependent upon the discharge conditions, and the relationship is covered by a characteristic curve for each pump (see Figure 2.11). This characteristic curve can be changed by changing the pump speed and impeller diameter, but for a selected pump it remains constant. In fact a pump provider will usually have the characteristics for a wide range of their pumps on one diagram (Figure 2.12).

One of the drawbacks of centrifugal pumps is that they do not give a constant flowrate: this will alter if the discharge conditions change – for example if the pipe diameter gets smaller because of deposit formation. They are also not self-priming, so pump positioning is important. They are reliable and cheaper than positive pumps, and will not be damaged if pumping against a closed discharge. They are probably the most widely used pumps on CIP systems. Flowrate is controlled by throttling valves or by altering pump speed. Figure 2.11 shows how this works; the flowrate will also fall for higher-viscosity fluids.

The other classification is the *positive pump*, whereby a fixed volume of fluid is forced out per revolution, as in a rotary pump, or per stroke, as in a piston pump. A diagram for a

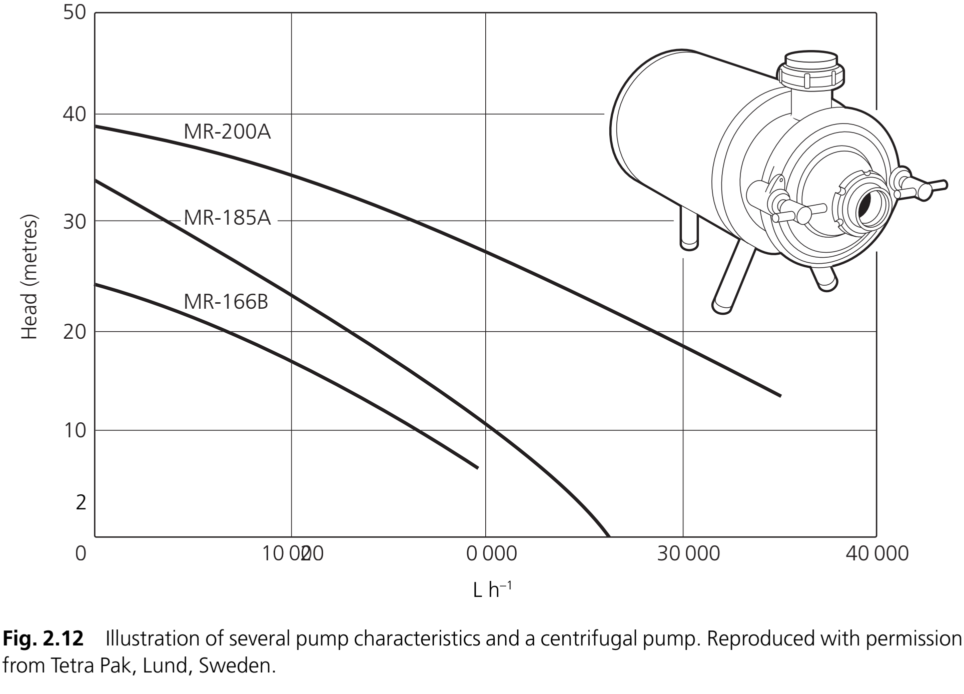


Fig. 2.12 Illustration of several pump characteristics and a centrifugal pump. Reproduced with permission from Tetra Pak, Lund, Sweden.

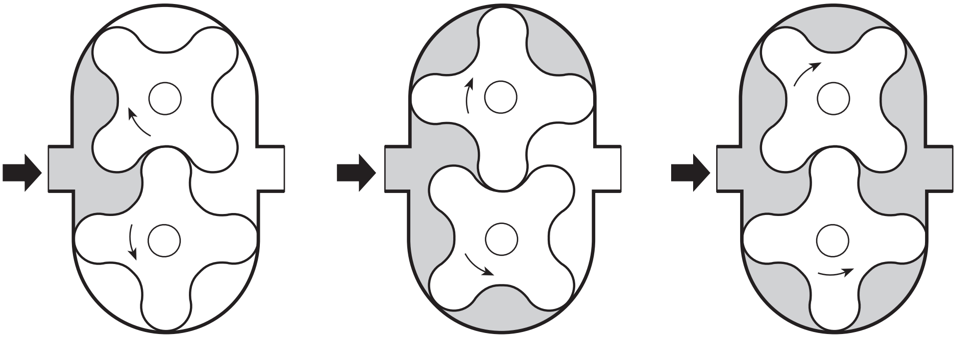


Fig. 2.13 The principal functions of the rotary pump. Reproduced with permission from Tetra Pak, Lund, Sweden.

rotary pump is shown in Figure 2.13. Positive pumps are used in more specialised applications where higher pressures or more constant flowrates are essential: piston, rotary or screw pumps (Mono type pumps). In theory, their main characteristic is that the flowrate is independent of discharge conditions, and should remain constant, although no pump is truly positive, mainly because some slip always occurs. Note that the concept is useful in pasteurisation and UHT sterilisation processes; a high-pressure homogeniser is another example of a positive pump. Processing lines incorporating a homogeniser often need a separate CIP pumping system to generate higher flowrates.

Such pumps cope better with high-viscosity fluids. In general, they are more expensive and require more maintenance. Flowrates can be regulated by changing the pump speed: this is often done by an electrical inverter, which changes the frequency of the supplied power. Pumps can be fitted with steam seals where ultra-clean or aseptic operations are required.

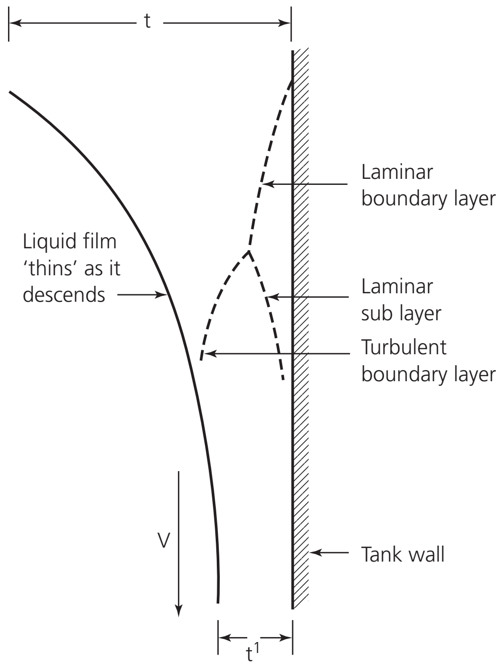
Thus the pump is an integral part of the CIP system. It is very important to ensure that the pump is properly maintained, and in particular that it is delivering the correct volumetric flowrate.

2.7 Tank cleaning heads and falling films

Liquid is distributed by a spray nozzle, where the pressure drop and/or flowrate relationship is very important. Again, the relationship between pressure drop and flowrate is an important characteristic of the nozzle, as it will affect the overall system loss. This is discussed in Chapter 6.

In tank cleaning, the detergent is sprayed into the top of the tank, and a falling film of detergent is created to enable cleaning. Thus the principles of falling films are encountered in the cleaning of tanks, and the detergent must be sufficiently turbulent and must contain sufficient energy to maintain its temperature, accounting for heat losses. Furthermore, the flow must be sufficiently high to minimise any bridging that might take place around more tenacious soiling.

The liquid detergent or sanitiser falls under gravity, and will reach its terminal velocity when the force acting upwards – that is, the wall shear stress – counteracts the gravitational



t = initial film thickness
t^1 = final film thickness (mean value)
Steady state vertical flow at velocity V

Fig. 2.14 Illustration of film formation.

force. Falling films can remain laminar for much higher Reynolds numbers. At certain stages of development, waves appear on the surface, producing an infinitely varying film thickness down the tank. These irregular wave patterns result mainly from turbulent flow at the liquid/air interface (see Figure 2.14).

Given the complexity of the falling film effect and the number of variables involved, it is very difficult to predict the exact requirement for each kind of tank size and cleaning application. The system is first over-designed to ensure that cleaning is more than adequately covered, and then the designer proceeds to optimise the performance to reduce energy, chemical and water and waste costs.

2.8 Some concluding remarks

A complete processing run, for example HTST pasteurisation, involves disinfecting the plant, running the plant (hopefully for many hours), rinsing, cleaning and perhaps a final disinfecting. Ideally, it would be very informative to know how much fouling had taken place, and how effective the cleaning process was. If this were possible, it should then be possible to try and understand how fouling or soiling occur, and how they are affected by raw milk quality and processing conditions. If this could be achieved, processing conditions could be optimised to minimise the amount of fouling that takes place. This in turn should reduce the cleaning time and make the process more efficient in terms of total time, reducing other costs such as cleaning and energy costs, as well as minimising effluent processing

Thus proper application of the principles of fluid dynamics, in terms of monitoring flowrates, temperatures and pressures, can lead to a better understanding of the effectiveness of the cleaning process. For example, fouling can be monitored by monitoring the relationship between pressure drop and flowrate, or by measuring overall heat transfer coefficients by monitoring changes in steam pressures and temperatures. Conversely, cleaning can be monitored by measuring pressure drop recovery. In fact, such measurements have shown that, on contact with hot caustic soda, the fouling deposit initially expands before it is removed. Such data have also been able to show that increasing the concentration of caustic soda does not always increase its effectiveness.

A relatively new area for understanding fluid flow is *computational fluid dynamics* (CFD). This is an exciting area, which is providing interesting insights into shear stress values at pipe walls that are difficult to evaluate by conventional methods. However, it is a highly academic subject: it requires a solid background in both fluid dynamics and numerical analysis, and significant errors have been made by people lacking knowledge in one or the other. An example of its application is in a paper by Jensen *et al.* (2006). These proceedings summarise papers presented at a recent international conference held on fouling, cleaning and disinfecting, which is relevant to the theme of this publication.

2.9 Appendix: definitions and equations

2.9.1 *Pressure*

The SI unit is the pascal (Pa) = 1 N m^{-2}
1 bar = 10^5 Pa or 0.1 MPa
Atmospheric pressure = 1.013 bar = 1.033 kg(f) cm^{-2} = 14.69 psi = 760 mmHg
Note: 1 mmHg = 1 torr

2.9.2 *Volume and volumetric flowrate*

1 m^3 = 1000 L = 10^6 mL (cm^3)
1 gallon (Imperial) = 4.54 L
1 gallon (American) = 3.785 L = 0.85 Imperial gallons
1 gallon h^{-1} = 1.261×10^{-6} $m^3\,s^{-1}$

Figure 2.15 illustrates an alternative procedure for determining friction losses at different flowrates in a straight pipe.

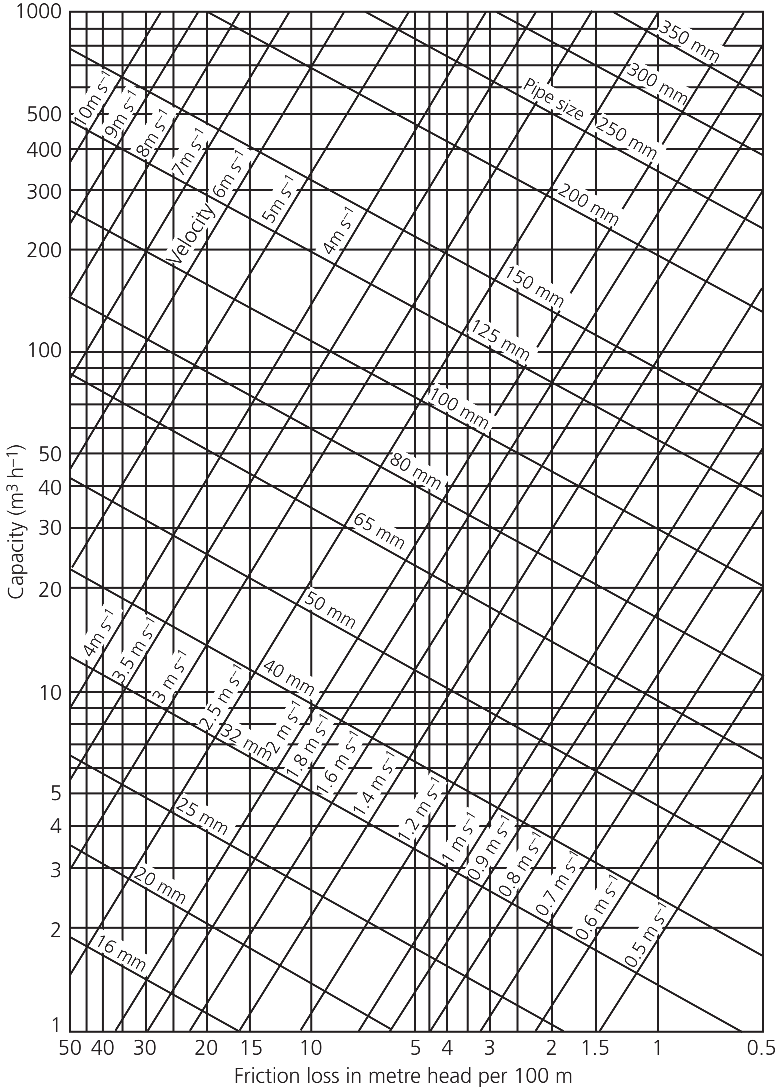


Fig. 2.15 Chart for evaluating head loss for water in a straight pipe.

2.9.3 *Temperature conversions*

°C = (°F – 32) × 0.56
K = °C + 273.15

2.9.4 *Temperature difference*

°C = °F × 0.56
1°C = 1 K

2.9.5 *Fixed points*

	°F	°C	K
Freezing point (water)	32	0	273.15
Boiling point (water)	212	100	373.15

There is an analogy between absolute temperature and absolute pressure, as zero on both scales represents the lowest temperature and pressures that are attainable. However, we do not measure temperature in K, but rather in °C. However, in many equations involving the gas laws and reaction rates (e.g. $PV = RT$) the temperature in K is required.

2.9.6 *Energy units*

1 British thermal unit (Btu)	Energy required to raise 1 lb water from 59°F to 60°F.
1 calorie	Energy required to raise 1 g water from 14.5°C to 15.5°C
1 joule (unit of work) W	Work done in moving 1 newton through 1 metre (J = N m)
1 watt (W)	Rate of heat transfer (1 joule per second, J s^{-1}).

2.9.7 *Some conversion factors*

4.18 J = 1 calorie
1 Btu = 1055 J or 1.055 kJ
1 therm = 10^5 Btu
1 kWh (unit) = 3.6×10^3 kJ or 3.6 MJ

Table 2.6 Viscosity (mPa s) of milk and whey at different temperatures (average values).

	Temperature (°C)			
Product	10	20	40	80
Whole milk	2.79	2.12	1.24	0.68
Skimmed milk	2.44	1.74	1.03	0.53
Whey	1.71	1.26	0.82	0.68

Data compiled from Kessler (1981).

2.9.8 *Specific heat*

Specific heat = energy required to raise unit mass by unit temperature rise (kJ kg^{-1} K^{-1}).

2.9.9 *Density of milk*

The density of cow's milk usually falls within the range 1025 to 1035 kg m^{-3}. It is generally measured with a special hydrometer, known as a lactometer, and the result can be used to estimate total solids (Lewis, 1993):

$$T = 0.25D + 1.22F + 0.72$$

where T is total solids (g 100 g^{-1}), D is 1000(density – 1) (density units are g mL^{-1}), and F is fat percentage (g 100 g^{-1}).

2.9.10 *Viscosity*

For all milk and milk products, there is considerable variation in composition, and hence in viscosity; it is recommended that the viscosity be measured wherever possible. The most convenient instrument for measuring low-viscosity fluids is the capillary flow viscometer, which is sensitive enough to be able to detect the small changes that occur when milk is heated or homogenised. Table 2.6 shows representative values for the viscosity of whole milk, skimmed milk and cheese whey at different temperatures. Most of these fluids exhibit Newtonian behaviour over a moderate range of shear rates.

Homogenisation and heat treatment both tend to increase the viscosity slightly, with homogenisation giving the milk a creamier mouth-feel. The effects of homogenisation become more pronounced as the fat content increases.

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3 Water Supplies in the Food Industry

S.I. Walker

3.1 Introduction

The dairy industry is by no means unique in its use of water during the manufacture of products for the consumer. However, where it does differ in some cases is in the amount of water leaving a site, which can be substantially greater than the water coming into the factory. This is particularly true where processes, such as cheesemaking and evaporation of milk, can greatly influence the quantities and qualities of water leaving site. Examples of some of the processes in relation to water usage and production of different dairy products are shown in Table 3.1. It should also be considered that, as the dairy industry continues to restructure itself to meet changing consumer demands, the size of dairy operations continues to increase, whereas the actual number of dairies decreases in any industrialised country. Although the concentration might be on production, the services and, in particular, water usage are of equal importance. This aspect is of great relevance in Europe, where the dairy industry is amongst the first to come under the remit of integrated pollution prevention and control.

3.2 Sources of water

Water for utilisation in any dairy factory can be obtained from several different sources. These include:

- natural water, including rainwater
- local authority provider

Table 3.1 Some examples of water usage in dairy applications.

	Water efficiency index (WEI) (m^3 water m^{-3} raw milk)		Specific wastewater discharge (SWWD) (m^3 effluent m^{-3} raw milk)	
Application	Average	Target	Average	Target
Fluid milk processing	1.86	0.9	1.3	0.50
Fluid milk processing (Tetra Pak packaging)	1.86	0.5	1.3	0.45
Soft-type cheeses	1.60	0.6	1.3	0.50
Hard-type cheeses	1.75	0.7	1.3	0.45

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- the products
- recycling

Figure 3.1 shows the possible areas where the water might come from, and its usage within the dairy.

3.2.1 *Natural water and rainwater*

Many older factories were traditionally built near rivers and other sources of natural water, such as springs. Thus the factory had a supply of water to assist in its processing, and also to use as an outlet for the wastewater from the site. The term 'natural' for water can be somewhat misleading. In principle, there are two available sources of natural water: (a) *surface water*, which includes lakes, rivers, streams, and polar ice caps; and (b) *groundwater*, such as springs and wells. Even within these two sources of water there can be a mix, because some surface water may percolate through rock to an aquifer, or a water table may rise to maintain a water level in a river. Water from these sources may be subject to seasonal change, in terms of the levels of concentration of certain contaminants, such as iron; and there will be differences in a river stream height between winter and summer. Well water can also vary in height, and therefore regular monitoring of the water quality is required.

Monitoring will also be required to ensure that accidental pollution is controlled. Once an aquifer is polluted, it is difficult to bring it back to its original purity, because the pollutants may not only be in the water, but also adsorbed onto the rocks of the substrata.

In certain parts of the world, an alternative source of natural water is *seawater*. Most seawater is extracted from near shorelines, and therefore, as well as higher levels of salt in

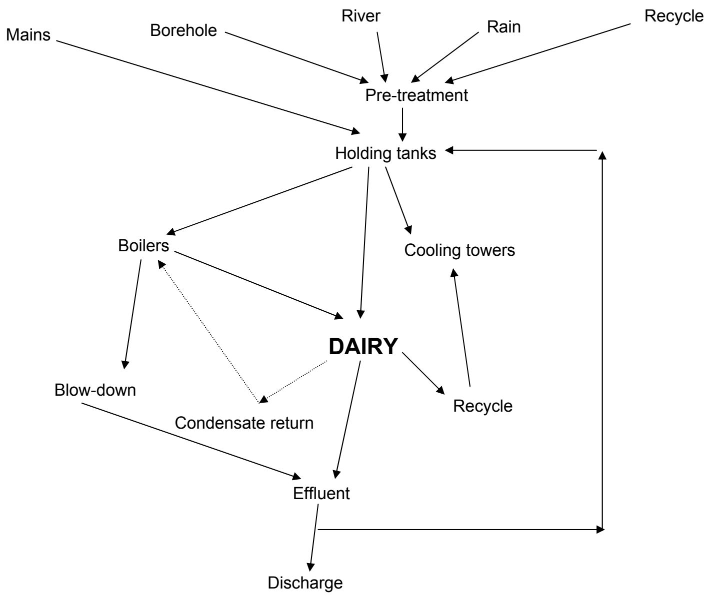

Fig. 3.1 Diagram showing water sources and applications in the dairy industry.

its composition, it can also contain more pollution from municipal and industrial run-offs. However, for countries with limited river or spring waters, this alternative is becoming increasingly more common.

Natural water is considered by many to be a cheaper option than water purchased from a local authority; however, natural water sources will require some form of treatment before use, and some examples of differences between surface water and groundwater sources are shown in Table 3.2.

In addition, if a factory is using surface water from a stream, then the types of contaminant can be very different from those from a well. This is because surface water tends to pick up organic materials such as farm and animal effluent, and in industrial areas these can even extend to chemicals. In well water, the surface water seeps through the various top layers of soil and rock, which act as a filter to remove any pollutants that may be present. However, as the water collects in underground aquifers, the level of naturally occurring salts in the water increases. Depending on the rock types that surround the aquifer, this could give a high level of different water salts. In general, if the total water salts are above 200–250 $\mu g\ g^{-1}$ (i.e. parts per million, ppm), then the water is considered *hard* (Haubry *et al.*, 1991). Examples of the elements that can be present in water are calcium, magnesium, sodium and potassium; these

Table 3.2 Main differences between surface and groundwater sources.

Characteristics	Surface water	Groundwater
Temperature	Varies with season	Relatively constant (10–15°C)
Suspended solids	Levels vary, but can sometimes be high (e.g. >100 $\mu g\ g^{-1}$)	Usually very low
Colour	Can be coloured owing to presence of suspended solids, such as clay	If any, it is due to dissolved solids
Mineral content	Varies with soil, rainfall and/or effluent	Largely constant and can reach high levels (i.e. >200 $\mu g\ g^{-1}$)
Carbon dioxide (CO_2)	Often high, especially in fast-flowing streams, but can be low in polluted waters	None
Hydrogen sulphide (H_2S)	None	Can be present
Nitrates	Normally low unless near fertilised farmland	Can be present
Silica	Usually present	Levels can be high
Mineral and organic micro-pollutants	Usually present	Usually none; sign of pollutant if present
Micro-organisms	Bacteria, viruses, plankton from animal and vegetable origins	Iron-utilising bacteria are frequently found
Chlorinated solvents	Rarely present	Often present
Eutrophic nature	Present if water is warm	Usually none unless from volcanic aquifer

Data compiled from Anonymous (2002).

elements are normally found in the form of ions, such as carbonates, nitrate or sulphates. These elements are not always considered to be problem, as many suppliers of bottled water use the naturally occurring minerals as a sales benefit to their consumers.

Treatment systems for natural water will vary, depending on the level and nature of the contaminants. The most common physical treatment systems are filters, demineralisation plants and reverse osmosis (RO). However, chemical treatment systems tend to be used for the removal of micro-organisms, and traditionally are based on some form of halogen treatment, such as bromine or chlorine.

One other source of natural water that can make a major difference to a dairy is *rainwater*. Although it is not usually captured and used directly, as dairies usually take up a large site area, the amount of rainfall can add substantially to the total amount of water leaving the site. Most sites will have two drainage systems:

- *foul drains*, which are the drains from production and auxiliary services, for example, vehicle washing and toilets; and
- *surface water drains*, the main contributor to which will be rainwater.

If a site has its own effluent treatment plant, then ideally the waste from toilets should be taken away by a separate drainage system; however, this is not always practical. The surface water drains from the production site should normally pass through a separate drain line straight off site. However, as for the toilet waste, in some cases it is not always possible to send the wastewater straight from site, and again it may have to be treated via the effluent plant route. In most European countries the local environment agency may well wish the production site to be self-contained, so that all water (from both foul and water surface drains) should pass through the effluent treatment plant within the site's boundaries. This is to ensure that, should a spillage occur – for example, from a milk tanker – then the site has facilities in place to contain the spillage.

3.2.2 *Authority-provided water*

The quality of the water utilised in a dairy or food factory should comply with national statutory standards or WHO standards for drinking water quality (see Table 3.3). Many of the treatment systems briefly mentioned above will be employed by the company to ensure that these standards are met. In the past, where the water delivered to a factory has failed, this tended to be due to poor distribution pipework, such as lead piping. Nowadays, most of the piping used is plastic.

Variations to water quality can occur where a water authority has several reservoirs to pull from, and in most food processing applications, this may not present a problem; however, in the case of one UK site in the brewing industry, changes in water hardness of more than 200 $\mu g\ g^{-1}$ occurred regularly overnight in a main site mains water storage tank. These changes were not particularly helpful when trying to ensure a consistent brew quality.

The hardness in water supply may affect cleaning efficiency, because some of the additives in the cleaning chemicals may be absorbed by the water hardness rather than removing soil(s) from the surfaces of the processing equipment. As a simple illustration, think of the scaling that can arise in a domestic kettle, if used in an area of naturally occurring hard water.

Table 3.3 Regulations concerning the quality of water intended for consumption.

Parameters	European Directive (guideline levels)	World Health Organisation (WHO) (recommendation levels)
Temperature (°C)	12	NR[a]
pH	6.5–8.5	6.5–8.5
Chloride (μg mL^{-1})	25	250
Sulphate (μg mL^{-1})	25	400
Calcium (μg mL^{-1})	100	NR
Magnesium (μg mL^{-1})	30	200
Sodium (μg mL^{-1})	20	NR
Potassium (μg mL^{-1})	10	NR
Nitrate (μg mL^{-1})	25	44
Hydrogen sulphide (ppb)[b]	Organoleptically undetectable	
Suspended solids (μg mL^{-1})	None	NR
Total bacterial count (cfu mL^{-1})		
at 37°C	10	NR
at 22°C	100	NR

[a] NR = not reported.
[b] ppb = parts per billion.
Data compiled from WHO (2004).

3.2.3 *Water from products*

When calculating a water mass balance for a site, it is worth remembering that some operations tend to actually contribute to the overall water volume being discharged from site. In the dairy industry contributions are made from operations such as evaporation of milk, especially where no condensate return system is employed, and also whey from cheese-making. In large cheese factories much of the whey will be used in other processes, such as evaporators, but at some point excess water from these additional processes will enter the main water discharge system.

3.2.4 *Water from recycling*

Not to be confused with water discussed in Section 3.2.3, this refers to wastewater and other effluent water that is recycled after processing to provide water for use in non-food areas, such as vehicle washing, cooling towers or boiler houses. If a factory has an adequate effluent treatment plant, then with minimal further treatment – filtering and/or chlorination, for example – recycling of this type of water can prove very cost-effective. For example, returns of up to 10% of the factory's daily site water (e.g. 60 000 L) might be available for external vehicle washing, boilers and cooling towers. At present this is a rare occurrence but, with continuing changes to legislation and environmental awareness, no doubt it will be a growing trend.

3.3 Improving water quality

Before water is used in a factory – or, indeed if it is to be recycled – its quality may have to be improved, especially if it is being used as an ingredient in the final product. Some of the areas where such water might be used are listed in Table 3.4, and Table 3.5 illustrates the questions to ask before proceeding with ordering suitable water treatment equipment. The objective of any installed system is to improve the water quality to meet a specified criterion: this is usually a higher specification than drinking water quality, especially if the water is required for inclusion as an ingredient, or for boiler water. There is, however, common equipment that can be used to meet these requirements. Also, there is some common terminology used in the water industry, which might be useful to a person trying to get to grips with the subject (see Table 3.6).

3.4 Equipment for improving water quality (coarse removal)

There are many specialist applications in improving water quality, but this review will highlight the most common physical separation systems used in the dairy industry. They

Table 3.4 Areas in a dairy plant where water is utilised.

Type of water	Application
Steam	Boilers Hot water sets
Heat exchangers	Cooling or heating of milk Steam condensate
Cooling towers	Cooling of water
Wash areas	Open plant cleaning Vehicle washing Tray cleaning
Cleaning-in-place (CIP)	Miscellaneous processing equipment/plant
Production area	Direct into products Reconstitution of powders
General	Toilets Canteens Hand wash stations Ornamental displays

Table 3.5 Intake of water treatment considerations.

Matters to consider	Comments
Source of water	Surface or ground
Water quality	Full seasonal analysis
Pressure	Minimum and maximum working pressures for pumping water around the factory or to a storage tank
Temperature	Surface varies, groundwater constant
Flow	Minimum and maximum demands from factory
Operation	Hours per day required
Storage capacity	Based on other criteria Could water become stagnant?
Footprint	Space available to build plant
Output quality	What/where water is being used?
Further treatment	Additional treatment at point of use
Regeneration	Will water be used once or recycled?
Budget	Capital and operating costs

Table 3.6 Selection criteria for water filtration units.

Particle size removal
Particle type
Suspended solids levels
Type of fouling
Flow range
Pressure loss
Liquid loss
Solids handling
Space required
Cost

may be used on their own, or in combination with other processes, to achieve the final result for the factory.

These systems tend to be for filtering out material in the water, and are predominantly used to remove visual particles, that is, larger than 1 µm. As with all equipment purchases within a dairy factory, there are selection criteria to be met (see Table 3.7), and these are very similar to those shown in Table 3.8.

3.4.1 *Screens and strainers*

Screens and strainers will remove materials such as leaves and other light debris from streams, usually greater than 50 μm in size. The limitations with the equipment tend to relate to the capacity and flowrates. The other main issue of concern is maintenance: the screens and strainers need to be regularly checked and cleaned by staff.

Table 3.7 Summary of size removal equipment.

Type	Material removed	Size (μm)
Strainers and screens	Coarse inorganic	> 50
Bags and filters	Fine inorganic	> 10
Sand filters	Very fine inorganic and some organic	> 1
Separators	Fine to coarse inorganic	> 10

Table 3.8 Parameters for recirculating water in cooling towers.

	Typical values for various water types		
Parameters	Soft	Hard	Acid dosed (hard)
pH	7.5–8.5	7.5–8.5	6.0–7.5
Alkalinity	1000	700	700
Calcium balance (%)	90–100	90–100	90–100
Conductivity[a]			
Chloride ($\mu g\ Cl\ g^{-1}$)[b]		300 (max.)	
Concentration cycles achieved by tower[c]			
Scale/corrosion inhibitor reserve[d]			
Suspended solids[e]		50 $mg\ kg^{-1}$	
Langelier saturation index (LSI)	−0.5 to +1.5	+2.5 to +3.0	Note[f]

[a] Conductivity is measured in microsiemens (μS), and it is normally limited. However, as a guide, the system might be kept < 1500 μS to prevent excessive recycling of water, which could result in scaling of the system.

[b] Although it is desirable to have a system showing a small amount of chloride, the level should be kept <200 $mg\ L^{-1}$ for stainless steel, as corrosion may set if these levels are regularly exceeded.

[c] To minimise wasting water and chemicals, the tower system should be recycled (target six times); then the tower system is completely replenished, and the limiting factors to this are corrosion and scaling to the system.

[d] A wide variety of inhibitors products are available to prevent scaling and corrosion on the tower; each supplier and each chemical has its own method of analysis and control, e.g. alkalinity or sulphite levels.

[e] If the level of suspended solids rises, this is usually the result of poor housekeeping and maintenance, i.e. from the structure of the tower and scale/slime formation.

[f] The LSI is a measurement of how likely the water is to scale or corrode the tower; if the water has natural hardness (e.g. calcium), it will have a natural barrier to corrosion.

3.4.2 *Bag and cartridge filters*

Bags and cartridges are of a similar nature to screens and strainers, and the operating principle is the same – to remove large particles from the water. They are generally used in in-line filtration systems, which are a familiar sight in most farm and dairy intake bays. This water filtration system uses a combination of a stainless steel case with an inner cloth filter to take out coarse material. Like screens and strainers, the capacity of bag and cartridge filters is limited, as they are influenced by similar flowrate and maintenance issues. They are slightly more efficient, though, as they can be sized to take out particles down to 5 μm, although the most common filters tend to be in the region of ~10 μm. In most large dairies the use of these filters might also be a requirement of the customer, to ensure the removal of *Cryptosporidium* spp.

3.4.3 *Sand-type filters*

The next level of filtration for incoming water would be another physical barrier provided by sand-type filters. There are several types available on the market: some use anthracite sand; others use absorption by activated carbon. Manganese dioxide might also be used on top of the sand to act as a catalyst in the removal of iron ions from the water. With these systems, the level of removal tends to be between 5 μm and 10 μm. Again there are flow-related issues, and these filters tend to be limited to 10 m^3 h^{-1} m^{-2} of surface area: this normally has a bearing on the footprint of these systems when such units are to be incorporated into an existing water treatment programme. The other limiting factor is that there is a pressure drop across the filtering unit (~0.05 MPa), and this will have to be taken into consideration when sizing the filtration system. In a refinement to these system, an ultra-fine medium might be used, and these units tend to be positioned in series to ensure that the treatment removes manganese dioxide. Generally, these filters rely on pH adjustment to ensure that manganese and iron ions are captured, and usually the medium used would be a chloride base or lime, such as calcium hydroxide.

If any of these systems is to be used, the materials of construction need careful consideration. Although stainless steel might look attractive in dairy applications, if used in this area, the back-flush cleaning operation on these units can damage the stainless steel piping, and a safer option would be glass-reinforced plastic (GRP).

3.4.4 *Separators*

These types of system tend to be used only for the removal of coarse solids, and their separation efficiency can sometimes be improved by the use of a polymer, which will help to bind the solids together and assist in the removal of smaller particle sizes. In general this type of filter is used only where higher volume throughputs are required, and as a preliminary stage in solids removal, for example any particle(s) greater than 10 μm. The other main problems with separators tend to be their energy costs to operate, the associated noise, their initial capital cost, and their maintenance. This last aspect is a particular problem, as the separator bowl is constantly removing grit and other harsh materials from water.

3.5 Equipment for improving water quality (fine removal)

3.5.1 *Softeners*

Softeners tend to be best associated with the pretreatment of boiler water. They provide a very simple yet effective method of improving water quality. Their primary function is to remove naturally occurring salts in water. In some areas these tend to be calcium and magnesium salts, with levels that can be >200 $\mu g\ g^{-1}$ (Table 3.2). The incoming water passes through the softener, which is filled with a strong acid cation exchange, normally in the form of common salt (i.e. sodium chloride). The ion exchange removes virtually all the water hardness. Although these units are associated with water treatment for boilers, in some areas this treatment could be used in production, for example where water is used as an ingredient.

3.5.2 *Reverse osmosis*

Membrane filtration of water is used in the dairy industry to produce desalinated water for applications such as boiler water, cooling tower make-up water, and when water is used as an ingredient. The advantage of reverse osmosis (RO) plant over the softener method is that it removes particles down to 0.001 μm, and also assists in the removal of micro-organisms and toxins.

The basic principle of this system is that it will separate out the saline part of the water. The purified water (called the *permeate*) will pass through the membrane, and the concentrated saline solution (called the *reject* or *retentate*) is left behind. As is shown in Figure 3.2, the rejected water can be reworked to assist in the filtration efficiency of the RO system.

3.5.3 *Electro deionisation (EDI)*

This is rarely used in the diary industry. These units use a combination of ion-selective membranes and ion exchange resins placed between two electrodes under a d.c. voltage potential. The objective is to remove ions from water that has been pretreated with RO. Water processed in this manner is known as *ultra-pure*: dairy plants that might use this type of technology would be those producing high-value or high-margin products, such as baby milk powders.

3.6 Applications of water in the dairy

3.6.1 *Water as an ingredient*

Water is used extensively as an ingredient in the dairy industry. For example, most large-scale production sites will use milk or whey powder as an ingredient that is brought in, rather than having one large site that processes liquid milk, and then uses it in a variety of applications. A typical example of this approach would be in yoghurt manufacturing. Much of the work relating to this is dealt with elsewhere in this publication, and therefore it will not be reviewed in this chapter, but it is worth emphasising that any water that is reused must be to a high quality, and should be checked on a regular basis.

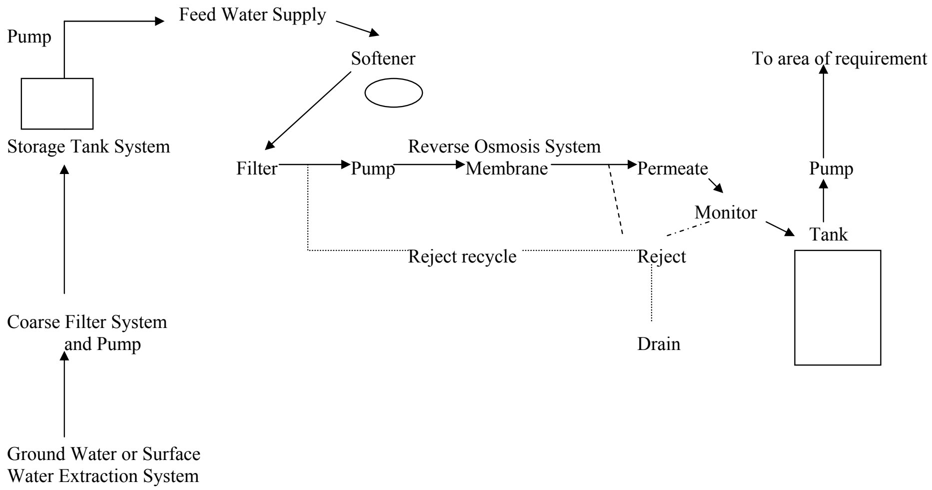

Fig. 3.2 Diagram showing areas of application for water purification systems.

3.6.2 *Water as a cooling agent*

Most dairy products – apart from cheese – are classed as short life, and therefore will need to be kept cool after processing to prevent the build-up of micro-organisms. Some dairies will use ammonia or other gases as a refrigerant; however, the other common process used is a *cooling tower*. The tower is usually constructed as a loop system (Figure 3.3), where water will be used as coolant within the factory, for example as the cooling fluid in a plate evaporator. After use, it passes out of the factory, either directly or indirectly, to the cooling tower; this cools the water down before it is reused in the factory or, in older systems, before it is discharged from the dairy.

Because of the heat of the water coming out of the evaporator, some of the water evaporates to the atmosphere, and this is normally topped up from mains water. The efficiency of this system is measured in terms of cycles: this is based on the amount of water in the system, and how often it is totally replaced. In this method of water cooling, the desired aim is a total of six cycles, but the amount will vary depending on the season: in winter the efficiency tends to be greater owing to the colder ambient climate.

Because of the evaporation and aerosols generated in this system, chemical treatment is normally applied (e.g. biocide) to help prevent bacterial build-up: in the worst cases, if not treated correctly, this can be seen as slime. More importantly, chemical treatment is used to control viruses such as *Legionella* spp. *Legionella* was first identified in 1976 in an outbreak in Philadelphia in the USA amongst delegates attending an American Legion convention – hence the name. Surprisingly, there is no legal requirement in the USA to take measures to control *Legionella*, but most of Europe has adopted the UK government's guidelines, which are published by the Health & Safety Commission (Anonymous, 2000). These guidelines, termed 'L8', outline the requirements for all water systems used in a dairy – not only the cooling systems, but also hot and cold services, such as showers and sinks – along with guidelines as to the frequency of monitoring and cleaning of water systems. They are impor-

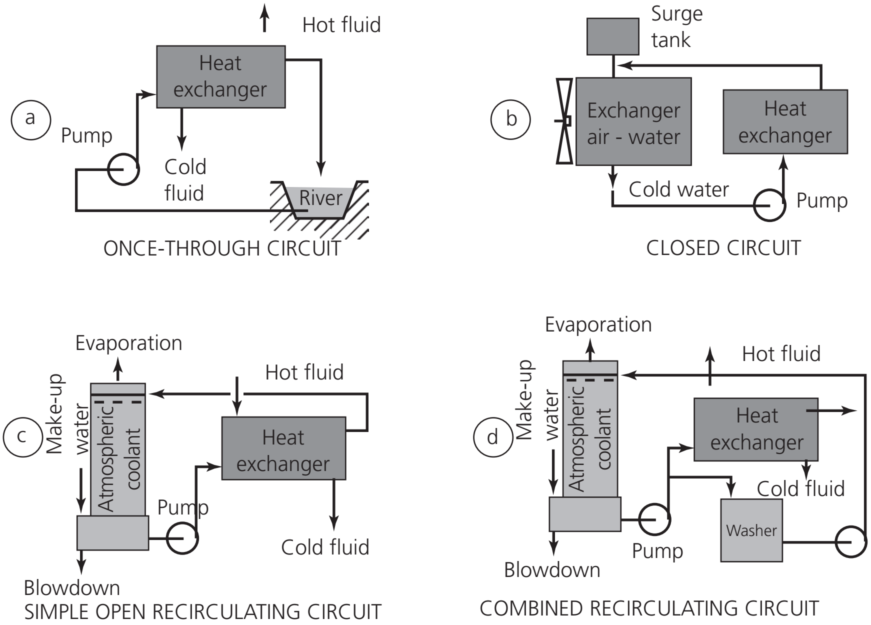

Fig. 3.3 Different types of cooling tower. From Anonymous (2007) and reproduced with permission from Degremont, Group Suez, France.

tant guidelines: as the attached report shows from an outbreak in the UK, the consequences of *Legionella* infection can be quite tragic (see UK Health and Safety Executive (HSE) Legionnaires website at www.hse.gov.uk).

Because of the water evaporation from the cooling tower, the concentration of solids in the tower may increase, and sometimes, because of the absorption of atmospheric carbon dioxide (CO_2), these solids can become acidic. Therefore it is sometimes beneficial to add other chemicals as well as biocides to help control corrosion and scale. To ensure that the system is working efficiently, it should be monitored regularly; simple tests, such as conductivity of water, give a rapid assessment of the build-up of solids. Total bacterial count (TBC) using a dip slide is also a good indicator, although with this method of analysis there is a time delay (two days at room temperature). A guide to the frequencies for water testing of a cooling tower is shown in Table 3.9.

3.6.3 *Water in heating applications*

Water for boilers

As with cooling water, water designated for boilers needs to be treated to prevent damage to the system from scaling and corrosion. In a boiler, the treated water is converted to steam, which can then be carried around the factory for a variety of applications, such as office heating, use as heat transfer medium in a plate heat exchanger or evaporator, or to maintain heat in a jacketed vessel, such as a cheese vat.

Table 3.9 Frequencies for water testing/specifications for water towers.

	Duration	
Parameters (mg L^{-1})	Make-up water	Cooling water
Calcium hardness	Monthly	Monthly
Magnesium	Monthly	Monthly
Total hardness	Monthly	Monthly
Total alkalinity	Quarterly	Quarterly
Chloride level	Monthly	Monthly
Sulphate level	Quarterly	Quarterly
Total dissolved solids (μ)	Monthly	Weekly
Suspended solids	Quarterly	Quarterly
Inhibitors	–	Monthly
Oxidising biocides	–	Weekly
Temperature (°C)	–	Quarterly
pH	Quarterly	Weekly
Soluble iron	Quarterly	Quarterly
Total iron	Quarterly	Quarterly
Concentration factor (i.e. number of tower recycles)	–	Monthly
Microbiological count	Quarterly	Weekly
Legionella spp.	–	Quarterly

Boilers come in a variety of sizes, depending on the demand for steam in the dairy – small dairies may even use a steam generator – but the basic principle is the same in all cases. When the water is converted to steam and is taken off as vapour, the water left behind in the boiler becomes more concentrated, because the solids in the water are heavier, and therefore are not evaporated as vapour. On most modern boilers, the level of *total dissolved solids* (TDS) is monitored automatically: once the TDS reaches a predetermined figure, the boiler automatically goes into a programme called *blow-down*. This removes the heavier solids in the water, and the boiler is then refreshed with additional water, either from condensate return (that is, returned boiler water from the factory) or from fresh water.

Water for use in boiler applications needs to be pretreated to remove as much of the solids and dissolved oxygen as possible. Normally the solids are removed by a softener unit. The cation exchanger, regenerated with sodium chloride (salt), removes the calcium and magnesium hardness from the water. If borehole water or rainwater is being used, it might be necessary to remove metals, such as iron and manganese. In most cases demineralisation of water is used, particularly in high-pressure boilers; this also helps to remove silica, which is found in nearly all natural water. Finally, it is important to remove any gases that might be present, particularly oxygen, in order to prevent corrosion of metal equipment. In most cases this can be done by the addition of chemicals known as *oxygen scavengers*. Alternatively, de-aeration of water might also be employed. Suggested requirements for water quality for industrial boilers are listed in Table 3.10.

Having taken a lot out of the water, it is then conditioned by the addition of other chemicals, such as phosphates or polyphosphates. These compounds react with the alkalinity of the boiler water to form heavy insoluble compounds, which can be taken out in the blow-down procedure. Natural and synthetic polymers might also be used as they help to increase the dispersion of the phosphates when applied. Furthermore, in some cases anti-foam might also be used to prevent the carry-over of fine water into the steam.

Regular checks have to be made, at least daily, on a boiler to ensure the water quality is maintained. Normally, the TDS and alkalinity are checked daily to ensure that the water quality is maintained: this provides an early indication if there are any problems in areas such as the chemical addition or automatic TDS blow-down valves, thus preventing scale or corrosion.

Water as condensate return

When the steam vapour has been around the dairy, if it does not pass out into the atmosphere it can be returned to the boiler house: this is termed *condensate return*, and it is estimated that steam gives up 610–670 kcal kg^{-1} of water as it condenses (Anonymous, 2002). However, it is still valuable and, if possible and practical, it should be reused not only for its heat value, but also because it does not need too much treatment as it will be free from the contaminants that are found in raw water. Another source of condensate in the dairy industry is evaporators: in a larger system, such as a seven-stage evaporator, it is estimated that as much as 12 kg of water can be evaporated from milk for every 1 kg of steam used; the concentration of skimmed milk solids in such an evaporator rises from 8–10 g 100 g^{-1} to 50 g 100 g^{-1} (Anonymous, 2003). Such condensate would require some form of treatment, normally to correct the pH (i.e. lactic acid formed by the metabolic activity of micro-organisms), and this could be achieved by introducing a neutralising or film-forming amine. These types of product would also help to protect the pipework and tanks used to transfer and hold the condensate return.

3.6.4 *Water for general use*

Throughout the dairy, there will be many areas where water is used so routinely that it is almost unnoticed, such as wash stations prior to entering the process areas, toilets, and canteens. Many factories will operate control systems to flush areas such as toilet bowls and urinals; however, it is estimated that for every person on site the average consumption of water per day associated with the site services will be in the region of 10 L (Welsh Water website www.welshwater.com). This can increase dramatically if taps are left running, or pipework is leaking. For example, if a tap is left dripping and loses water at 1 L min^{-1}, this equates to 1.4 tonnes of water per day.

Table 3.10 Suggested water quality for boilers[a].

	Operating pressure (MPa) [b]							
Parameters (mg L^{-1})	0.2–0.7	2.08–3.10	3.11–4.14	4.15–5.17	5.18–6.21	6.22–6.89	6.90–10.34	10.35–13.79
Feed water								
Dissolved oxygen (measured before scavenger addition)	< 0.04	< 0.04	< 0.007	< 0.007	< 0.007	< 0.007	< 0.007	< 0.007
Total iron	< 0.1	< 0.05	< 0.03	< 0.025	< 0.02	< 0.02	< 0.01	< 0.01
Total copper	< 0.05	< 0.025	< 0.02	< 0.02	< 0.015	< 0.015	< 0.01	< 0.01
Total hardness (i.e. $CaCO_3$)	< 0.3	< 0.3	< 0.2	< 0.2	< 0.1	< 0.05	ND	ND
pH (at 25°C)	7.5–10.0	7.5–10.0	7.5–10.0	7.5–10.0	7.5–10.0	8.5–9.5	9.0–9.6	9.0–9.6
Chemicals for system protection	Use only volatile alkaline material							
Non volatile (total organic compounds, TOC)	< 0.1	< 0.1	< 0.5	< 0.5	< 0.5	< 0.2	< 0.2	< 0.2
Oily matter	< 0.1	< 0.1	< 0.5	< 0.5	< 0.5	< 0.2	< 0.2	< 0.2
Boiler water								
Silica	< 150	< 90	< 40	< 30	< 20	< 8	< 2	< 1
Free hydroxide alkalinity (i.e. $CaCO_3$)	Not specified	Not specified	Not specified	Not specified	Not specified	Not specified	ND	ND
Specific conductance ($\mu\ cm^{-1}$) at 25°C	< 3500	< 3000	< 2500	< 200	< 1500	< 1000	< 150	< 100

[a] Industrial water-tube, high duty, primary fuel fired and drum type; values in table assume existence of a de-aerator.

[b] With local heat fluxes > 473.2 kW m^{-2}; use values for the next higher pressure range.

ND = not detected.

3.6.5 *Water for cleaning purposes*

Water is the commonest form of cleaning material used in the dairy industry. From open plant cleaning to cleaning-in-place (CIP), the main medium for carrying the chemicals used will be water. Most chemicals are used at concentrations between 1 and 5 g or 1 and 5 mL per 100 mL, and therefore the rest of the make-up will be from water. The principles of CIP are reviewed in Chapters 1 and 2. As with water for general use (see Section 3.6.4), water in these areas can easily be lost, despite sites having trigger hoses and automated dilution systems. Most people can recall walking through a dairy where either hose pipes are running at full bore so the floor 'looks nice and shiny', or the workers, knowing a visit is due, have disappeared for another break and have tied the trigger hose so that it is running full bore into the drain.

3.7 Water leaving the dairy

Most water leaving a dairy will undergo some form of treatment, either on site or via a local water authority. Exceptions to this might occur if the site has a separate surface water run-off system and this water has capturing areas, such as the roof space and the ground, and is allowed to run off without any treatment. There are some plants that take in water as a cooling medium from rivers or canals, and this water might be allowed to discharge directly back into the water source without treatment. In both these examples it would be prudent to check the water quality regularly to avoid inadvertent pollution of the local environment. Increasingly, though, sites are being required to treat their wastewater streams, as environmental pressures grow on industry to 'clean up its act' (UK Government website on legislation to clean-up pollution – www.ofwat.gov.uk), and some examples are reviewed in subsequent sections.

3.7.1 *Minimum treatment*

The minimum treatment that a processing site may undertake for the water leaving its premises is – absolutely nothing. Some sites, by their very nature, produce waste that might be seen as beneficial to a local effluent plant, especially if the dairy makes added-value products, such as yoghurts containing sugars: this would help to encourage the bacterial growth in a sewage treatment plant. There are problems with this approach, though: most dairy plants have some levels of fat in the wastewater, and in the case of cheesemaking the acidic whey can block and corrode the pipelines as it passes from the dairy to the treatment plant. Therefore it is very unlikely that this approach would be followed. Not only will the system eventually pollute the local environment, but the site will probably also end up with an expensive regular bill for the removal of the solids that are generally associated with dairies.

3.7.2 *Buffering of wastewater*

The easiest treatment undertaken by a dairy operation is to use *interceptors* or *balancing tanks*: these units help to buffer out extremes of pH from the variety of operations within the dairy. Traditionally, in a cheesemaking operation, milk is collected and then stored overnight in silos, from where it is processed the following day. After the milk has left the silo, these units are cleaned, usually with an alkaline detergent, and the pH of the wastewater from this area would tend to be 8–10. As the cheese is produced in the vats, the whey drained off is acidic (pH 4.5–6). If the two wastewater streams are then combined by the buffering capacity of an interceptor or balancing tank, the overall effect is for the wastewater that finally leaves the site to be within discharge parameters, normally pH 6–10. This simple technique has other benefits, too. It can dramatically reduce the amount of chemicals used to correct the final pH of the discharge water to ensure that it is within consent. It also allows any fat to separate out, for subsequent waste disposal. In the past, this fat might sometimes have been sold for its calorific value: for example to feed grain producers, for use as a binding agent in animal feed products.

The problems with treating site wastewater by this method stem from the fact that production in the dairy tends to have peaks and troughs. At peak times the interceptors cannot cope, and the wastewater is not treated correctly. Also, there is a problem with the smell, especially if the fat is not removed on a regular basis. The fat blanket leads to anaerobic conditions, which encourage the growth of bacteria that produce obnoxious gases, such as hydrogen sulphide. The emission of these gases leads to problems for the people working in the area, and can also taint the product. If there is a peak rush of water through the system, this could lead to a pollution problem in the local environment.

3.7.3 *Effluent treatment*

Most dairy sites, driven either by legislation or by concern for the environment, undertake more complex treatment of wastewater before their water is discharged. There are many variations, but the most common system comprises the following elements.

Initial screen

This removes any large particles that may go down the drain, such as hairnets. There are a variety of screens on the market, with a range of screen mesh: most sites would opt for a screen of 1 mm for the coarse material, and there may be a second unit for finer material. Most such screens incorporate a wash system, because by the nature of dairy waste it tends to be fatty, and can start to smell and block. This part of the treatment of the water needs regular checking to ensure that there are no blockages, and to alert production if anything untoward is seen. In some sites, once a treatment system is in operation the drains become a magnet for anyone not wishing to comply with good housekeeping (hairnets or plastic bags are common place); rather than spend a few minutes collecting waste and putting it in a suitable container, people will sometimes spend 5 minutes with a hosepipe on full, chasing a lump of cheese around the dairy floor until it goes down the drain.

Balancing tanks

Ideally, these should be able to hold one day's worth of wastewater, and to assist with the buffering of the pH level. They should also incorporate some method for keeping the contents agitated, such as a rotating paddle(s) or a circulation pump. Agitation will prevent the fatty material from separating on the surface of the wastewater, which would otherwise induce anaerobic fermentation. To prevent the emission of smells, these tanks are normally enclosed, although to keep costs down the tanks may sometimes be open, which does at least allow the opportunity to ensure that there are no dead spots occurring within the tank system. If there is a fairly standard waste stream from the factory for the wastewater, then some plants undertake the pH correction at this point.

Phase separator

From the balance tank, the next stage is usually to undertake some form of solids removal. This separates out the solids from the aqueous phase, and chemical treatment is normally used to accelerate the reaction. This might be via an inorganic route using a metal, such as aluminium or iron, to capture the solids, or using an organic material, such as tannin, a wood-based material. These chemicals are generally termed *coagulants*; usually, the system is further assisted by the use of a polymer.

The addition of the coagulant, normally into a turbulent flow to assist the mixing of the chemical, brings about chemical reactions in which the positively charged coagulant reacts with the negatively charged solids in the milk (see Figure 3.4). As a result the suspended solid particles come together and 'pinpoint flocs' start to appear. The solids start to separate out from the liquid phase (Figure 3.5). Flocculation follows the coagulation stage, and consists of gentle mixing; the flocs continue to grow in size into a more defined solids phase, so they can be easily separated from the liquid phase. During this stage of the effluent treatment, the

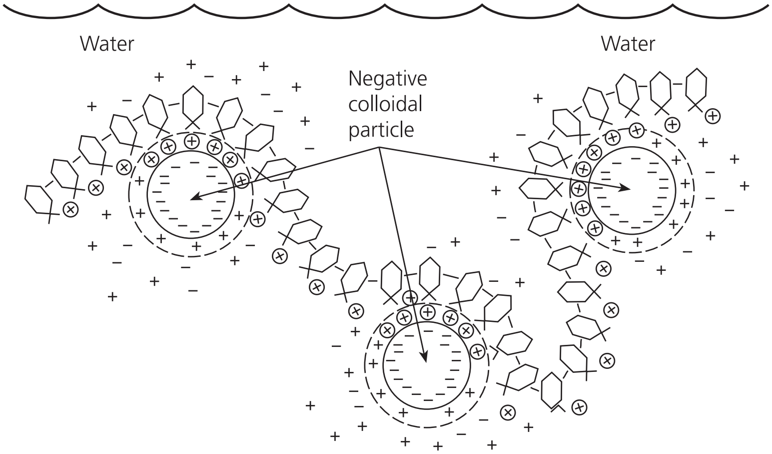

Fig. 3.4 Charge reaction of coagulant in wastewater. Reproduced with permission from Hydro International, Navan, Ireland.

Fig. 3.5 The effect of adding coagulant and polymer during the treatment of wastewater.

polymer is added to aid in the binding of the small suspended particles: this accelerates the production of larger ‘flocs’ that are easier to remove by a suitable physical process.

Other chemical treatment methods used to remove the solids from wastewater in the diary industry include *acid cracking*. This is one of the oldest forms of wastewater treatment that exists. An inorganic acid (nitric or phosphoric) is used to reduce the pH to 3. At such a low pH the solid materials split or break up. The pH is then corrected with sodium hydroxide (caustic soda) or calcium hydroxide (lime), and the solids bind together. Again, a polymer might be used to accelerate this process.

Chemical processes such as those described may need to be assisted by a physical system, and the most common examples are *dissolved air flotation* (DAF) and *cavitational air flotation* (CAF). These units introduce air under pressure into the wastewater as very small air bubbles. As the wastewater enters the treatment unit, the solids are lifted to the surface by the air bubbles (Plate 1). They are then separated from the water, which flows from the plant and can then be:

- discharged from site
- recycled to the factory
- reused as ‘grey water’ return to the DAF unit
- treated further

3.7.4 *Wastewater treatment*

Discharge from site

This is the commonest procedure in handling wastewater from liquid milk processing sites. It is good practice to have some form of pH control on the final discharge point to act as a ‘policing’ system, and to ensure that, if anything goes wrong in the processing plant, it can be picked up at the discharge point. In addition, the site might wish to monitor the flow volumes, and provide a facility to take wastewater samples either for testing by the site laboratory or for collection by the regulating authority.

Recycle to the factory

If the site can ensure that the wastewater quality is satisfactory (see previous section), the addition of a turbidity system to monitor the clarity of the wastewater may offer an opportunity to recycle it. This water tends to be used in external applications, such as vehicle or crate washing. It may also be used in cooling towers or boilers, in which case it may require further treatment (e.g. using sand filters) to ensure its quality.

Recycle as ‘grey water’ to effluent plant

Some DAF plants have the facility to recycle a small volume of the clean wastewater (up to 10%; Hydro International website www.hydrointernational.ie), which is then mixed into the pressurised air unit (see ‘Phase separator; in Section 3.7.3 above) and blown into the stream of wastewater coming into the treatment plant. This extra pressure applied assists in helping to blow the solids to the top of the DAF cell (Figure 3.6).

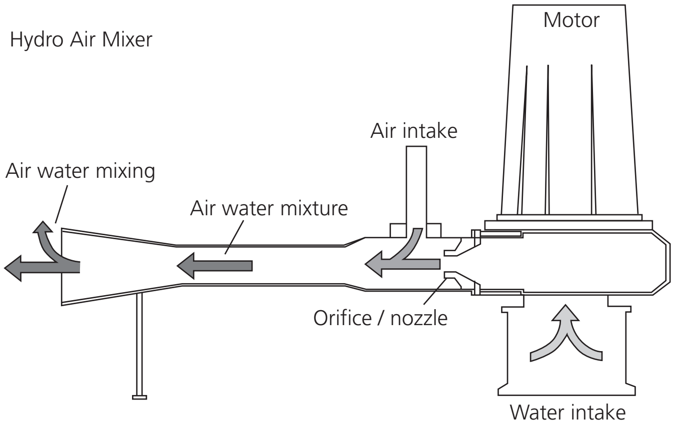

Fig. 3.6 Hydro air mixer/blower used in effluent plants. Reproduced with permission from Hydro International, Navan, Ireland.

Further treatment

The above treatment methods remove much of the solids, but the dissolved solids still remain in the aqueous phase. Examples of these include sugar and colouring matter. It is extremely difficult to remove these, and this cannot be achieved with chemical treatment of the wastewater. Therefore, in some dairy plants, a further stage of wastewater treatment is required, in the form of biological treatment. If the site has the capacity, and produces added-value products (e.g. yoghurts), then it may have a biological treatment plant as well, which removes the dissolved *chemical oxygen demand* (COD) from the water.

Two types of biological treatment of wastewater are available on the market. The *aerobic* type uses large amounts of air, which is blown into the water to encourage rapid bacterial growth. The *anaerobic* type relies on specific bacterial growth to produce methane gas. The gas produced is of value, as it can be used as an energy source for the boilers on site. Typical systems using this type of anaerobic wastewater treatment might save 15% of the annual energy bill of a factory (Veolia website www.veolia.com).

The most important factor for biological treatment is to keep the pH as consistent as possible. If the wastewater is undergoing any pretreatment, or is being sent straight from a production area to the biological treatment plant, it is important to maintain its pH, because the first step in anaerobic digestion is the production of acid-forming bacteria. Without buffering and control of the pH of the wastewater, the acid-forming bacteria may increase and become dominant, causing the anaerobic system to ‘turn sour’ and not function properly.

A biological treatment system is a ‘living’ system, and therefore needs more control. Seasonal changes might upset the system, particularly the air temperature around the plant. If the temperature of the wastewater from a factory is relatively steady and the air surrounding the plant is extremely cold, this may cool down the biological treatment plant so that the microbial activity of the system will be reduced. Ideally, the plant should be run in the region of 20°C. Also, the treatment plant should have a wide range of micro-organisms, which are able to deal with the varying dissolved solids in the wastewater. A typical count is in the range of $1–38 \times 10^6$ colony-forming units (cfu) mL^{-1}, and examples of some micro-organisms and their role in a treatment plant are shown in Table 3.11 (see also Horan, 2003).

Biological treatment of wastewater further improves the quality of the water and, as already mentioned, some processing sites might to be able to recycle the resulting wastewater, particularly in areas that do not come in direct contact with the food (e.g. for boilers, cooling towers or vehicle washing). This area of water treatment is of increasing interest to sites as the cost of water supply and discharge continues to increase. Some dairies will pay twice for the water they need on site: once to receive the water from the local water authority in the first place, and then again to discharge the water into the local sewage treatment system. The cost of water continues to increase in the UK; prices vary widely between the regions, but the average cost for industrial sites receiving water in 2006 was 70p t^{-1} (€1 t^{-1}) (www.ofwat.gov.uk). In the UK the discharge cost of water is calculated using the *Mogden formula*. A charge is set by calculating the cost to discharge water from the site to the local sewage works and for processing the water to a desired quality. A typical discharge cost of water in the UK will be £1 t^{-1} (€1.40 t^{-1}), even if the water is ‘clean’, so that the overall true cost of water becomes £1.70 t^{-1} (€2.50 t^{-1}). If the wastewater from a dairy site could be recycled, the cost of the water treatment would be in the region of £100 000 (€140 000),

Table 3.11 Micro-organisms commonly found in effluent plants.

Type of micro-organism	Comments
Aeromonas spp.	Main organisms present in anaerobic systems; useful for reducing lactose to acetate, e.g. lactate + $H_2O \rightarrow$ acetate + $2H_2$ + CO_2 + x kJ energy
Methanobacterium spp.	Responsible in anaerobic systems for further breakdown of acetate to produce methane
Bacillus spp.	Help to break down protein
Pseudomonas spp.	Help to break down carbohydrates and assist in denitrification
Nitrosomonas and *Nitrobacter* spp.	Used for nitrification of wastewater. Sensitive to environmental change, and can quickly be wiped out. Important in reducing ammonia to nitrate compounds and eventually nitrogen, e.g. $NH_4 + 2O_2 \rightarrow NO_3 + 2H + H_2O$ $2NO_3 + O_2 \rightarrow 2NO_2 + 2O_2$ $NO_3 \rightarrow NO_2 \rightarrow NO \rightarrow N_2O \rightarrow N_2$
Algae	Tend to form in low-flow conditions or long periods of sunlight associated with poor conditions if found; normally seen as a slime

based on the site discharging 250 t d^{-1}. However, the cost saving would result in a payback in under 9 months when compared with the cost of using the wastewater treatment provided by the local authority.

3.7.5 *Problems associated with biological treatment plants*

Micro-organisms

As well as the desirable micro-organisms systems present in the wastewater, sometimes there are unwanted microbial species that can become dominant. An example is the filamentous (long and thin) bacteria. If their count exceeds 1×10^6 cfu mL^{-1} they can cause a problem known as *sludge bulking*, whereby there is a loss in the solids' ability to settle when it is formed in the biological system (Horan, 2003). The concentration of filamentous bacteria in the treatment system may be influenced by husbandry management of the dairy herd, when the cows' feed is changed from winter silage to fresh grass.

To correct the problem of sludge bulking, chlorination of the wastewater is recommended, but if it is not properly managed, it can wipe out the entire microflora in the biological treatment plant. Chlorination needs careful management; ideally, the system should be isolated and a small amount of sodium hypochlorite should be added. A typical recommended level of sodium hypochlorite in the biological treatment plant ranges between 5% and 14%, which is equivalent to 1 μg free chlorine mL^{-1} in the plant (Horan, 2003).

Microbial nutrient deficiency

In most dairy plants, nutrient deficiency in the treatment wastewater is not a problem, because the nature of the waste from the factory – for example dissolved sugars (natural or added) and fruit from a yoghurt plant. The fruit residues may cause a problem because of the high level of carbon: excess carbon will ultimately reduce other nutrients, and mainly the nitrogen to phosphorus (N:P) balance. If the N:P balance is too low, then the bacteria that do not require much of these nutrients, such as the filamentous organisms mentioned earlier, will become dominant in the wastewater. An ideal N:P balance is 5:1. To correct this balance, a fertiliser is normally brought and spread onto the system, or mixed into a liquid and then applied into the system. This approach helps to redress the balance of nitrogen and phosphorus in the wastewater.

Low organic loading

In the dairy industry this should not present a problem, thanks to the nature of the wastewater but, if it *does* occur, the growth of filamentous bacteria might be an indication. To overcome this problem, some of the bio-sludge from the system is returned to the inlet of the unit, which will then redress the organic balance in the wastewater. Some plants bring in sludge from other sites, including domestic sewage treatment plants; sometimes, because of the microbial make-up of this sludge, it can take a couple of weeks for the system to recover fully as the bacteria adjust to the wastewater entering the biological treatment system. An alternative approach is to buy a mixed, freeze-dried culture of micro-organisms from a reputable distributor, who will have a range of these preserved organisms (perhaps found in a dairy effluent plant) that can be used to re-seed the system.

Low oxygen level

This can be a problem for dairy plants, as the oxygen level is controlled by mechanical means. Usually, the oxygen is supplied by large air blowers, which need to be sized according to the tank size. A recommended level for oxygen in a biological treatment plant is 1–2 mg L^{-1} (Horan, 2003). A low oxygen level in the system promotes the growth of filamentous bacteria; also, anaerobic bacteria can proliferate. If these organisms become dominant, there is a risk of anaerobic gases forming, such as hydrogen sulphide (H_2S). The danger is that the gas can develop in pockets, and it is toxic at very low levels (< 10 μg mL^{-1}). The most common way to check that the oxygen level is being maintained is to test for the amount of dissolved oxygen, and this is a fairly standard test (Gilbert *et al.*, 1982).

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4 Chemistry of Detergents and Disinfectants

W.J. Watkinson

4.1 Introduction

The objective of this chapter is to provide the reader with a better understanding of the use of detergents and disinfectants, so as to be able to make an informed decision on which materials to use, or be better able to evaluate competitive product offerings against each other. It is hoped that a better understanding of why detergents are used, which ones to use where, their importance, and how they can affect the overall hygiene of a dairy will be better understood. For disinfectants, the selection criteria are given by considering the differing performance of the active ingredients, and which disinfectants are best suited for which applications within the dairy industry.

4.2 Why do we clean?

In order to understand detergency, how detergents are formulated, how they work, and the other technical aspects of detergents, we need to examine the reasons why objects are cleaned, whether these objects are ourselves, our clothes, our possessions, or industrial items.

4.2.1 *Appearance*

Appearance is the most obvious reason for cleaning, but it is not the easiest to explain. From the customer's point of view a clean appearance gives confidence – for example, the tableware in a restaurant or in the food processing industry. A clean and tidy appearance gives operators pride in their equipment, and they are happier working in a clean environment than in a dirty one. In a plant that is maintained in a clean condition, soiling will quickly be seen to be out of place.

4.2.2 *Micro-organism contamination*

Cleaning alone is no guarantee that articles are necessarily free of contamination from micro-organisms. It is, however, very much easier to disinfect a clean surface than a soiled one. Disinfection will be dealt with separately later in the chapter.

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4.2.3 *Plant efficiency*

Dirty plant does not operate efficiently. Scaled heat transfer surfaces are slower to alter the temperature of the solution being treated, leading to lower production throughput and increased energy usage for the same amount of production. A good example of these surfaces would be in pasteurisers. Similarly, scaled conveyors or excessively dirty conveyors are unnecessarily heavy and need more power to move them.

4.2.4 *Safety*

An accident in a factory can have expensive repercussions, as well as causing absenteeism and bad feeling amongst employees. Dirty equipment is often slippery and dangerous. Greasy floors and stairs are treacherous, greasy utensils can slip from hands, and forklift trucks can skid on wet or greasy floors. Therefore a clean environment makes the working conditions much safer.

4.3 Soil to be removed

Soil is any material that is out of place. In food factories it is usually food residues or water scale. The nature of the soil will determine the correct chemistry and processes to use to remove it. Soils can initially be divided into two basic types: those that are water soluble and those that are insoluble in water. *Water-soluble soils* such as sugar and salt are readily dealt with and present few, if any, difficulties in their removal. *Water-insoluble soils* present greater problems in their removal: they are divided into organic and inorganic soils.

Organic soils are all derived from animal or vegetable (living) matter, and include oils, fats, grease, protein, starch and carbohydrates. If these types of soil have been subjected to excessive heat, such as in ovens, they appear as carbonised deposits, and become more difficult to remove.

Inorganic (or mineral) soils are all derived from the earth’s crust. The main soil of this type that is commonly encountered is water hardness or limescale, although milkstone deposits are also examples of inorganic soils. Before discussing how to remove these soils efficiently, it is important to understand the terms ‘pH scale’, ‘acids’ and ‘alkalis’.

The *pH scale* readily distinguishes acids from alkalis, as well as strong acids from weak acids, and strong alkalis from weak alkalis. It consists of numbers ranging from 0 to 14. The number 7 comes in the middle of the scale, and represents a point that is neither acid nor alkali: this is the neutral point. Solutions of pH 7 to 0 are acidic: the lower the number, the stronger the acid. Solutions with pH 7 to 14 are alkaline: the higher the number, the stronger the alkali solution. The pH range is depicted in Figure 4.1.

Common *acidic* materials are vinegar (acetic acid), car battery acid (sulphuric acid), and citric acid (found in lemon or lime juice). Phosphoric acid, hydrochloric acid, nitric acid and sulphuric acid are called mineral acids, and are used extensively in industrial processing.

The *alkalis* include caustic soda (sodium hydroxide), caustic potash (potassium hydroxide), and soda ash (sodium carbonate).

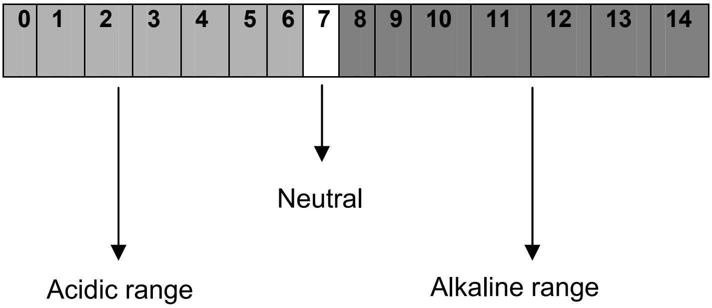

Fig. 4.1 The pH scale.

4.4 Chemistry of water

Water has characteristics that make it particularly suitable for cleaning purposes.

It is often called the *universal solvent*. It can dissolve, suspend or disperse both inorganic (mineral) and organic (plant and animal) materials. It is this ability to dissolve a great number of different chemicals and soils that permits water to be utilised as the medium or carrier for cleaning (liquid/solid interface) surfaces.

Water carries detergent and energy (both mechanical and thermal) to the soils, and then carries the soils from the cleaned surfaces. It therefore becomes an integral part of, and acts like a chemical raw material within, the cleaning/detergent solutions.

Water has a chemistry of its own.

Unfortunately, pure water is rarely found in nature, because of its solvent properties. The contaminants or impurities in water can be solids, liquids, or gases. In addition, water harbours macro- and micro-organisms. Most of these extra ingredients in water can, individually or in combination, cause problems during a cleaning operation.

4.5 Water attributes important to dairy and beverage cleaning and disinfection

The main attributes of water used for cleaning and disinfection purposes are as follows:

- *Sanitary quality*. Water used in food plant sanitation must be potable – that is, fit for human consumption.
- *Microbiological standards*. The following are the main microbial specifications for water, measured in colony forming units (cfu) mL^{-1}: total bacterial count < 100, coliforms absent in 100 mL, and *Escherichia coli* absent in 100 mL.
- *Taste, odour and colour*. Objectionable tastes, odours and colours are usually acquired from rotting vegetation and algae, and are best removed by activated carbon filtration. Suspended matter comprises clay, silt and other organic materials; suspension of more than 1 ppm causes visible turbidity, and is best removed by sedimentation/filtration.

- *Total dissolved solids (TDS)*. This is a measure of all the chemicals dissolved in the water, and is generally not problematic for cleaning and disinfection.
- *Dissolved gases*. Some, such as carbon dioxide, can form weak acids, resulting in the need for additional alkali, or in some cases may cause corrosion.
- *pH*. This should ideally be between 6.5 and 7.5. Below pH 6.5 corrosion problems will occur. The maximum alkaline pH value allowed is 10.
- *Alkalinity*. High bicarbonate alkalinity may contribute to scale formation.
- *Silica*. This is generally not significant in cleaning and disinfection, but on stainless steel surfaces it can form dull layers that are difficult to remove.
- *Sulphides/sulphates*. allowable level is 250 µg SO_4 L^{-1}.
- *Chlorides*. These should not be more than 250 µg mL^{-1} (maximum), but preferably should be below 50 µg mL^{-1} because of the possibility of corrosion, especially in acidic conditions.
- *Iron*. The maximum is 200 µg Fe L^{-1}; staining will be the main issue here.
- *Manganese*. The maximum is 50 µg Mn L^{-1}.
- *Total hardness*. This is the total of all dissolved calcium and magnesium salts, usually expressed as equivalent $CaCO_3$.

Table 4.1 details the conversion factors between the varying methods used to report hardness – both temporary and permanent. *Temporary water hardness* consists of calcium bicarbonate and magnesium bicarbonate. Both salts are quite soluble in water, and consequently might be present in waters in significant concentrations. Under the influence of changes in temperature, pH or pressure, these salts may convert to calcium and magnesium carbonates, which are relatively insoluble in water and precipitate.

Permanent water hardness usually consists of calcium and magnesium salts of sulphate and chloride. These impurities are soluble in water, but are not affected by changes in temperature, pH or pressure. Excessive water hardness requires increased concentrations of cleaning chemicals. Table 4.2 gives general guidelines for the levels of hardness that occur in cleaning processes.

4.6 Basic detergency: how does a detergent work?

There are four basic cleaning techniques for the removal of water insoluble soils.

Table 4.1 Conversion factors to equivalents of calcium carbonate ($CaCO_3$) from other reporting methods.

One unit of hardness as:	Equivalent as µg $CaCO_3$ mL^{-1}
Grains US $gallon^{-1}$	17.1
Degrees Clark	14.3
Degrees French	10.0
Degrees German	17.7

Table 4.2 Levels of hardness expressed as calcium carbonate ($CaCO_3$) and generic classifications used in the dairy and food industry.

One unit of hardness as:	Hardness of $CaCO_3$ ($\mu g\ mL^{-1}$)
Soft water	0–60
Moderately hard	60–120
Hard	120–200
Very hard	>200

4.6.1 *Chemical reaction*

It is possible to alter the structure of some types of insoluble soil chemically so that they become soluble in water, and hence are easily removed. The cleaned article is finally rinsed with fresh water. The most common example of this type of cleaning is the removal of limescale from the heating elements in cleaning-in-place (CIP) tanks and scale from water lines. An acid type of detergent is used that reacts with the limescale deposits to form water-soluble by-products, which are easily rinsed away.

4.6.2 *Solvent cleaning*

The best example of solvent cleaning is the removal of sugar by water rinsing. In solvent cleaning, the soil dissolves in the cleaning solution, and is therefore removed from the substrate. For example, in the transport industry, petroleum-based solvent cleaners are used for cleaning oily car engines, gearboxes and chassis.

4.6.3 *Abrasive cleaning*

The use of abrasives for cleaning represents a mechanical action rather than a chemical application. Examples include sand blasting, which is very effective for renovating building exteriors, and the use of domestic scouring powders.

4.6.4 *Dispersion–suspension cleaning*

This fourth technique of cleaning is by far the most common use of detergents. The chemical nature of the soil is not changed, and yet a water-based solution can be used to remove a water-insoluble soil. The cleaning solution must effectively *wet* the articles to be cleaned. If the soil layer is very thick, then the complete layer must be *penetrated* by the cleaning solution. These functions are achieved by using chemicals that reduce the surface tension of the water, so that it can spread easily, rapidly across and through the soil layer. The detergent solution must also *lift* the soil from the article to be cleaned. Once removed from the article, the soil must then be *suspended* in the bulk of the cleaning solution, not redeposited either on the cleaned article or on the bottom of any tank or container in which the cleaning operation is being performed. To maintain the soil suspended in solution, it is usual for it to be broken up by the cleaning solution into very small particles. Finally, a good detergent must be free-rinsing so that all detergent residues can be easily *rinsed* from the cleaned surface using clean water.

These principles of wetting, penetrating, lifting, dispersing, suspending and rinsing are applied to most cleaning applications in the dairy industry. It is worth looking at the definitions of these and some related terms in more detail.

- *Emulsification* is the mechanical action of breaking up fats and oils into very small particles, which are uniformly mixed with the water used. In a stable emulsion, the oil particles are held apart and left suspended uniformly, for long periods of time. This action is mechanical, as in homogenisation, and emulsifying agents can be used to decrease the amount of mechanical action required to form an emulsion. Often gentle agitation will be sufficient to emulsify fats in the presence of a good emulsifying agent.
- *Wetting* is the action of the cleaning solution on solid surfaces, either clean or dirty. If a surface is to be properly cleaned, the solution must make full contact with the surface and the soil. This is achieved by lowering the surface tension of the solution, so that it spreads and thins out to cover a greater area of the soil.
- *Penetration* is the action of a liquid entering into porous materials through cracks, pinholes or small channels. This action could be considered a part of wetting, and in order to have a good wetting action, it is necessary to have good penetration.
- *Deflocculation* or *dispersion* is the action of breaking up aggregates or flocs into smaller particles, which are more easily suspended and flushed off from the equipment.
- *Suspension* is the action that holds insoluble particles in a solution. It prevents the settling of solids that might form deposits, and it also makes it easy to flush the insoluble particles from surfaces.
- *Peptising* is the breakdown of insoluble protein soils into smaller molecules, which then become soluble.
- *Rinsability* is the property of a solution or suspension that enables it to be flushed from a surface easily and completely. This is accomplished by reducing the surface tension of the water used. Insoluble material must be well suspended and present in minimal quantities to promote good rinsing properties.
- *Sequestration* is the removal or deactivation of metal cations in solution from further reaction by the formation of a soluble complex. An example of sequestration is the solubilising of calcium and magnesium hardness salts with ethylenediaminetetra-acetic acid (EDTA) to prevent their precipitation by alkaline detergents.
- *Synergism* is the term applied in cases where the sum of the actions of two or more materials mixed together is greater than the sum of their individual actions.
- *Buffering* is the action of any material that resists change in pH when an acid or alkali is added to it, or when it is diluted. The effect of a buffer in the cleaning process is to maintain the desired pH of the cleaning solution for the duration of the clean.

4.7 What materials make up a detergent?

A good detergent should possess all the properties listed above. They cannot be provided by any single chemical, and so it is necessary to mix together two or more raw materials to provide a product that fulfils all these requirements. Such products are called *built detergents*, because they can be considered as being built from a number of individual building blocks or individual raw materials.

4.7.1 *Surfactants: synthetic surface-active agents*

The main function of a surfactant is to make water-insoluble oily deposits, which are molecules found on most surfaces during cleaning processes, soluble in water. The molecule of a surfactant has two distinct parts: a *hydrophilic* or water-loving group, which permits it to dissolve in water; and a *hydrophobic* or water-hating group, which seeks substances other than water in which it is soluble and can dissolve. Most commonly, the substances in which the hydrophobic groupings are soluble are oil and fatty-based materials. When an aqueous solution of a surfactant is in contact with an oil surface or film, the hydrophobic group of the molecule dissolves in the oil. The surfactant lifts it from the surface, and using the hydrophilic portion makes it soluble in water.

Soap was the most widely used surface-active agent before the invention of synthetic surfactants. However, soap suffers from some disadvantages: most significantly, it reacts strongly with the calcium and magnesium salts dissolved in hard water to form insoluble soaps. These compounds precipitate out in the form of a scum, which is then very difficult to remove from the surface. Another disadvantage of soap is that, by its nature, it is alkaline: therefore, if it is mixed with acidic solutions or cleaners, it is neutralised and thus loses its surface active property. The use of *synthetic surfactants* can overcome these disadvantages. By the nature of their chemical properties, they reduce the surface tension of water or aqueous solutions, helping in the process of penetration and in the formation of stable dispersions or emulsions.

There are three main groups of surfactants:

- *Anionic surfactants* are mainly sodium salts of complex organic materials, They are usually high foaming, with excellent wetting and soil-dispersing characteristics.
- *Non-ionic surfactants* do not ionise in solution. Many have low foaming characteristics, which makes them well suited for use in conditions where solutions are subjected to high pressure or spraying, such as in CIP.
- *Cationic surfactants* have the poorest detergent qualities. They demonstrate good bactericidal action, resulting in their extensive use as disinfecting agents.

In many cases mixtures of surfactants are used, but it must always be borne in mind that cationic and anionic surfactants cannot be mixed together. The anions and cations will neutralise each other, usually with the elimination of their desirable properties.

In a detergent formulation the surfactants will usually be present in quantities determined by the type of application for which the product is intended. For example, in the manual cleaning of surfaces, the formulation of a liquid general-purpose cleaner is based on relatively high levels of a mixture of suitable surfactants. However, for a heavy-duty CIP cleaner the amount of surfactants may be at the lowest level possible to provide good surface wetting to assist in the penetration of the soil by the alkaline ingredients, and at the same time promote rinsing – all this with complete freedom from foam production, which is undesirable.

Since surfactants are synthesised under controlled conditions, it is possible to tailor-make a range of molecules to meet very specific requirements. As a result, there are hundreds of variations of surfactant available in the marketplace, and it requires great skill and knowledge to select the right surfactant for any given application. Surfactants are powerful tools in the process of cleaning, and their incorporation is usually desirable in order to enhance the total qualities of any given formulation. Although they may be present in very small quantities – for example concentrations of less than 0.1 g or mL 100 g^{-1} or mL^{-1} – they make an important and significant contribution to the cleaning process.

4.7.2 *Inorganic components of detergents, or builders*

A range of alkaline sodium salts are used extensively in formulating detergent products for cleaning hard surfaces. The blending and grouping of selected alkaline materials together with other possible additives results in what is called a *built detergent*. The most commonly used chemicals are caustic soda, soda ash, silicates and phosphates.

Caustic soda (sodium hydroxide)

Caustic soda is a very strong alkaline material, and is the most commonly used material in formulating detergents for use in CIP and other mechanical means of cleaning because of its relatively low cost and high strength. It exhibits excellent removal of proteinaceous soils and fatty oils by saponification. Caustic soda is extremely corrosive to many surfaces, particularly the skin, and it is unsuitable for use in hand-cleaning operations. It also has a deleterious effect on soft metals such as tin, zinc and aluminium, and care must be taken to ensure that any surface to be cleaned with a caustic-based cleaner is resistant to its corrosive action.

When diluted in hard water, caustic soda alone will cause the formation of calcium and magnesium scale or sludges: therefore, the water chemistry and detergent formulation must be considered when making caustic solutions using hard water. This can be addressed either by using formulated caustic detergents, or by occasional descaling with acids. Caustic soda does not have good free-rinsing characteristics, and is therefore difficult to remove from surfaces after the treatment unless carefully selected caustic-stable surfactants are incorporated in the formulation to assist and promote more rapid removal.

In spite of these obvious disadvantages, caustic soda is widely used in heavy-duty cleaning applications where solutions are to be applied by circulation or spray, because of its rapid reactivity with many organic soils.

Soda ash (sodium carbonate)

Soda ash provides high alkalinity, and softens water by precipitation of calcium and magnesium carbonates, provided that the pH of the solution is over 9, and remains so after precipitation has occurred. Its prime use is in alkaline powdered formulations, where it provides soluble alkalinity, which assists in the cleaning process, and is used, in the light form, to absorb large quantities of liquid materials onto its surface while remaining dry to the touch and free flowing. This is particularly useful in formulating complex mixtures where additives must be included.

Silicates

Silicates are used for specific properties in detergents. They soften water by the formation of precipitates, which can easily be rinsed away by the water and do not tend to deposit on the surface being cleaned, unless it is heated. Silicates are able to suspend soil in solution, and prevent their redeposition. The buffering action of silicates means that in mildly acidic conditions their alkalinity is maintained almost until they have been exhausted. This allows the detergent to maintain a relatively constant pH value that varies little with concentration. Finally, silicates effectively inhibit the corrosion of aluminium by other strong alkalis, and are regarded as an important constituent of cleaners for use on soft metals.

Phosphates

The phosphates are broadly divided into two classes: the *orthophosphates* and the condensed or *complex phosphates*. The most widely utilised orthophosphate is trisodium phosphate, which provides a relatively high alkalinity and, at the same time, softens water by a process of precipitation producing a flocculent (non-adherent precipitate). The most widely utilised complex phosphates, in descending order of alkalinity, are tetrasodium pyrophosphate, sodium tripolyphosphate, sodium tetraphosphate, and sodium hexametaphosphate.

All these complex phosphates have the power of softening water by sequestering metallic ions. Sodium hexametaphosphate has the greatest power to sequester calcium ions, and tetrasodium pyrophosphate is best for magnesium ions. The power of sodium tripolyphosphate lies between the two; it is widely used in order to provide a good spectrum of activity. The complex phosphates are able to de-flocculate and keep in suspension insoluble materials such as clays, and also to assist in the emulsification of oily materials. Related to this property of de-flocculation (the breaking up of large masses into small ones) is the property of peptising, which is the ability to keep finely divided solids in suspension and prevent their coagulation.

The complex phosphates suffer from one defect. In solution, particularly at raised temperatures or in the presence of strong caustic soda, they tend to revert to the orthophosphate, with a resulting loss of their sequestering powers. In addition, phosphates cause eutrophication in some inland watercourses, leading to very rapid growth of the micro-organisms, to the point where the oxygen in the water is consumed and other life forms have difficulty in surviving. The detergent industry has replaced phosphates in detergents wherever possible. It is generally agreed, however, that the phosphates are not easy to replace, and their substitutes are not as cost-effective. Hence their continued use is limited and controlled, and approval should be sought before using detergents containing phosphates.

4.7.3 *Sequestrants*

In detergent formulations, sequestrants are used primarily to prevent the precipitation of hard water scale. When used in sufficient quantity, and under the right conditions, they also have a cleaning effect: they help in the removal of complex soiling by complexing the metallic ions (e.g. calcium and magnesium) in the insoluble salts of fats and protein, thus creating their more soluble forms (e.g. sodium).

There are two main types of sequestration: stoichiometric and threshold. In *stoichiometric sequestration* one molecule of sequestrant binds a fixed number of molecules of metal ion. Stoichiometric sequestrants can be further broken down into *inorganic sequestrants* (e.g. phosphates) and *organic sequestrants* (e.g. EDTA).These materials tend to form stable complexes under certain conditions, usually dependent on pH, and are useful in aiding the soil removal process by making the soil more soluble.

Threshold sequestrants act by modifying the crystals involved in scale formation so that they are unable to assemble into large deposits – rather like trying to stack golf balls instead of building blocks. Examples of these materials are the phosphonates (e.g. aminotrimethylene phosphonic acid). The purpose of these materials is really to reduce scale formation while rinsing with hard water and to prevent the scale that is formed from being deposited. As the amount of sequestrant needed is far lower than the metallic ion present, small amounts left behind after the detergent is flushed away will prevent or slow down the formation of scale on the surface during rinsing.

4.7.4 *Acids*

The most common acids found in dairy cleaning are the *inorganic acids*, such as phosphoric acid and nitric acid, and the *organic acids*, such as citric acid. Other mineral acids that may be used from time to time are hydrochloric acid and sulphuric acid, and other organic acids are hydroxyacetic acid and gluconic acid.

Acids are generally used to remove mineral scales, such as hard water and milkstone scales, or to remove soils that are tenacious and difficult to remove using alkaline cleaning. An important advance in recent years in dairy cleaning has been the introduction of acidic cleaning in CIP to replace caustic cleaning for milk lines and silos. This would seem to contradict the science of cleaning as described above, as the soiling from milk is largely organic, and therefore should be cleaned with alkali. With the pressures on the modern dairy to reduce the time and water needed for cleaning, detergent formulators have found a way of combining emulsifiers with acids so that the turbulence of CIP has sufficient mechanical energy to emulsify the fats and proteins so that these soils can be cleaned effectively in an acidic environment.

4.8 Factors affecting detergent performance

Having looked at the chemistry of detergents, we now need to look at the cleaning processes, and how they affect the quality and performance of the clean. The *cleaning wheel* of time, action/mechanical, concentration and temperature (TACT) represents the most influential variables in any cleaning process:

- *Time*. If a detergent is not given sufficient time to complete all of the required actions – penetration, suspension, dispersion and rinsing – the result will be poor cleaning.
- *Mechanical action*. In general, detergents will not remove any soil unless a certain amount of mechanical action is applied. This action may be applied in many different ways, including wiping, rubbing, brushing, flushing and high-pressure jets.
- *Concentration*. This is one of the most obvious and most important factors to be considered. Any product will have a certain optimum concentration at which it will do a specific job. Concentrations below this level will not work as well, and anything more than enough is wasteful.
- *Temperature*. In most cases, the hotter the detergent solution is, the more efficiently it will work. There are a few exceptions to this rule, though: for example, an enzyme-based detergent would tend to be deactivated by excessively high temperatures.

All cleaning processes are combinations of the above. The overall efficiency of the cleaning job will depend on the interaction of all four factors.

A general rule of thumb is that, should one variable be altered, the other three will have to compensate for that alteration. For example, if the water temperature is not sufficiently high for a CIP, either time, chemical concentration or mechanical action will have to be increased. This applies only within the constraints of the cleaning process parameters. If the temperature is too low for the CIP, no further compensation by the other three factors will facilitate adequate cleaning.

One other factor that is often overlooked in the design of a cleaning process is the *detergent to soil ratio*. This means ensuring there is enough detergent solution to do the job; the greater the soil load, the higher the detergent usage will need to be. If the volume of the solution is not adequate to handle all the soil, then either it will need to be increased or the concentration will need to be adjusted to compensate. There will be a concentration above which the benefit will not be seen, and instead the volume will need to be increased.

4.9 Methods of application

The way the detergent is applied will have a strong bearing on the type of cleaner selected.

4.9.1 *Manual cleaning*

In manual cleaning, it is likely that the cleaner will contact the user's skin; this severely limits the choice of products. Materials that are mildly alkaline, mildly acidic or neutral are suitable. As neutral and mild detergents are used only at moderate temperatures, if soil loads are heavy additional mechanical action, such as scrubbing, will be necessary.

4.9.2 *Circulation cleaning (CIP, spray cleaning)*

In this method of application, the cleaning solution is subjected to high turbulence, and so any tendency to foam is undesirable. Circulation cleaners are usually much stronger or aggressive than manual cleaners; they will be high in alkalinity or acidity, and should be handled with care. In circulation cleaning, high temperatures and mechanical action are often achieved. Given these factors, together with the aggressive nature of the chemicals used, this method is the most cost-effective way to clean, as it is also not labour intensive. However, the capital costs of installing CIP systems can be very high.

4.9.3 *Soak-cleaning*

This is the least efficient cleaning system, unless it is improved by the incorporation of some degree of agitation. Where equipment components are soak-cleaned in tanks, the introduction of a small agitator or air line to provide agitation will usually result in a marked improvement in the action. This method relies on long periods of time to perform the clean. The choice of detergent is determined by the object being cleaned, and how it will be removed from the soak bath. Strong chemicals can be used if adequate safety arrangements are in place to prevent personal contact with the solution.

4.9.4 *Spray-washing*

These systems are normally purpose-designed, and are similar in requirements to CIP cleaning. The need for low foaming characteristics is as great, and, in general, mild cleaners should be used whenever possible to minimise the dangers of overspray and bounce-back of solution. The pressure at which spray cleaning is performed is often a point of debate: the higher the pressure, the easier it is to remove tenacious soils, but the greater the chances of spreading the soil around the area being cleaned and of forming aerosols. In general, medium pressure is best suited in combination with a properly formulated detergent, followed by low-pressure rinsing. The other variable to consider is the volume of water applied. This is determined by the design of the pump systems.

4.9.5 *Long-contact vertical surface cleaning using foams or gels*

These well established techniques are achieved by using specially formulated detergents and various mechanical techniques. The most widely is the injection of air into the cleaning solution, which is applied to the surface to be cleaned through purpose-designed nozzles. The main attraction of foam is its ability to adhere to vertical or inverted surfaces and provide a prolonged contact time. These formulations have high levels of high-foaming surfactants to form the foam or gel to provide the contact time, and to help in penetrating and emulsifying the soil as well as aid the rinsing away of the cleaning materials. They also act as carriers of the active ingredients, be they alkalis, acids, disinfectants, or any other actives required.

4.10 The science of disinfection

4.10.1 *Background*

Disinfectants are a specific subgroup of overall detergents whose specific function is to kill and remove micro-organisms – obviously important in keeping a dairy plant microbiologically clean. Different species of micro-organism are destroyed, removed, or inhibited at varying rates depending on environmental and other factors. The organisms may be unwanted contaminants, food spoilage organisms, toxin generators, or organisms that adversely affect human health. In all cases they should be controlled and minimised to acceptable limits so as not to be a threat to human health or cause food spoilage.

4.10.2 *Objectives of effective disinfection*

Equipment and utensils are disinfected to ensure that hygienic conditions are attained. This is a very important step in the general cleaning operation, for the following reasons.

- A variety of micro-organisms may remain on dairy processing equipment after it has been washed, even though it may, on visual inspection, appear to be clean. The organisms may be types that have slowly been accumulating on the equipment or in the product during the processing operation.
- While processing equipment is idle, significant numbers of bacteria may develop, even though the equipment has been cleaned and disinfected. This is especially true of surfaces that are difficult to dry. There are usually sufficient nutrients to support bacterial growth, even on a clean surface.
- Water supplies occasionally become contaminated. When such water is employed for washing or rinsing of equipment, spoilage organisms may contaminate the equipment. The use of a disinfectant in the water employed to rinse equipment helps to prevent such contamination.

4.10.3 *Factors affecting the performance of disinfectants*

Time

Sufficient contact time must be allowed for chemical and physical reactions to occur. The time required will depend on the nature of the disinfectant, its concentration, the pH, the temperature, the nature of the organisms, and the existence in the bacterial population of cells having varying susceptibilities to the disinfectant.

Temperature

For disinfectants, the action of heat (temperature) is inversely related to time: the lower the temperature, usually, the longer the time required to kill the organisms. In the case of chemical disinfectants, the warmer a disinfectant is, the more effective it is. This is based partly on the principle that chemical reactions in general are speeded up by raising the temperature. Usually, within the range of growing temperatures for micro-organisms, a rise in temperature of 10 Celsius degrees increases reaction rates by between two and eight times. However, as many disinfecting actions are partly physical in nature, the laws governing chemical reactions do not apply exclusively. Higher temperatures generally reduce surface tension, increase acidity, decrease viscosity, and diminish adsorption. The first three aspects increase and the fourth diminishes the effectiveness of a disinfectant. It is therefore important to establish the temperature stability of the disinfectant.

Concentration

Studies involving the effect of concentration on the lethal activity of disinfectants have employed the *concentration exponent* (i.e. dilution coefficient), which is a measure of the effect of changes in concentration (or dilution) on cell death rate. To determine the concentration coefficient, it is necessary to measure the time needed for two different concentrations of the disinfectant to produce a comparable degree of death in a bacterial suspension.

Within narrow limits, the more concentrated the disinfectant, the more rapid and certain is its action. Effectiveness is generally related to concentration exponentially, not linearly. For example, doubling a 0.5 mL 100 mL^{-1} concentration of phenol in aqueous solution does not merely double the killing rate for bacteria, but may increase it by 500–900%. Doubling the concentration again may increase the effect by only a negligible amount. There is clearly an optimum concentration: thus the concentration of a disinfectant beyond a certain point accomplishes increasingly less, and is wasteful. The manufacturer's use instructions should always be followed when using disinfectants, and the disinfectant should never be applied at less than the minimum recommended concentration, or for less than the specified time.

Surface tension

The surface tension of the disinfectant solution is important when it has to adsorb onto the bacterial cell membrane to effect a kill: this applies, for example, to the cationics, such as the quaternary ammonium compounds (QACs). The formulating of properly designed surface-active disinfectants will take this into account. Disinfectants based on surface activity will rapidly lose effectiveness if their solutions are contaminated with substances that alter the surface tension, or are of opposing charge to the active material. For example, QACs are inactivated by anionic surfactants.

pH

Always ensure that disinfectants are used at their recommended pH to maximise their effect. The pH can influence biocidal activity in the following ways:

- *Changes may occur in the molecule.* Substances such as phenol, benzoic acid, sorbic acid and hydroxyacetic acid are effective only or mainly in the non-ionised form. As the pH rises, an increase in their degree of dissociation occurs, lowering their effectiveness. Glutaraldehyde is more stable at acid pH, but is considerably more potent at alkaline pH.

- *Changes may occur on the cell surface*. As the pH increases, the number of negatively charged groups on the bacterial cell surface increases, with the result that positively charged molecules such as QACs have an enhanced degree of binding.

Number and location of organisms

Obviously, it is easier for a disinfectant to be effective when there are few micro-organisms against which it has to act. Similarly, the location of micro-organisms must be considered in assessing its activity. Biofilms are one category that needs special mention. If the disinfectant does not penetrate the biofilm, then the organisms under the film are able to live and reproduce, leading to the possibility of an infection when the film is ruptured. A further example of this occurs in the cleaning of equipment used in the large-scale production of cream, where difficulties may arise in the penetration of a disinfectant to all parts of the equipment.

Organic matter

Organic matter occurs in various forms, such as earth, food residues or faecal matter, all of which may interfere with the bactericidal activity of disinfectants and other anti-microbial compounds. This interference generally takes the form of a reaction between the disinfectant and the organic matter in addition to (or in the worst case instead of) the micro-organisms to be killed, thus leaving a reduced concentration of the disinfectant for attacking the micro-organisms. Alternatively, the organic material may protect the micro-organisms from attack. In addition, organic matter decreases the effect of disinfectants against bacteria, viruses and fungi. This is particularly true of hypochlorites. Because of the lower chemical reactivity of iodine and iodophors, they are influenced to a lesser extent. Phenols may also show a reduced activity in the presence of organic matter. Disinfectants should therefore, as a rule, be used only on pre-cleaned surfaces.

Metal ions

Depending on the type of disinfectant chosen, its activity may be reduced or enhanced or remain unchanged in the presence of metal cations. The antibacterial activity of many compounds is increased or activated against Gram-negative bacteria when EDTA is present. Because hard water has increased magnesium and calcium levels, it could therefore have a negative effect on disinfectant efficacy.

Type of organisms

Generally, Gram-positive bacteria are more sensitive to biocides than are Gram-negative bacteria. The main reason for this difference in sensitivity probably resides in the relative composition of the cell envelope. Many antibacterial substances may not be sporicidal, but may be sporostatic – phenols, QACs, biguanides and alcohols, for example. Comparatively few substances are actively sporicidal: examples include glutaraldehyde, formaldehyde, halogens, ethylene oxide and acid alcohol.

4.10.4 *Choosing the most appropriate disinfectant*

The value of any substance as a disinfectant depends on several factors. It must:

- be highly effective against a wide variety of micro-organisms, in low concentrations so that it is economical to use;
- be non-corrosive and non-staining to materials likely to be in contact with the disinfectant, such as metals and linings;
- have a non-offensive odour and taste;
- be as specific as possible for micro-organisms – that is, not deactivated by extraneous materials;
- be a good surface tension reducer – that is, have good wetting and penetrating properties;
- be stable in storage;
- be readily available, and not expensive;
- be easily applied under practical conditions of use;
- be completely bactericidal within the required time limits for the application.

When selecting a disinfectant, the different types should be considered for their respective merits. In broad terms, there are two classes of disinfectant according to the type of microbial inactivation: physical and chemical. The only *physical disinfectant* widely used in the dairy industry is steam. *Chemical disinfectants* used in the dairy industry can be classified into two further groups: oxidising and surfactant-based non-oxidising disinfectants.

Heat

Heat, in the form of steam, hot water or hot air, may be used for disinfecting. However, the practicalities of its use to disinfect must be carefully considered. Heat is often more costly than chemical disinfection, and is more difficult to use, particularly in open systems such as vats and tanks. Further disadvantages of heat are as follows.

- Processing plant is left hot, and may need to be cooled prior to use, which may cause non-sterile air to be sucked in and cause contamination.
- Heat destroys the elasticity of rubber, so that gaskets and seals harden, creating sites for microbial contamination.
- It often takes longer for the effective temperature to be reached across the complete system than the time needed for chemical disinfection.

However, when correctly applied and controlled, heat is very effective.

Oxidising disinfectants

These are chemicals that react chemically with the microbial cell wall through an oxidation reaction, either changing its capability to absorb nutrients or rupturing it; both lead to the death of the organism.

Chlorine-based disinfectants

These are widely used, and they have excellent bactericidal power against a wide range of micro-organisms. In properly blended products they are relatively non-staining, and are easy to prepare and apply. Chlorine-based disinfectants are generally economical to use. However, some of the available chlorine disinfectants may be readily consumed by organic matter other than bacteria, so effective cleaning is essential. Although chlorine gas has been used for the disinfection of swimming pool water, it is too hazardous for general purposes, and chlorine-containing compounds are used. These compounds can be roughly divided into two types: the inorganic substances containing hypochlorite ions, and the organic chlorine release agents.

Sodium hypochlorite dissociates, and the N-Cl group common to the organic compounds hydrolyses in water to form varying amounts of hypochlorous acid (HOCl). It is this acid that is the active element in chlorine disinfection, and the relative proportion of HOCl in such mixtures is affected by pH. The optimum pH for activity is around 7, but, because this does not favour stability, alkaline formulations are normally used.

- *Range of activity.* Chlorine-releasing materials are effective against all vegetative bacteria, viruses and, at higher concentrations, bacterial spores, yeasts and moulds. The activity is reduced at low temperatures – significantly so in the presence of organic matter.
- *Mode of action.* The precise mechanism of action is unknown, but these compounds are highly reactive, and are strong oxidising agents. It follows that microbial proteins will be subject to attack by chlorination of the amino groups and oxidation of the thiol (–SH) groups.
- *Applications.* These products are suitable for use in recirculation. Apart from sodium hypochlorite, the products are fully formulated materials, which should be used in accordance with the manufacturers' instructions. Levels of available chlorine at the concentration for use typically range from 50 to 250 $\mu g\ mL^{-1}$. In general, sodium hypochlorite solutions contain between 8 and 12 $\mu g\ mL^{-1}$ available chlorine, and can be used on their own to disinfect after cleaning, to treat rinse water, or with a suitable dairy detergent for cleaning and disinfection. The blending of detergents with sodium hypochlorite should be done only when clear instructions to do so are recommended by the detergent manufacturer. Sodium hypochlorite must never be added to, or allowed to come into contact with, acidic detergents. It should not be used on tinned surfaces, such as zinc or aluminium. Formulated products contain inhibitors, which enable them to be used on these metals with care. All chlorine-based products are potentially corrosive to all metals, and contact times and temperatures should be controlled to manufacturers' recommendations. They may safely be used on glass, vitreous enamel, plastics and rubber.
- *Health and safety.* Concentrated hypochlorite solutions are caustic and toxic, and can react quickly and strongly in contact with organic matter. Chlorine-releasing powders are hydrolysed in a moist atmosphere, which may induce heat evolution or an explosion if organic materials are mixed with them. Under no circumstances should these compounds be mixed with acids, as this could result in the evolution of toxic chlorine gas.

Iodophors

These compounds are defined as combinations of elemental iodine and wetting agents, usually buffered with acid so that the dilutions used are on the acid side of the pH scale. The wetting agent or surfactant serves as a carrier for the iodine, and aids rapid penetration of the soil. Iodophors are usually formulated with acid, the latter enhancing the bactericidal properties by maintaining the pH of the use solution on the acid side.

- *Range of activity*. Iodophors are generally effective against Gram-negative and Gram-positive vegetative bacteria, yeasts, moulds, viruses and, at higher concentrations, against spores. Action is rapid and lethal rather than inhibitory. Activity is reduced at low temperatures, but less so than with most other disinfectants. As already discussed, an acid pH is required.
- *Mode of action*. The activity of iodophors is provided by an amount of free iodine that exists in equilibrium with the bulk of the inactive complex. As the iodine is used up, further iodine is rapidly released from the complex. It is very reactive and, in particular, is known to combine with –SH groups in microbial enzymes and other cellular proteins.
- *Applications*. Iodophors can be used for spray or soak applications. Most of these disinfectants are high foaming, but low-foaming varieties exist, and these can be used for recirculation. Typically, up to 50 $\mu g\ mL^{-1}$ of iodine is used for surface and recirculation applications. Iodophor solutions range from pale yellow to amber brown in colour. As the iodine is used up, the colour diminishes, and this property gives crude, but sometimes useful, guidance to the amount of remaining iodine. Iodophors are suitable for use on molybdenum-containing stainless steel (Note: short contact times only on other grades), glass, vitreous enamel and most rubbers. Some rubbers absorb iodine and many plastics stain, and iodophors should not be used on tinned surfaces, copper, brass, zinc or aluminium. Iodophors are completely formulated products, and should not be blended with other materials. They should be stored in a cool place.
- *Health and safety*. Most iodophors would be classified as slightly toxic, but this will depend on the overall formulation. Highly acidic products are corrosive for storage and handling.

Peroxide-based disinfectants

These types of disinfectant have many advantages over the conventionally used halogen disinfectants. The major advantage of these materials is the rapid disinfecting action, and the environmentally acceptable breakdown products: for example, hydrogen peroxide forms oxygen and water. Their main advantages are as follows.

- Their decomposition products are environmentally acceptable.
- The acid-based disinfectants have a rapid disinfecting effect on a broad spectrum of organisms.
- They are low-toxicity disinfectants.
- Most will not cause tainting of food-stuffs.

Their disadvantages are:

- They are dangerous to handle.
- They have a water-like appearance.
- They are destabilised by organic matter and trace amounts of metallic ions.
- They are extremely strong oxidising agents, which may explode or catch fire when concentrates come into contact with organic materials.

The most important of the peroxide-based compounds for disinfection purposes are hydrogen peroxide and peracetic acid. *Hydrogen peroxide* is a slow-acting disinfectant, but has applications, especially in packaging applications. *Peracetic acid* is a fast-acting disinfectant, and has wider application. It is provided as a relatively stable equilibrium mixture of peracetic acid, water, hydrogen peroxide and acetic acid. Peracetic acid is a strong oxidant.

- *Range of activity*. Hydrogen peroxide is bactericidal and fungicidal, but long contact times are necessary. These can be shortened by increasing the temperature, but proper application instructions must be followed as detailed by the equipment or disinfectant supplier. Peracetic acid is effective against bacteria (including spores), yeast moulds and viruses; its effectiveness is dependent on temperature, and it is rapidly deactivated by organic matter.
- *Mode of action*. These materials attack oxidisable compounds of any kind, such as the –SH groups and amino groups, in a relatively non-specific way.
- *Applications*. Peracetic acid and hydrogen peroxide are non-foaming, and can be used in spray and recirculation applications. Manual use is not recommended because of the corrosive nature of the products, and the pungent odour of peracetic acid. Active peracetic acid is used in dilutions typically containing between 50 mg L^{-1} and 750 mg L^{-1}; such solutions are mildly acidic (pH 3–5), and may be used alone or be added to up to 20 g L^{-1} phosphoric acid to provide combined descaling and disinfection. Hydrogen peroxide is typically used hot at concentrations of 2.5–10 g L^{-1}. Apart from the addition of peracetic acid to acid, these products should not be mixed with other materials. Dilutions of hydrogen peroxide are non-corrosive to dairy plant, and can be widely used. Peracetic acid may be used on glass, vitreous enamel, plastics and rubber, although some types of rubber may be degraded. It can be used on stainless steel provided that the chloride level of the water used is not greater than 150 mg L^{-1}. It should not be used regularly on tinned surfaces, aluminium or zinc, and not at all on copper or brass.
- *Health and safety*. Concentrates of both hydrogen peroxide and peracetic acid are corrosive and oxidising. They should be kept in a cool place, and should not be contaminated with rust or organic matter. The vapour of peracetic acid should be avoided.

Non-oxidising surfactant-based disinfectants

Quaternary ammonium compounds (QACs)

QACs are disinfectant compounds in which four organic groups are linked to a nitrogen atom, producing the cation. Some common examples are dioctyl dimethyl ammonium bromide and lauryl dimethyl benzyl ammonium chloride. Not all QACs are good disinfectants. Their activity is related to the lengths of the alkyl chains attached to the nitrogen atom, and the presence of one or two such chains. QACs are generally high foaming, and have a slight wetting and detergency at use concentrations.

- *Range of activity.* In general, QAC disinfectants are effective against vegetative bacteria, but with a greater effectiveness against Gram-positive organisms than against Gram-negative types. They are effective against yeasts and moulds, but in these cases higher concentrations are required. They are not generally effective against viruses or bacterial spores. The activity of many QACs is markedly depressed by organic matter, and also by hardness in water. The latter, which is due to an effect of calcium and magnesium ions on the bacteria, can sometimes be controlled by the addition of a sequestering agent. QACs' activity is greatest at alkaline pH and lowest at acid pH, and their effectiveness increases with temperature.
- *Mode of action.* QACs, in common with other cationics, are readily adsorbed to the normally negatively charged bacterial cell because of their positive charge. Following adsorption, QACs penetrate the cell wall and react with the cytoplasmic membrane. The membrane is damaged, and its function is disrupted. At high QAC levels the cytoplasm is coagulated; at low levels the QACs cause a small amount of membrane damage, which is repairable. This is seen as bacteriostasic (i.e. prevention of cell growth, but not death). Only when cell damage is sufficient does microbial death result.
- *Applications*. QACs are high foaming, and are not suitable for use in recirculation. They can be used in soak baths for manual disinfection procedures, and for fogging and spray applications. The use concentrations will normally be between 150 and 250 mg L^{-1} of active QAC. These products should not be mixed with other chemicals, and they are readily deactivated by many materials found in detergents and other products. QACs are non-corrosive and may be used on all materials. They are substantive: that is, they stick to surfaces, such as stainless steel, which provides a degree of residual activity, but which may give rise to problems with taint or with starter cultures. QACs are stable and have long shelf lives.
- *Health and safety.* QAC-based products may cause eye and skin irritation. They will certainly dry the skin, and care should be taken during handling.

Acid anionic

This type of disinfectant allows the acidified rinse to be combined with the disinfecting procedure, and they have a rapid activity against most micro-organisms, especially psychotropic bacteria. Acid anionic compounds do not have a bacteriostatic residue, and are not affected by hard water or organic residues. Unlike the cationics and the amphoterics, the anionic surface active agents are not antimicrobial at neutral pH; however, at $pH < 2$, several anionics possess an excellent antimicrobial activity, and in this form (with mineral acid, usually phosphoric) are used as disinfectants. Depending on the anionic surfactant used, these products are high or low foaming, and they possess good detergent properties.

- *Range of activity.* These disinfectants are effective against vegetative bacteria, but they are less effective against yeast and moulds, and not effective against bacterial spores. The pH must be maintained at ~2 or below. Organic matter and water hardness depress their activity.

- *Mode of action.* At low pH (2 and below), the net charge on the surface of the bacterial cell changes from negative to positive, and at these pH levels the anionics become adsorbed in the same way that cationics do at neutral pH. Disruption of the cell membrane and of cell proteins has been observed, but the precise mechanism has yet to be identified.
- *Applications.* The precise range of applications will depend on foam generation. Only the lowest-foaming varieties will be suitable for recirculation. Concentrations for use will vary with the product, but active levels will be similar to those of QACs. Acid anionics are supplied as formulated products, and should not be mixed with other disinfectant products. They are corrosive, and should not be used on tinned surfaces, copper, brass, aluminium or zinc; however, they may be used on glass, vitreous enamel, plastics and rubber.
- *Health and safety.* Acid anionics are corrosive by virtue of their acid content, and should be handled with caution. They should not be mixed with sodium hypochlorite, because toxic chlorine gas will be released.

Biguanides and chlorhexidine

Chlorhexidine and polymeric biguanides have a wide spectrum of antibacterial activity against both Gram-positive and Gram-negative bacteria. They are not sporicidal, and have low activity against fungal spores, many of which are resistant. As they are cationic, their activity is reduced in the presence of soaps and other anionic compounds. Another cause of loss of activity is the low solubility of their phosphate, borate, citrate, bicarbonate, carbonate or chloride salts. Any system that contains these anions will precipitate chlorhexidine.

Biguanides are derivatives of guanidine, a naturally occurring substance found in certain vegetables and cereals. Two guanidine molecules can be linked together and polymerised (i.e. further joined together) to produce substances with disinfectant properties. The two common examples are chlorhexidine, which is extensively used in hospitals, and the more polymerised polyhexamethylene biguanide (PHMB), which is used in the dairy industry and will be discussed further. Unlike QACs, the biguanide disinfectants are low foaming, and do not have significant wetting or detergent properties.

- *Range of activity*. PHMB is more effective against Gram-negative bacteria than the QACs. In other respects its activity is similar to that of QACs, but it is not effective against viruses or bacterial spores. PHMB is not significantly affected by hard water salts; it is reversibly deactivated at $pH < 3$, and precipitated above pH 10 by anionic detergents, caustic soda and hypochlorites.
- *Mode of action*. As with the QACs, PHMB is adsorbed to the bacterial cell; this is followed by penetration of the wall, and reaction with and damage to the cytoplasmic membrane. Depending on the concentration used, this damage will be temporary (i.e. bacteriostatic) or permanent (i.e. bactericidal).
- *Applications*. PHMB has lost favour as a disinfectant, and is generally used alone for disinfection by manual or recirculation procedures. The concentrations for use will typically be between 100 and 200 mg of active PHMB L^{-1}. PHMB should generally not be mixed with other chemicals as it is precipitated above pH 10, and good rinsing must be achieved between the cleaning cycles and PHMB treatment. PHMB is essentially non-

corrosive, and may be used on all materials. There can be some tarnishing of copper, and some degradation of poor rubber.

- *Health and safety*. PHMB is of low toxicity, but may cause some skin irritation in concentrated solutions. Dilute solutions are safe to handle.

Amphoteric disinfectants

Amphoteric disinfectants have the advantage that they are not as dependent on the pH of the use solution as either the QACs or acid anionic disinfectants, owing to their amphoteric nature. However, they are usually blended with either QACs or anionics to improve the efficacy of those active agents (i.e. QACs or anionics) at varying pH. These disinfectants are surface-active agents that, under certain circumstances, can exist as an anionic species (negatively charged), as a cationic species (positively charged), or as a zwitterionic species (with both charges). Certain of these materials exhibit a significant disinfectant effect, and are used for this purpose. The amphoteric disinfectants are high foaming, and have wetting and detergent properties.

- *Range of activity*. The amphoterics are equally effective against most vegetative bacteria, and they are also effective against yeasts and moulds at higher concentrations. They are not effective against bacterial spores or viruses, and their activity is depressed by organic matter.
- *Mode of action*. The mode of action of amphoterics is not well documented; however, it is probable that these materials, like the cationics, are active at the cell membrane.
- *Applications*. Amphoteric disinfectants are suitable for use in spray, soak and general manual applications. They are too high foaming for recirculation, and concentrations for use will be in the order of 250 to 1000 mg active material L^{-1} (typically 0.25–1.0% of product). These products are not generally mixed with other materials for use, and deactivation may occur if this is attempted. Amphoterics are non-corrosive, and safe to use on all materials.
- *Health and safety*. The amphoterics are of low toxicity and, in dilute solution, are essentially harmless.

A general summary of the activities of common types of disinfectant available to the dairy industry is shown in Table 4.3. Note that although alcohol disinfectants are given in the table, they are generally used only for specific manual applications, and therefore are not discussed in this chapter.

4.11 Construction materials and their corrosion: influence on choice of detergents and disinfectants

4.11.1 *Aluminium and its alloys*

Aluminium is attacked by all acid-based products unless they are specifically formulated to be inhibited. Aluminium is also attacked by soda ash and caustic soda. The use of silicates, however, prevents alkaline attack of aluminium. This also applies to chlorinated products.

Table 4.3 General summary of activities of commonly found disinfectants in the dairy and food industry.

	Type of micro-organisms							
Type of disinfectant	Gram +ve	Gram −ve	*Mycobacterium* spp.	*Pseudomonas* spp.	Yeasts	Moulds	Spores	Viruses
Halogens[a]	++	++	++	++	++	+	+	++
QAC[b]	++	+	−	+/−	++	+	−	+/−
QACs + chelator	++	+	−	+	++	+	−	+/−
Peracetic acid	++	++	++	++	+	+	+	++
Amphoterics	++	++	−	+/−	++	+	−	+/−
Amphoterics + chelator	++	++	−	+	++	+	−	+/−
Alcohols (60–70 g 100 mL^{-1})	++	++	−	++	+	+	−	+/−
Acid anionic	+	++	−	+/−	+/−	+/−	−	−
Acid biguanides	++	++	−	+	+	+	−	+/−

[a] Some examples are chlorine, iodine and chlorine dioxide.

[b] Quaternary ammonium compounds.

Note: ++, very active; +, slightly active; −, not effective; +/−, doubtful.

The potential disadvantage of a high silicate content is that it can cause a dull appearance of the metal alloy, which is difficult to remove.

4.11.2 *Mild steel*

Mild steel, commonly used for tank and vessel construction and/or pipelines, is susceptible to attack by neutral or acidic materials. In particular, hydrochloric acid is highly corrosive to mild steel, whereas caustic and formulated caustic products do not corrode mild steel.

4.11.3 *Stainless steel*

Stainless steel is resistant to corrosion, and is susceptible to attack by hydrochloric acid or by chlorine-containing solutions only where the pH is below 7. Note that chlorides in water can corrode stainless steel, if the water has an acidic pH. This can be accelerated even further in the presence of oxidising agents.

4.11.4 *Copper*

Copper is susceptible to attack by acids and by caustic soda. The rate of corrosion by acids varies: for example, with phosphoric acid it is fairly slow, but with nitric or sulphuric acids it is extremely rapid. Caustic products do attack copper, but the rate of attack is slow enough that caustic detergents can be used – with care – for the cleaning of copper vessels.

4.11.5 *Galvanising*

Galvanising is a zinc coating applied to steel components by many manufacturers in an effort to prevent corrosion. Any alkaline or acid product will rapidly attack and remove galvanising, so although it would prevent corrosion by water, it does nothing to protect metal against the detergents.

4.12 Conclusions

When selecting a detergent or a disinfectant, it is important to have a good understanding of the processes and chemistry involved. The chemistry and make-up of the water to be used in cleaning and disinfection must be considered at all times, and this is one of the key elements to a successful operation. For detergents, knowledge of the soils to be removed, the make-up of the surface to be cleaned, and the cleaning time available will determine both the type of detergent and the method to be used. For optimum cleaning performance, based on time, costs and quality, often complex detergents need to be used. An understanding of the materials used in formulated detergents is useful in assessing the properties of these materials.

When selecting a disinfectant, compatibility with both the water to be used and the detergent chemistry used prior to disinfection must be considered, together with the types of organism expected to be present. As for detergents, the function of disinfectants is determined by their physical and chemical properties, and these need to be considered, together with the disinfection data for the product (kill rate under specific conditions) when determining the most effective product for the prevailing conditions.

There is no universal detergent or disinfectant. The success of cleaning and disinfection processes will be determined by consideration of all the factors described when designing these processes: the choice of chemical, the water used to dilute the product, and how the materials will be applied to the surfaces to be cleaned and disinfected.

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5 Designing for Cleanability

A.P.M. Hasting

5.1 Background

Cleaning plays a key role in ensuring that the final product from the manufacturing process achieves the desired quality standards on a consistent and cost-effective basis. Failure to clean effectively may have serious implications in terms of product quality and storage characteristics as well as process operation and control. There are ever-increasing legislative and commercial pressures for hygiene standards to be improved, and cleaning is not always given the attention it merits. Cleaning-in-place (CIP) is widely applied throughout the dairy and food processing industries as a whole, as the improvements possible in cleaning and hygiene standards become apparent. CIP provides improved control and monitoring over manual cleaning, and hence greater quality assurance.

CIP may be defined as the circulation of chemicals and/or water through plant that remains assembled as for production, such that all product contact surfaces are cleaned, and where necessary disinfected or sterilised, to an acceptably high and consistently reproducible standard. While the focus of CIP is usually on cleaning of the process line, the process covers several distinct stages, and designing for cleanability must take all these into account:

- product recovery or rinsing
- cleaning
- disinfection or sterilisation

Any discussion of design in relation to hygiene and cleaning tends to focus on the design and installation of the equipment. Although this is indeed important, it is not the only design aspect that is critical to the effectiveness of CIP, and there are several interrelated factors that have a direct bearing on the ease of cleaning process plant, as shown in Figure 5.1.

Plant and equipment design is clearly important, because, if the physical construction is such as to prevent or restrict the cleaning solution from reaching all the product contact surfaces under the desired conditions, cleaning will be compromised. The design of the cleaning process itself is also important in terms of the physical and chemical means used to remove the soil from the equipment. The product and process design may have a major impact on the type, quantity and composition of soil that has to be removed from the system, and will define the challenge that the cleaning process has to overcome. A system of poor mechanical or process design may therefore be uncleanable, irrespective of the cleaning process imposed on it. It is therefore essential that consideration of cleaning, both manual and CIP, is integrated into the mechanical and process design from the outset, rather than a final process to be incorporated into an already fully specified plant.

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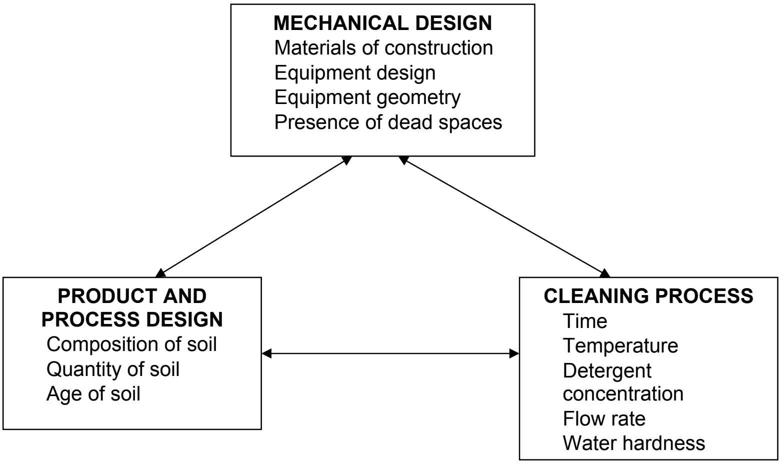


Fig. 5.1 Factors affecting the cleaning of a food process plant.

5.2 Equipment design and installation

Food processing equipment is designed and built to be fit for purpose. A design with excellent hygienic characteristics, but unable to meet its functional requirements, will be of little or no value in a practical production environment. Upgrading existing designs to meet more rigorous hygienic requirements may well be both unsuccessful and expensive: hence the importance of taking hygienic requirements into account as early as possible during the design stage.

5.2.1 *European Union (EU) regulatory requirements*

The EU Machinery Directive 89/392/EEC and its amendments 91/368/EEC, 93/44/EEC and 93/68/EEC (EU, 1989, 1991, 1993a, 1993b) made it a legal obligation for machinery sold in the EU after 1 January 1995 to be safe to use, provided the manufacturer's instructions were followed. The Directive included a short section on requirements for hygiene and design, which stated that machinery intended for the preparation and processing of foods must be designed and constructed to avoid health risks. Subsequent to this Directive, a European Standard EN 1672-2 (Anonymous, 1997) has been adopted to further clarify the hygiene rules established in the original directive (see Holah, 1998).

5.2.2 *The European Hygienic Engineering and Design Group (EHEDG)*

The EHEDG (www.ehedg.org) is an independent group that includes food manufacturers, equipment suppliers and research institutes. The aim is to deal with specific issues relating to the hygienic manufacture of food products by providing design criteria and guidelines on all aspects of equipment, buildings and processing. The emphasis on guidelines is a deliberate attempt to avoid the prescriptive, individual design specifications sometimes found with other international organisations. Extended summaries of many of these guidelines have been published in *Trends in Food Science and Technology* (see Table 5.1), and full versions of the documents may be obtained via the website.

5.3 Hygienic design principles

Hygienic design principles can be broadly classified under three main headings:

- *Materials of construction* should be: (a) resistant to food and cleaning materials under the conditions of use, such as temperature, pressure and concentration; (b) non-toxic; and (c) smooth, non-porous and free from crevices.
- *Equipment geometry and fabrication* should: (a) be self-emptying and draining; (b) avoid creation of stagnant areas where product can accumulate; (c) avoid sharp corners and metal-to-metal contact; and (d) be designed to protect product from external contamination.

Table 5.1 Summaries of relevant EHEDG guidelines in *Trends in Food Science and Technology*.

Information/test methods
Hygienic equipment design criteria
Welding stainless steel to meet hygienic requirements
Hygienic design of closed equipment for the processing of liquid food
Hygienic pipe couplings
Hygienic design of valves for food processing
Hygienic design of equipment for open processing
A method for assessing the in-place cleanability of food processing equipment
A method for assessing the in-place cleanability of moderately sized food processing equipment
A method for the assessment of in-line pasteurisation of food processing equipment
A method for the assessment of bacteria-tightness of food processing equipment
Microbiologically safe continuous pasteurisation of liquid foods
Microbiologically safe continuous flow thermal sterilisation of liquid foods
The continuous or semi-continuous flow thermal sterilisation of particulate foods
Hygienic packing of food products
Microbiologically safe aseptic packing of food products
Passivation of stainless steel
Hygienic design and safe use of double-seat mixproof valves
General hygienic design criteria for the safe processing of dry particulate materials
The prevention and control of *Legionella* spp. including Legionnaires' disease in food factories

Adapted from Cocker (2003).

- *Cleaning* requires that: (a) equipment be easily dismantled for cleaning; (b) surfaces be easily visible for inspection; and (c) if using CIP, it can be demonstrated that results obtained without dismantling equipment are satisfactory.

The principles of hygienic design are generally accepted as straightforward and non-controversial. However, they have limitations when being applied in practical situations:

- They are difficult to apply in absolute terms.
- They are defined in isolation from the product being processed.
- They are related to equipment rather than the process.

It is therefore important to be aware of the product bring processed, its thermophysical properties, and hence its likely interaction with the equipment geometry. A low-viscosity fluid, such as skimmed milk, will interact with particular equipment geometry in a very different way from a viscous non-Newtonian material, such as yoghurt or butter. The hygienic implications resulting from this may also be very different in terms of the likelihood of product being retained within a geometry for a significant time, which may result in microbial growth and product contamination.

In many practical applications, the various pieces of equipment may require different levels of hygiene, depending on the risk to the final product. For example, a mixer for raw meat need not be designed to the same level as a slicer for cooked meats (Timperley & Timperley, 1993). With this in mind, the EHEDG has developed definitions for different hygiene levels, ranging from unhygienic to fully aseptic (Table 5.2).

It may, however, still be possible to produce microbiologically acceptable product using unhygienic equipment, but there are both cost and risk implications, as this may result in a requirement for:

- shorter run lengths between cleaning;
- longer cleaning times;
- more aggressive cleaning regimes;
- a less consistent and robust process; and
- increased product and equipment testing, thus moving from quality assurance towards quality control.

Table 5.2 Characteristics of different equipment hygiene standards.

Hygiene classification	Bacteria-tight	Full CIP/SIP[a] capability	Cleanable and sterilisable after dismantling
Aseptic	Yes	Yes	Yes
Hygienic Class I	No	Yes	Yes
Hygienic Class II	No	No	Yes
Unhygienic	No	No	No

[a] CIP/SIP – cleaning-in-place/sterilisation-in-place.

5.4 Hygienic design requirements

The European Standard EN 1672-2 (Anonymous, 1997) summarises hygienic design requirements under several generic headings:

- materials of construction
- surface finish
- joints
- fasteners
- drainage
- internal angles and corners
- dead spaces
- bearings and shaft seals
- instrumentation
- doors, covers and panels
- controls

5.4.1 *Materials of construction*

Food contact materials must meet specific requirements. They must be inert to the product under all in-use conditions, such as temperature and pressure, as well as to any chemicals used, such as detergents or biocides. They may also need to be resistant to pressurised hot water or steam sterilisation. They must be corrosion resistant, non-tainting, mechanically stable, smooth and non-porous. No toxic materials should be used for food contact, and particular care must be taken when elastomers, plastics, adhesives and signal transfer liquids are used, as these may contain toxic components that could be leached out into product. The suppliers of such components must provide clear evidence that the materials meet all legislative requirements.

Stainless steel

There are a wide range of stainless steels available as construction materials for food industry equipment: the choice is dependent largely on the corrosive properties of the product or other chemicals that will come into contact with the material. The most common choices are the austenitic stainless steels (American International Standards Institute, AISI 304, AISI 316 and AISI 316L), which have good mechanical properties and fabrication characteristics, as well as having an attractive appearance.

- *AISI 304* (Deutsche Industrie Norm (DIN) Werkstoff No. 1.4301) – the cheapest of these – finds a wide range of application in the food and beverage industries, as it has generally good corrosion resistance in a wide range of environments as well as being easy to form and weld.
- *AISI 316* (DIN Werkstoff No. 1.4401) has molybdenum added (2–3 g 100 g^{-1}) to enhance corrosion resistance.

- *AISI 316L* (DIN Werkstoff No. 1.4404) is a low-carbon version (max 0.03 g 100 g^{-1}) compared with AISI 316 (max 0.08 g 100 g^{-1}). This reduced carbon content facilitates easier welding, and hence is often recommended for use with pipework and vessels.

All stainless steels are susceptible to corrosion in the presence of chloride-containing environments (Covert & Tuthill, 2000). Corrosion may take different forms – pitting, crevice or stress corrosion – but a common characteristic is that they are localised and are affected by factors such as chemical environment, pH, temperature, fabrication methods, tensile stresses, oxygen concentration and surface finish.

Other more exotic materials, such as Incoloy 825, titanium and duplex steels (Anonymous, 1983b), are available for high-corrosion environments, but at substantially increased cost.

Plastics

Plastics are used in a range of applications, such as site glasses, pistons, conveyors and hoses. Careful initial specification of the plastic is essential to ensure that:

- The plastic is approved for use in contact with food; the supplier must provide evidence of the approval certification. Particular concern relates to possible tainting of product due to the leaching-out of potentially toxic components.
- The plastic is capable of withstanding in-use conditions during production and cleaning, including temperatures, pressures and concentrations.
- The plastic has sufficient mechanical strength to withstand the mechanical shocks that are likely to occur during normal operation.

The following plastics are considered to be easy to clean, and are suitable for use in hygienic applications (Lewan, 2003):

- polypropylene (PP)
- polyvinyl chloride (PVC) (unplasticised)
- acetal copolymer (AC)
- polycarbonate (PC)
- high-density polyethylene (HDPE)

Polytetrafluoroethylene (PTFE) is often considered to be a potentially attractive material, because of its high chemical resistance. However, care must be taken, because it can be porous and thus difficult to clean. In addition it may be insufficiently resilient to provide a permanently tight seal, and it is therefore considered unsuitable for aseptic processing (Lewan, 2003).

Elastomers

Many types of elastomer are used for seals, gaskets and joint rings. Such rubber-based compounds are widely used because of their high elasticity and hence their ability to return to their original shape once removed from the source of the stress. Specification of such materials can be difficult, for the following reasons (Lewan, 2003):

- There are no agreed standards for compounding, and hence a wide range of elastomer compounds may be used by the supplier.
- All conditions to which the material is subjected must be considered, including in-use process conditions, oil and fat compatibility, and exposure to UV or ozone, as well as the mechanical characteristics of the application, such as whether the elastomer is in a static or a dynamic environment.

The most widely used elastomers are:

- nitrile rubber
- nitrile/butyl rubber (NBR)
- ethylene propylene diene monomer (EPDM), although it is not resistant to oils and fats
- silicon rubber
- fluoroelastomer (Viton)

Silicon and Viton have the highest temperature capability, and can be used at up to 180°C.

Routine replacement of elastomers is a normal part of plant maintenance: the frequency is application dependent, and is a function of the physical and chemical stresses imposed on the material. Symptoms of elastomer deterioration are usually seen as a hardening of the material, leading to a loss of elasticity and eventually a loss of sealing function. This deterioration is frequently associated with the cleaning process, with the combination of aggressive chemicals and elevated temperatures. Failure to replace elastomers in such circumstances can result in more rapid corrosion owing to chemicals being able to access the area between the elastomer and the construction material.

5.4.2 *Surface finish*

All food contact surfaces must be smooth, non-porous and easily cleanable (Holah & Thorpe, 1990), and free from large, randomly distributed irregularities, such as pits, folds and crevices. Good cleanability is the key requirement, and it is generally accepted that this is a function of smoothness, such that the rougher the surface the longer the time needed to clean the surface. A maximum roughness is therefore specified for food contact surfaces by both the EHEDG and the American 3-A organisation (Curiel *et al.*, 1993). *Surface finish* is the term used to describe the smoothness of a material surface. All materials, whether metallic or non-metallic, are subjected to several different processes before achieving their final form, each of which can influence the final condition of the surface. *Surface roughness* (R_a) is defined as the 'average departure of the surface profile from a calculated centreline' (Verran, 2005), and is the most common measure used to define or specify a surface. A probe is scanned across the surface, and a centreline is plotted so that the areas of the profile above and below this line are equal. The R_a value represents the average departure of the profile from this centreline, and is usually expressed in micrometres (μm). The R_a value gives an indication of the amplitude of the surface irregularities, but, as it is based on a linear trace, gives no information on either the three-dimensional nature of the surface or the two-dimensional topography.

Figure 5.2 shows two profiles that have the same R_a value, but very different surface topography. It might be expected that the lower profile would be easier to clean, because the features are larger, whereas with the upper profile there is likely to be an increased risk that soil or micro-organisms could more easily become entrapped within it.

A surface roughness of 0.8 μm is usually specified, with higher R_a values being acceptable if they can be demonstrated to be cleanable. Table 5.3 highlights the effect of different treatments of stainless steel on surface finish. Note that cold-rolled steel has a roughness of 0.2–0.5 μm, and therefore should not need to be polished in order to meet surface roughness requirements.

5.4.3 *Joints*

Joints fall into two categories: *permanent joints*, such as welds; and *dismountable joints*, such as pipe couplings. The general recommendation in the design of joints for pipework systems is that welding should be used where possible, and pipe couplings specified only where necessary, for example where the equipment components may need to be removed for inspection or maintenance.

The primary function of a weld is to provide a permanent joint of the required mechanical strength while meeting any legislative requirements, such as pressure vessel codes. Key hygiene requirements are that welds be smooth and continuous, as poor welding can compromise product quality in an otherwise hygienically designed plant. Welding can be

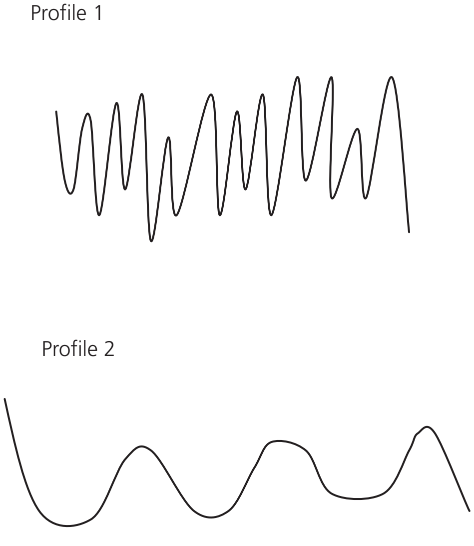


Fig. 5.2 Surface profiles showing different topographies.

Table 5.3 Examples of surface treatments of stainless steel and the resulting surface roughness.

Treatment	R_a (μm)
Cold rolling	0.2–0.5
Hot rolling	>4
Glass bead blasting	1.0–1.2
Descaling	0.6–1.3
Bright annealing	0.4–1.2
Pickling	0.5–1.0
Electropolishing	Depends on original finish
Mechanical polishing with aluminium oxide or silicon carbide of abrasive grit number:	
500	0.1–0.25
320	0.15–0.4
240	0.2–0.5
180	> 0.6
120	> 1.1
60	> 3.5

carried out manually using a tungsten inert gas (TIG) process or, preferably, automatically using an orbital welder. For welding of pipework, the internal surfaces should be purged with an inert gas. For vessels of up to about 4 mm wall thickness, cold-rolled sheet is available, which should have a surface finish well within the 0.8 μm specified. Other than being free from grease and dirt, the weld area does not generally require any special preparation. After welding, the weld must be ground flush and polished with a 150 grit to achieve the required surface finish. For thicker-walled vessels, hot-rolled plate may have to be used, and since this is likely to have surface roughness (R_a) in excess of 4 μm, it would be unacceptable for hygienic operation. Hence the whole vessel must be polished to the required finish after the internal weld has been ground flush with the surface.

For pipework, some key requirements for assuring hygienic welds have been proposed (Anonymous, 1993):

- The pipe diameters must be the same; if not, the smaller pipe should be expanded to match the larger.
- The wall thicknesses should be the same.
- The pipe ends should be cut such that the cut face is at right angles to the longitudinal axis of the pipe.
- Misalignment of the pipes must be limited to 20% of the wall thickness.
- Ideally, there should be no gap between pipe faces, but up to 0.25 mm is acceptable.

The pipe couplings most widely used in the food industry are:

- clamp type to BS 4825-3 (BSI, 1991a)
- International Dairy Federation (IDF), often called International Sanitary Standard (ISS), (BSI, 1991b) or International Standards Organisation (ISO, 1976)
- RJT and SRJT to BS 4825-5 (BSI, 1991c)
- DIN 11851 (DIN, 1964)
- Swedish Metric Standard (SMS)

According to Sillett (2006), the key features for pipe couplings are:

- *Dimensions*. Avoid mixing metric and imperial dimension fittings, as the difference in diameter will create a step in the tube wall at the joint, even if the two halves fit together.
- *Avoidance of uncleanable crevices*. The design of some couplings is such that a crevice is created on assembly, e.g. RJT.
- *Gasket retention*. Where frequent dismantling of coupling is required, a retained-gasket design can prevent the gasket falling to the ground and becoming recontaminated.
- *Gasket materials*. Commonly used elastomers are available depending on the application. Some gaskets, such as the IDF type, are available with a metal reinforcement ring and are less prone to damage, distortion and overtightening.
- *Overtightening*. This can result in the gasket being extruded into the tube bore, creating a step that cannot be cleaned. In addition it can cause premature failure.
- *Misalignment*. Some fittings, such as clamp and IDF, are difficult to reassemble unless the two parts of the coupling are well aligned. Others, such as RJT, can withstand a significant degree of misalignment.
- *Interchangeability*. It is essential to be able to use mating fittings that will fit regardless of manufacturer. Problems are sometimes seen with fitting RJT nuts and male parts owing to different manufacturing methods. Clamp fittings have also frequently been found to be incompatible, and purchase from a consistent supplier is necessary.

Table 5.4 shows how the various fittings compare against some of the major requirements.

5.4.4 *Other constructional features*

Lelieveld *et al.* (2003) extensively reviewed different constructional features, which are briefly discussed as follows.

Fasteners

Exposed screw threads, nuts, bolts and screws must be avoided wherever possible in food product contact areas. Alternative methods of fastening can be used where the washer used has a compressible rubber insert to form a bacteria-tight seal.

Table 5.4 Comparison of different types of coupling.

Coupling type[a]	Crevice present	Gasket retention	Possible overtightening	Misalignment acceptable	Comments
Clamp	No	No	Yes	No	Good joint if correctly installed; interchangeability can be a problem
RJT	Yes	Yes	No	Yes	Unsuitable for pasteurised or sterile circuits, owing to crevice; excellent for flowplates, owing to wide dimensional tolerance on mating bends
IDF/ISS	No	No	Yes	No	Widely used on pasteurised products where dismantling is infrequent; frequent dismantling can cause threads to wear and become sharp
DIN	Yes	Yes	No	Yes	Widely used; crevice can be avoided by use of a modified gasket
SMS	Yes	Yes	Yes	Yes	Becoming more widely used; crevice can be avoided by use of a modified gasket; does not require axial displacement to break the joint

[a] Refer to text for further information.

Drainage

All product contact surfaces should be self-draining, as any residual fluids can lead to microbial growth or, in the case of cleaning fluids, result in contamination of the product (Anonymous, 1983a). An additional consequence of failure to drain plant and equipment fully may be an increased corrosion risk, particularly if there is a source of heat present during some or all of the production process. An example is a sterile tank for a UHT application, where an external jacket is incorporated to cool the tank after steam sterilisation. If the jacket cannot be fully drained of cooling water, the steam sterilisation process will heat the metal to 120–130°C and cause some of the residual water to evaporate. This evaporation will result in any chloride ions present being concentrated, which in combination with the elevated temperature may substantially increase the corrosion rate if the cooling water has a chloride level of 50 mg L^{-1}, which is generally considered suitable for service water applications. Figure 5.3 shows how the concentration increases as more water is evaporated, and the combination of elevated temperature and concentration can create an aggressive corrosion condition. This is likely to pose a greater risk when the residual water level is small, and hence the amount of water evaporation required to increase the concentration significantly is also small.

Failure to drain fully can also pose the risk of microbiological growth, particularly if the system is left unused for sufficient time to allow growth to occur. If the installation of a

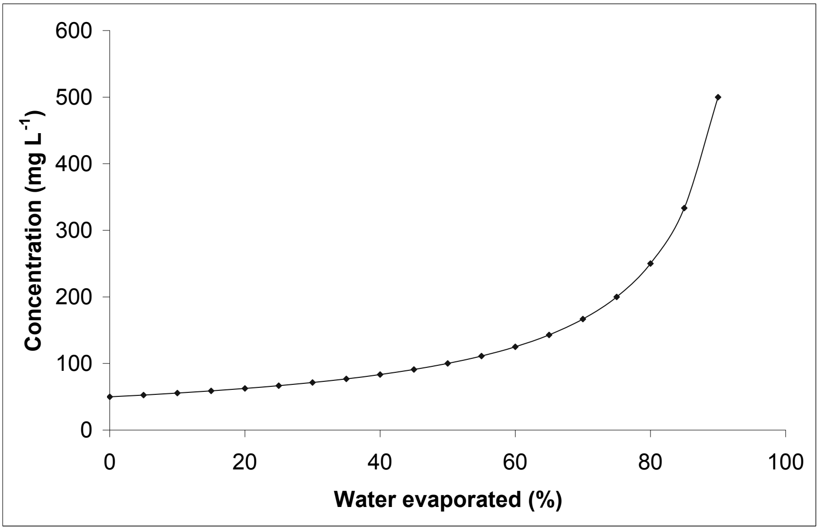


Fig. 5.3 Effect of evaporation on residual solute concentration.

line is such that complete drainage may not be achieved, it may be preferable to leave the system full of water after CIP rather than attempt to drain it. If the water contains a low level of preservatives, such as potassium sorbate and lactic acid, these will help to minimise the risk of microbial growth occurring during the time the process line is not running, and they can be rinsed out immediately prior to production.

Internal angles, corners and dead spaces

Angles and corners should be well radiused to facilitate cleaning. Corners should preferably have a radius equal to or larger than 6 mm, with a minimum radius of 3 mm (Figure 5.4). Where the radius of a corner has for technical reasons to be less than 3 mm, the design must be such as to compensate for the reduced cleanability. Sharp corners (< 90°) must be avoided wherever possible, although in some cases, such as positive pumps, this is not practical.

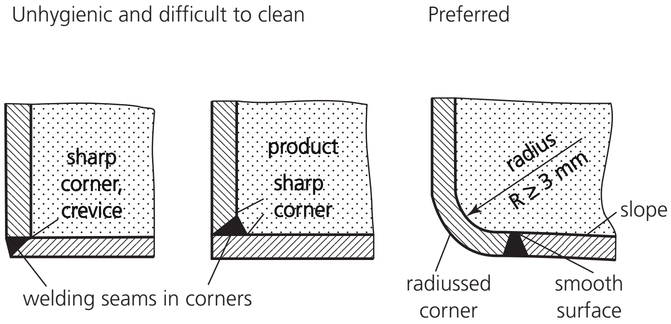


Fig. 5.4 Unhygienic geometries and preferred solutions.

Dead spaces are areas outside the main bulk product flow where product can accumulate at the beginning of production, and may remain for an extended period of time. If the environmental conditions, such as temperature, are favourable, microbial growth may occur, resulting in recontamination of the bulk flow. It may also be difficult to clean and disinfect or sterilise such an area after production has finished, owing to the lack of fluid movement within the dead space. Most such problems occur as a result of the physical assembly, installation and integration of individual equipment components with connecting pipework: examples are T sections in pipes, incorrect orientation of equipment, and installation of sensors into pipework.

Tamplin (1990) reported a flow visualisation study that was the first work to highlight the significance of dead spaces in pipework for cleaning-in-place. This investigated both vertical and horizontal tees with one of the branches blanked off, thus creating a dead space. Different orientations and dimensions of the dead space were studied under bulk fluid velocities of between 0.3 and 1.5 m s^{-1}, and the results are summarised in Table 5.5. This clearly showed the importance of both fluid velocity and the geometry of the dead space, with, in some cases, the highest flow velocity being unable to clean the dead space. Further experimental and theoretical studies have confirmed these findings (Grasshoff, 1980), leading to the recommendation that, where dead legs exist and cannot easily be removed, the flow of fluid should be towards the dead space rather than away from it. The length of dead legs should be minimised, even if they cannot be eliminated.

A common error is to assume that a dead space will still be capable of being thermally disinfected or sterilised owing to heat penetrating into the stagnant zone even if the fluid cannot. A recent study by Asteriadou *et al.* (2006) has shown that, even with turbulent flow within a horizontal pipeline, the temperature within a vertical dead leg will fall rapidly with distance from the junction between the horizontal and vertical sections. This will potentially compromise the thermal process owing to a failure to reach the required minimum conditions.

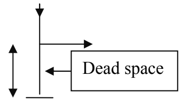
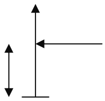
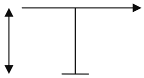
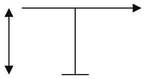
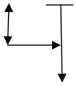
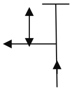
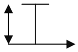
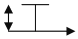
Bearings and shaft seals

Where possible, bearings should be mounted outside the product area to avoid possible contamination of product by the lubricant, or damage to the bearings due to ingress of product. Where bearings have to be located within the product area, such as a foot bearing for an agitator shaft, the design of the shaft and bush must be such as to allow the cleaning fluid access to all the surfaces. Typically, this can be done by providing grooves in the shaft through the whole length of the bush to provide a flow path for product. Seals must be easy to clean and also to remove for maintenance or replacement. Where lubricants are used, they must be cleared as being acceptable for food contact.

Instrumentation

Instruments must be constructed from appropriate materials, and where they contain a transmitting fluid this must be approved for food contact in the same way as for lubricants. Frequently, the problems with instruments lie in the way they are installed rather than their being inherently unhygienic (Figure 5.5).

Table 5.5 Cleaning performance of pipework.

	Orientation	Flow direction	Length (mm)	L/D ratio	Flow velocity ($m\ s^{-1}$)		
					0.3	0.9	1.5
L		Into dead space	127	2.5	−	+	+
L		Away from dead space	127	2.5	−	+	+
L		Across dead space	127	2.5	−	+/−	+
L		Across dead space	76	1.5	−	+/−	+
L		Away from dead space	127	2.5	−	−	−
L		Into dead space	127	2.5	−	−	+/−
L		Across dead space	127	2.5	−	−	−
L		Across dead space	76	1.5	−	−	+/−

Note: + acceptable; +/− fair; − inadequate.

5.5 Cleaning process equipment

Any process line consists of a series of equipment components linked together by pipework. There are many equipment components used in the food industry, and it is not practical to try and cover all types within this chapter. However, some of the most commonly used equipment items are reviewed, highlighting any areas where the general requirements described in Sections 5.3 and 5.4 are not appropriate.

5.5.1 *Effect of fluid flow on cleaning*

The previous section has highlighted the influence of flow velocity on the cleaning of unhygienic geometries, such as dead spaces. Fluid velocity is potentially one of the parameters over which the equipment designer has some degree of control. The other major factors

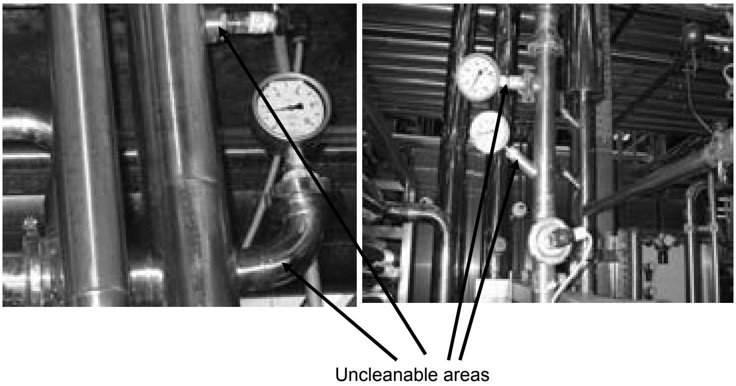


Fig. 5.5 Unhygienic installation of process instruments.

influencing cleaning effectiveness, such as cleaning time, temperature, detergent type and concentration, are more likely to be associated with recommendations from the detergent supplier. Various experimental studies have shown that the fluid velocity of the cleaning solution strongly influences the time to clean. Timperley & Smeulders (1988) studied the cleaning of a plate heat exchanger and showed that the cleaning time decreased rapidly as the fluid velocity was increased above 0.1 m s^{-1} (Figure 5.6). However, the rate of improvement decreased as the velocity increased, with an asymptote occurring at around 1.5 m s^{-1}, and this figure is widely quoted as the target design value. Timperley (1981) suggested that the influence of flow velocity was due to a reduction in the boundary layer thickness rather than an increasing Reynolds number.

There has also been debate about whether the improvement was due to the increased shear forces acting on the deposit attached to the surface of the equipment. Hasting (1988)

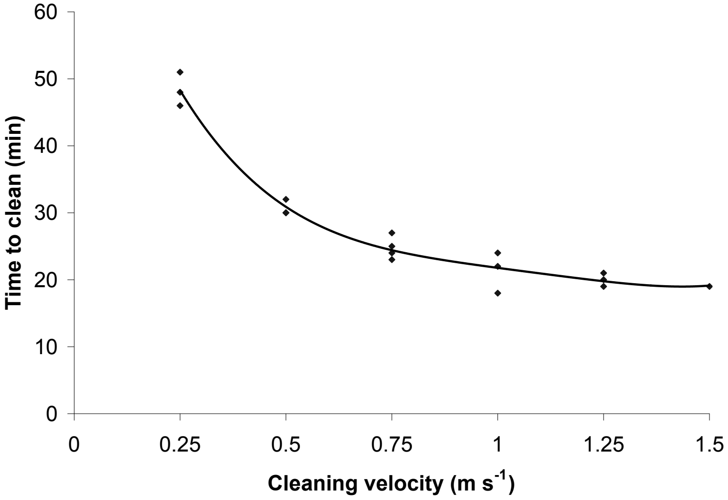


Fig. 5.6 Cleaning time as a function of cleaning fluid velocity.

concluded that the controlling mechanism was boundary layer thickness rather than shear forces, since if the shear forces were controlling, increasing the fluid velocity should eventually result in an instantaneous removal of all deposits from the surface. He suggested that the boundary layer thickness, and hence the rate of detergent diffusion across the layer, was controlling, particularly at low flow velocities. However, as the velocity increased, the boundary layer eventually became sufficiently thin such that the diffusion resistance was no longer controlling, this point being shown by the asymptote in Figure 5.6. At this point another mechanism would become controlling, for example the reaction rate between detergent and soil. This highlights the complexity of the process, particularly when cleaning heat-induced soils.

The experimental studies have been focused mainly on heat-induced dairy-type soils, and are unlikely to be valid for all soils encountered within the food industry, but nevertheless there are general recommendations that can be made:

- Increasing the fluid velocity is beneficial for the cleaning process; if possible, a design velocity of 1.5 m s^{-1} should be used, unless data for the specific soil indicate otherwise.
- Although 1.5 m s^{-1} may be the optimum velocity in many cases, it is likely that equipment can be cleaned satisfactorily at lower velocities than this, provided a longer cleaning time is acceptable.

5.5.2 *Pipelines*

There are generally two types of circuit within which lengths of pipework have to be cleaned: first, where the circuit is predominantly pipework such as product transfer lines; and second, where the pipework is only a minor part of the circuit, such as in a pasteuriser. In either case the user will not be concerned solely with cleaning straight runs of pipe, but also with more complex geometries, including joints, bends, tees and valves. The design of the cleaning system should be based as far as possible on 1.5 m s^{-1}, which results in substantial flowrates for larger-diameter pipework (see Table 5.6). Hygienically designed pipework can usually be satisfactorily cleaned at lower velocities, at the expense of an increased cleaning time.

When designing pipework for cleaning, the following potential problem areas should be considered.

- Linking several pipelines into a single circuit for cleaning may limit the flow delivered by the CIP pump, owing to pressure drop.
- If pipework has sections of differing diameter, ensure that the smallest-bore section does not pose a severe restriction to flow.
- Pipeline components such as valves and flowmeters can also restrict flow.
- If several lines are to be cleaned in parallel, ensure that the CIP pump has sufficient capacity, and that the arrangement of the line does not lead to maldistribution of cleaning flows, causing poor cleaning in some areas.
- Pay attention to providing adequate pipework support and provision for thermal expansion during CIP.

Table 5.6 Recommended cleaning flowrates for various pipe sizes.

Pipework			Flowrate required ($m^3 h^{-1}$)
Outside diameter (mm)	Wall thickness (mm)	Internal diameter (mm)	
25.4	1.2	23.0	2.2
38.1	1.2	35.7	5.4
50.8	1.2	48.4	9.9
63.5	1.6	60.3	15.4
76.2	1.6	73.0	22.6
101.6	1.6	98.4	41.0
127.0	1.6	123.8	64.9
152.4	1.6	149.2	94.3

5.5.3 *Pumps*

A wide range of pumps are available for use within the food industry. Design for cleanability is an important criterion in pump selection, but in many cases the functional requirements of the pump, such as delivery pressure and flowrate, determine the type of pump used, even if they are not the most hygienic option available.

Generic categories of pumps are shown below:

- peristaltic
- diaphragm
- centrifugal
- positive pump – rotary type (gear/lobe)
- positive pump – reciprocating type (homogeniser)
- positive pump – screw type (mono)

Although all such pumps can be unhygienic if poorly designed, the positive-type pumps are usually considered to be less easily cleaned than the others because of the nature of their design. They are, however, widely used owing to their suitability for particular applications, such as the rotary type for handling more viscous fluids, the reciprocating type for generating the high pressures required for homogenisation, and the rotary screw mono type for gentle handling of products containing sensitive particles.

Generic requirements for pumps include the following:

- Any leakage from the pump body must be easily visible.
- Shaft seals should where possible be of the mechanical type and be easily accessible for inspection and maintenance.
- Passage shapes should be smooth, avoiding sharp changes in cross-section.
- Bearings should be located outside the product area, and should be sealed or be of a self-lubricating type.

- Pumps should be self-draining where possible.

Positive pumps may be either fixed or variable speed. If the pump is a variable-speed type, the speed, and hence the flow, should be maximised during the CIP process. However, even at maximum flow, a positive pump can often create a restriction to CIP flow in the rest of the line, and it may be necessary to be able to bypass the pump so that an increased flow can be achieved. Figure 5.7 shows how this can be achieved in practice. The valve enables the bypass to be opened during CIP to give an increased flow, while the pump can continue to operate, and draws fluid through the pump to clean it. If the CIP flowrate is Q and the bypass flow is B, the total CIP flow is $Q + B$. The four-port valve can also act as a pressure relief valve during production, so that if the pump discharge pressure becomes too high, the valve seat will lift, the pressure will relieve, and fluid will circulate around the pump. The arrangement is hygienic, as there is no stagnant area created by the installation of the four-port valve.

5.5.4 *Valves*

A wide range of valves are available for different applications within the food industry, such as control of product routeing, flow control and overpressure protection. Generic requirements for valves include the following:

- It must be possible to drain a valve completely in at least one installation position without the need for dismantling.
- Valve seats should ensure an effective seal where full shut-off is required. They should also be designed to resist wear, and allow regular maintenance, since wear or distortion may affect cleanability and hygiene.

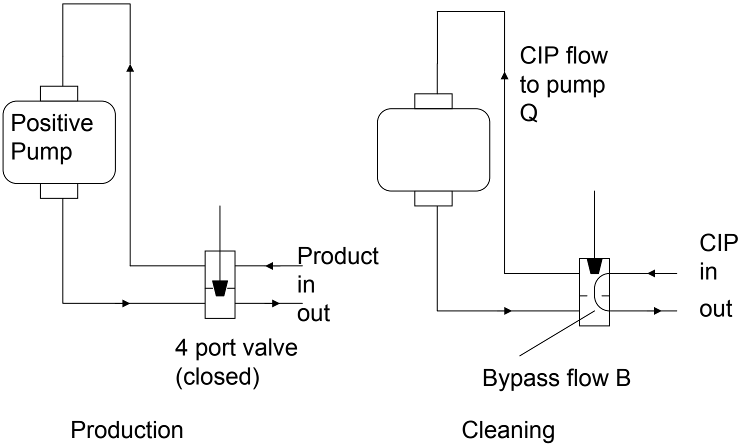


Fig. 5.7 Use of a bypass to allow increased cleaning flows.

- Seals should be positively retained to avoid distortion, and be flush with adjacent surfaces.
- The number of seals in a valve should be minimised.
- Springs in product contact should be avoided where possible but, where this is unavoidable, should have minimum surface contact.
- The valve design must provide for rapid external detection of internal leakage.
- For aseptic applications, dynamic seals on valve shafts in contact with product must provide a barrier between the product and the environment to prevent microbial recontamination. This can be achieved by using a diaphragm- or bellows-type seal. Where the stroke of the shaft is too great to enable such a seal to be used, it is necessary to use a double-seal arrangement. Such a seal has the following requirements: (a) the distance between the two seals must be greater than the distance moved by the shaft, in order to prevent micro-organisms being brought into the aseptic zone during operation; and (b) the space between the seals must be capable of being sterilised prior to production and maintaining asepsis during production. This is usually achieved by flushing the space between the seals with pressurised steam and monitoring the temperature.

Specific requirements for valves are shown in Table 5.7.

Table 5.7 Hygiene requirements for different valve types.

Valve type	Requirements
Pinch/diaphragm	Leakage detection essential, otherwise potential source of contamination if diaphragm damaged. Can be used as a back-pressure valve.
Linear plug valve	Shaft seal can be diaphragm or lipseal type; diaphragm preferred for aseptic applications.
Mixproof (e.g. double seat) valve	Vent space between seats must be drainable to atmosphere, cleanable, and designed to prevent build-up of pressure in case of a leak from the seat. For aseptic applications, the vent space must be flushed to prevent ingress of micro-organisms. Outlet of vent line should be visible so leakage can be easily detected.
Butterfly valve	Regular maintenance essential as the circular rubber seal can deteriorate with use, creating cavities that may be uncleanable. Not suitable for aseptic applications.
Ball valve	Traditional ball valves unsuitable for CIP because area between ball, housing and seat faces is uncleanable. New designs claiming to overcome these limitations are becoming available.
Plug cock	Manual valves; unsuitable for CIP, but generally easy to dismantle for manual cleaning.
Pressure relief valve	Must be self-draining to prevent accumulation of product residues. It must be possible to lift the valve seat so that this and the outlet pipework can be cleaned. For tank applications it is necessary to ensure that spray devices are installed so that cleaning fluid can access the valve.
Non-return valve	Valve must close when the pressure on both sides of the valve is equal.

5.5.5 *Heat exchangers*

Heat exchangers are available in a variety of geometries, all of which are designed to maximise heat transfer and product quality in the most cost-effective way. Their selection will generally be strongly influenced by the characteristics of the product being handled, especially the viscosity and the presence or absence of particles. Three main types of heat exchanger are commonly used, each of which poses specific design challenges with regard to cleaning.

Plate heat exchangers (PHE)

This is probably the most widely used type of exchanger in the food industry, and has been the subject of continuous development and improvement over the years. The main features from a cleaning perspective are the relatively narrow gap between the plates, and the large number of metal-to-metal contact points in the flow channels. These contact points provide the mechanical strength necessary to withstand the required operating pressures. General hygienic design requirements do not recommend metal-to-metal contact owing to concerns about retention of soil and micro-organisms within the metal-to-metal contact area. As an example, a 4000 L h^{-1} capacity plate pasteuriser for an emulsion product may have nearly 100 000 metal-to-metal contact points on the product side. While this may be non-optimal from a hygienic design perspective, the key issue is whether the exchanger can be cleaned effectively. Practical experience gained over many years has shown that PHE can be cleaned consistently to a standard compatible with the highest hygiene standards, such as aseptic processing, provided an appropriate cleaning regime is delivered.

The most critical parameter in cleaning PHE is the fluid velocity. Figure 5.6 clearly shows this, but a key point to note is that the fluid velocity within a plate heat exchanger is generally low owing to the narrow gap geometry. For example, in a milk pasteuriser, the product velocity during production may be as low as 0.1–0.2 m s^{-1}, particularly when the pasteuriser is designed for high levels of heat recovery. This velocity would then have to be increased significantly in order to achieve the optimum cleaning velocity, and this will have an impact on the pressure drop through the exchanger.

The pressure drop (ΔP) through the heat exchanger is a function of the velocity (v) and friction factor (f), such that

$$\Delta P = K \times \left(f \times v^2 \right)$$

where K is a constant.

The friction factor f is, however, proportional to $v^{-0.25}$, and hence the pressure drop is proportional to $v^{1.75}$. Figure 5.8 shows how the pressure drop will increase relative to the production pressure drop as the fluid velocity is increased, assuming the viscosities of product and cleaning fluid are the same. This shows that the pressure drop at 1.5 m s^{-1} will be about 60 times that during production, which is impractical, because even if the increased flow could be delivered by the CIP pump, the exchanger could not withstand the internal pressure generated. However, even if the full 1.5 m s^{-1} cannot be delivered, it is important to design in the capability to maximise the flow velocity during cleaning, since even a relatively

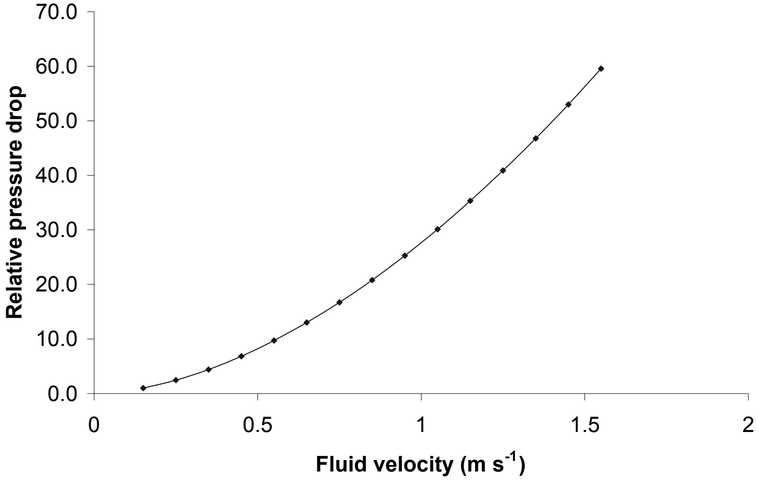


Fig. 5.8 Effect of increased flows on pressure drop.

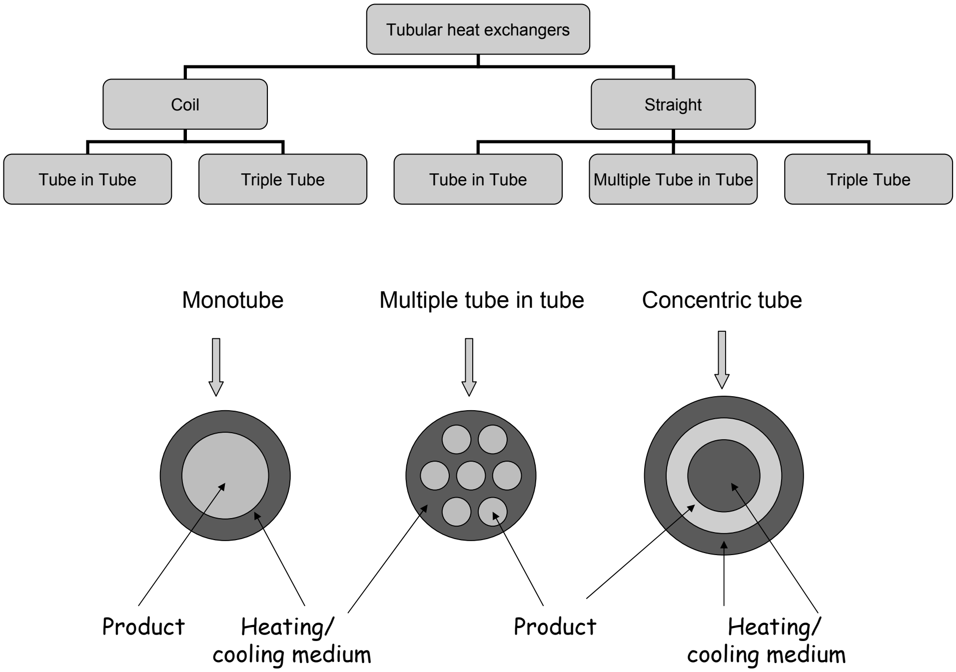
modest increase in velocity from 0.15 to 0.3 m s^{-1} will provide a significant improvement in cleaning. This is particularly important for exchangers handling higher-viscosity products, since the design velocities in these applications may well be significantly lower than for low-viscosity fluids.

Other potential ways of increasing flows during cleaning are to bypass sections of the exchanger at stages during the cleaning cycle. For example, in a milk pasteuriser, circulate all the CIP fluid through the diversion line for a period of time. This will clean the diversion line but, by bypassing the regenerative cooling and final cooling sections, it should be possible to achieve an increased flow.

For applications where PHE are to be considered for processing a product containing particles, such as fruit juice with fibres, special designs termed *wide gap* or *free flow* are available. These have a significantly wider gap than a conventional plate exchanger (~6 mm compared with 2–3.5 mm), and there are few contact points within the product flow channel. This does, however, have a detrimental impact on the maximum working pressure, which may be as low as 0.4–0.5 MPa compared with 2 MPa for some conventional exchangers. The same principles in terms of fluid flow apply for cleaning; in addition, with particulate products, it has been shown that using a combination of forward and reverse flow can be beneficial.

Tubular heat exchangers (THE)

Tubular heat exchangers can be generally characterised as shown in Figure 5.9. The heat transfer surface can be based on plain tube, or an extended surface in which the tube is fabricated so as to give a corrugated form, which is claimed to enhance turbulence and hence heat transfer. Specific design issues affecting equipment cleanability are as follows.



All flows of product and heating medium are counter current

Fig. 5.9 Classification of tubular heat exchangers.

- Some designs of coiled tube contain spacers to maintain the correct dimensions in the gaps between the different diameter tubes; these are potentially difficult to clean if on the product side.
- Coiled tubes cannot be visually inspected or dismantled: hence if severe fouling occurs, resulting in blockage of the flow gap, it will be very difficult to clean the system.
- Straight-tube designs are usually supplied in 3- or 6-m modules, which can be linked together to create the required system and are installed horizontally. Extended surface tubes installed horizontally cannot be fully drained and, depending on the depth of the corrugation, may be difficult to drain fully even if installed at a slight angle to the horizontal.
- The modules can be linked together by pipework, for example 180° bends, to create a complete system. The cross-sectional area of the pipework may well be greater than that of the product side of the exchanger, and hence it is necessary to take this into account when specifying the required CIP flowrate.
- Triple-tube units are the most complex of the straight-tube geometries, and particular attention must be paid to the design of the inlet and outlet areas, including seals, to ensure that these do not result in stagnant areas where product can accumulate and create difficulties during cleaning.

- Straight-tube designs, such as single and multiple tube-in-tube, can be visually inspected by removing the end connections from the module, which then gives access to the inside of the tubes. Many designs are also of the floating-shell type, such that the inner tube or tubes can be removed from the outer shell so that the outer surface of the tubes and the inner surface of the shell can be inspected and if necessary manually cleaned. In order to be able to carry this out, sufficient space must be provided to allow the inner tube(s) to be removed from the shell.

Scraped surface heat exchangers (SSHE)

This class of heat exchanger is particularly applicable for heating and cooling duties where high-viscosity products have to be processed, or where the products contain particles. The heat exchanger consists of a jacketed cylindrical tube of diameter up to 300 mm with a rotating shaft along the centreline of the tube. A series of blades are fixed to the rotating shaft, and these continually remove product from the wall to ensure uniform heat transfer to the product. Heating or cooling media flow through the external jacket. The exchangers may be of vertical or horizontal design. Different configurations of rotor and blades can be used, depending on the application. Choice of materials is important for such units, because the rotating blades come into very close proximity to the internal surface of the cylinder, and wear of both blade and cylinder can be a problem. Damage to either can result in a physical contamination hazard for the final product.

Cleaning of SSHE can pose problems owing to the relatively large diameter of the exchanger, such that the cross-sectional area is generally much greater than the inlet and outlet pipework. This leads to very low linear flow velocities within the machine. The rotating shaft and blade system will assist in creating high turbulence at the internal surface of the wall, but there are other areas of the machine that may not be subjected to such turbulence. These include the inlet and outlet zones, the seal areas and the shaft itself, and are all known as potential problem areas. System design should therefore aim to try and maximise cleaning flowrates. This can, however, be difficult to achieve with a CIP pump that may be cleaning the associated equipment such as pipework and valves, as it may not be possible to provide the required flow. In this case the configuration shown in Figure 5.10 provides a simple and effective way of substantially increasing flows and ensuring effective cleaning. Since the booster pump is installed in parallel to the exchanger, and directly adjacent to it, the pressure drop through the pipework will be small, and will not severely restrict flow. The additional valves are required to enable the booster pump to be coupled in only during cleaning. The rest of the circuit can be cleaned with the usual CIP pump, and the booster pump will just circulate the increased flows around the scraped surface unit. Care must be taken to ensure that the additional valves can be integrated hygienically into the line, and that the recirculation line can be fully drained after CIP has been completed.

5.5.6 *Tanks*

A wide range of tanks are used in the food industry and can generally be characterised as *storage tanks*, whose function is purely storage of raw materials, intermediate or final products at different stages of the production process, and *process vessels*. Process vessels

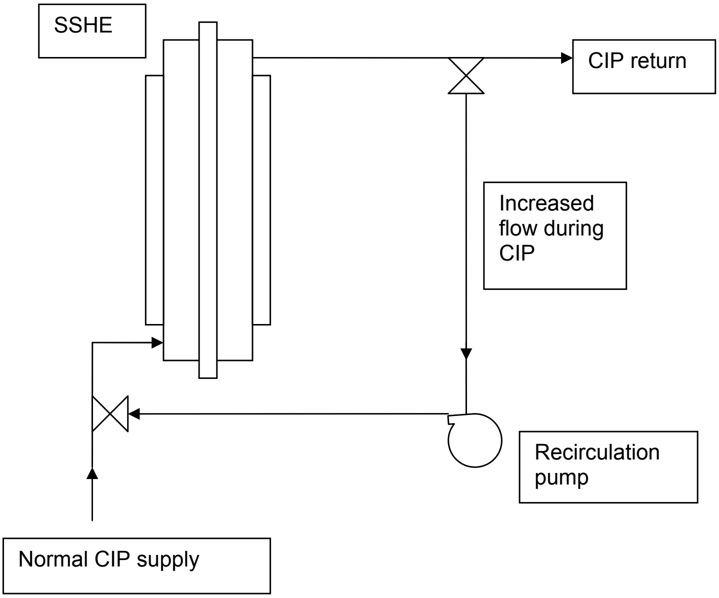


Fig. 5.10 Use of recirculation pump to increase CIP flows.

can include mixing, blending, heating, cooling, separation and fermentation operations, and may therefore be more complex in design than plain storage tanks because of the presence of various internal components. However, the design criteria are the same for all types of tank. Many different internal components can be used within a tank, as shown in Table 5.8, and any such components must conform to the appropriate hygienic design guidelines

Table 5.8 Typical internal components of tanks and process vessels.

Baffles
Man-ways
Sight glasses
Anti-vacuum devices
Pressure relief valves
Product inlet and outlet lines
Vent lines
Instruments
Agitators
Vortex breaker
Heating/cooling coils
CIP spray devices

relating to materials of construction, fabrication and cleanability – in terms both of the individual component and of how the component is integrated into the construction of the overall tank.

Of these components, the spray device may well be of minimal cost in comparison with the overall vessel, but it is the critical design component in terms of ensuring effective cleaning and hence process hygiene. It is therefore essential that the correct cleaning requirements and spray devices be clearly specified to the fabricator at the design stage. Many different types of spray device are available, and they are covered in more detail in a later chapter of this book (see Section 6.5). The design will depend on whether the cleaning requirements for the vessel can be delivered by wetting and irrigation of the surface, or whether there is a need for a greater mechanical impact. Whichever type of device is selected, it is essential that they are installed such that all contact surfaces can be reached effectively. For most vessels with internal components, a single spray device will be insufficient, because the presence of the component is likely to cause 'shadowing' and prevent cleaning fluid from reaching all the surfaces. In certain circumstances spray devices may be submerged below the liquid surface, and hence they must be self-cleaning and self-draining.

One important design point that is often given insufficient attention is removal of liquid from the bottom of the tank during CIP. It is always much easier to pump liquid into a vessel than to extract it, and failure to maintain the vessel empty during CIP may have the following consequences.

- Rinsing may take substantially longer to achieve.
- Poor cleaning of surfaces that are covered by liquid will occur, because flow velocities will be very low.
- Redeposition of soil may occur on the surfaces.

The design of a vessel can be such as to maximise the drainage capability by:

- providing a sufficient slope on the base of the vessel;
- providing an outlet of sufficient size for the cleaning flowrates required;
- selecting an appropriate extraction pump to remove liquid from the tank;
- installing the extraction pump close to the vessel, minimising the number of bends, and avoiding changes in pipework diameter.

5.6 Conclusions

The following aspects are important in cleaning-in-place of processing equipment.

- CIP plays a key role in the delivery of cost-effective process hygiene.
- A system of poor mechanical or process design may be uncleanable, regardless of the cleaning process being applied.
- Consideration of CIP should be integrated into the mechanical and process design at an early stage, rather than as a final process to be incorporated into an already fully specified plant.

- Flow velocity is a critical parameter in providing an effective cleaning process; maximising the fluid velocity is beneficial for the cleaning process, and if possible a design velocity of 1.5 m s^{-1} should be used unless data for the specific soil indicate otherwise.
- Failure to achieve 1.5 m s^{-1} does not necessarily mean that effective cleaning cannot be achieved, but the process is likely to be non-optimal.

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6 Perspectives in Tank Cleaning: Hygiene Requirements, Device Selection, Risk Evaluation and Management Responsibility

R. Packman, B. Knudsen and I. Hansen

6.1 Introduction

The main focus in this chapter is on the engineering and management aspects of the design, specification, selection and application of the types of cleaning-in-place (CIP) device that are most commonly used in the dairy industry. These aspects are then considered in terms of the influence they exert on the effectiveness and operating costs of tank-cleaning processes.

In the long term, there are never any unequivocally ‘correct’ answers in tank cleaning. The technologies available and the processes used are continually evolving and changing, as are our expectations and requirements for what they can achieve. The design and efficiency of tank-cleaning equipment, and the effects that such cleaning procedures have on the processing costs and quality of dairy products, are considerations that have long been underestimated, overlooked or ignored.

This chapter therefore focuses on the questions that are most appropriate for determining whether a specific tank-cleaning device can meet given specifications, and on the analytical tools that are suitable for identifying which tank-cleaning solution is the best in any given context.

6.2 Background

6.2.1 *More than just equipment*

The dairy industries have to deal with increasingly stringent demands regarding product hygiene. Constant improvements in quality and efficiency are also required in order to maintain profit margins. At the same time, the production set-up must be as versatile as possible – designed for rapid, glitch-free changeovers between different products and different product specifications, yet with minimum waste and a minimum of production downtime, which seriously erodes earning potential.

In virtually all kinds of process technology, the ability to maintain high levels of hygiene is crucial for product quality. The speed and efficiency with which cleaning can be done are crucial both for operating costs and for production downtime, and therefore have a significant impact on a company’s profits. In addition, environmental concerns are being given increasing attention, with a focus on recycling water, reducing the use of chemicals, and limiting waste.

The way in which tanks and other dairy processing equipment are cleaned plays an important role in determining the degree to which some of these objectives can be achieved, and optimising CIP cycles is a key part of this.

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6.2.2 *Many aspects of tank cleaning*

Each company has a particular reason for focusing on better cleaning and hygiene, and this focus usually consists of many different components, in a balance that will often vary over time. Some typical concerns include:

- control of the microbiological environment
- the need to ensure sterility for subsequent processing
- the prevention of cross-contamination
- the need to remove sludge and sediment
- effective heat transfer

6.2.3 *Ways to tackle tank hygiene*

Dairy equipment can be cleaned in many different ways, depending on the degree of sophistication and the level of automation required – or available. Two of the prime approaches to tackling tank hygiene are cleaning-out-of-place (COP) and cleaning-in-place (CIP).

Cleaning-out-of-place (COP)

In some circumstances, tanks can be cleaned manually. Typical examples of such procedures include:

- cleaning by hand and brush;
- cleaning by hand using high-pressure spray lances;
- cleaning by filling the vessel with cleaning liquid for washing/boiling (the 'fill, boil, agitate and dump' approach).

However, the human element inevitably associated with manual tank cleaning results in natural limitations to what can be achieved. For this and other reasons, COP procedures are now generally considered incapable of meeting the levels of hygiene associated with commercial operations aimed at the global consumer market. Nevertheless, these systems are still in widespread use, both as legacy installations and in dairy facilities where cleaning and hygiene issues are considered less of a priority – for whatever reasons.

Cleaning-in-place (CIP)

CIP is a method where complete items of plant equipment are cleaned automatically, without dismantling or opening the equipment. This involves little or no manual work. In the modern dairy industry, such cleaning is a high priority and is usually automated as much as possible, in order to eliminate problems associated with human error.

There are three different types of automated CIP equipment normally used for spray-cleaning the insides of tanks, including vessels used in dairies and in conjunction with dairy products. These are as follows.

- *Static spray balls* are normally used where initial purchase cost is the prime focus, where hygiene requirements are limited, and where there is a lack of awareness about the significant built-in limitations of this technology.
- *Rotary spray heads* are normally used for tanks that are relatively easy to clean, and where good hygiene and low overall operating costs are important.
- *Rotary jet heads* are normally used for large tanks that are relatively difficult to clean, and where there is a high degree of awareness about the practical limitations of conventional tank cleaning technology, with consistently high standards of hygiene and low overall operating costs being considered particularly important.

Each of these solutions provides specific advantages in particular installations, but the following is a useful rule of thumb. The static spray ball is the traditional CIP solution, but has been increasingly superseded by more modern, more economical and more effective installations. Furthermore, rotary spray heads and rotary jet heads provide the key advantage of avoiding the use of large volumes of chemicals and cleaning liquids, and thus cut down on operating costs and cleaning time. Rotary solutions ensure a better, more easily controlled cleaning effect by making sure that the cleaning liquid impacts directly on the tank wall, in a carefully controlled manner.

6.3 Two basic approaches to tank cleaning

The dairy industry normally uses two basic approaches in the spray cleaning of tanks and vessels: high volume/low pressure and low volume/high pressure.

6.3.1 *High volume/low pressure*

The traditional approach is to use large volumes of low-pressure liquid, sprayed onto the inside of the tank from a static spray ball head. The aim is to ensure that cleaning liquid then flows over the entire inner surface. This means that most of the cleaning actually stems from a flushing and rinsing effect: that is, the jets from the perforated spray ball hit only a certain, very limited number of points on the inside of the tank, and do so at almost the same place every time. In essence, therefore, the cleaning effect stems from the cleaning liquid cascading down the tank wall, and down any components fitted within the tank, as a result of gravity.

However, this is by no means a controlled process. It is normally possible to achieve the required levels of hygiene only by increasing the volume or temperature of the cleaning liquid, the concentration of chemicals, the time spent on cleaning, or a combination of these parameters. The lack of control also means that it is difficult to achieve the kinds of consistent, reproducible cleaning results that are the key to effective quality assurance programmes.

Using large volumes of cleaning liquid has a significant impact on operating costs, as well as on the costs of the infrastructure required to route the liquid to the tank, and then to treat and dispose of the exit products. This high-volume/low-pressure process has only limited effectiveness in removing product build-ups and residues as well as biofilms and bacterial cultures, which often feature a pronounced tendency to adhere to even extremely smooth surfaces. This kind of cleaning is therefore normally unsuitable for cleaning tanks used for processes characterised by the presence of foam and/or high viscosity, thixotropicity or stickiness.

6.3.2 *Low volume/high pressure*

The more modern and more effective method of cleaning is to use an alternative approach that involves directing a smaller volume of cleaning liquid at higher pressure onto the surfaces that have to be cleaned, normally via a controlled spray or jet. The cleaning effect then arises from a scrubbing action caused by the physical impact of the spray or jet, rather than by mere rinsing caused by the sheer volume of liquid. This effect is multiplied when a controlled cleaning pattern ensures that the spray or jet impacts on every single point on the tank's surface. This means that cleaning using these methods usually requires much less cleaning liquid and fewer chemicals, as well as being faster, thus minimising production downtime (see Figure 6.1).

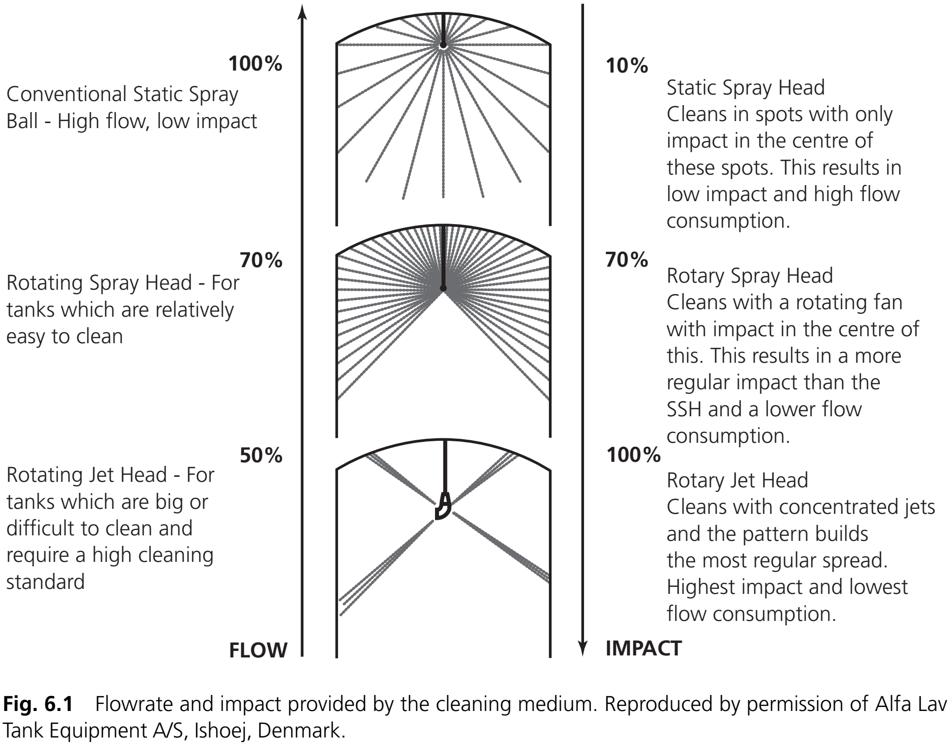


Fig. 6.1 Flowrate and impact provided by the cleaning medium. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

6.4 Key parameters in determining tank-cleaning effectiveness

6.4.1 *The Sinner circle*

In 1960, H. Sinner proposed that the energy necessary to remove soil from a surface of processing equipment can be divided into four contributing factors:

- chemistry (i.e. the composition of detergents);
- heat applied (i.e. temperature);
- mechanical force used (i.e. impact or shear stress);
- contact time of the detergent at the given time and concentration of detergent.

These four key considerations can be expressed in a pie chart, and the role of each parameter can be expressed by the size of its share of the 'pie' (see Figure 6.2). However, the *Sinner circle* has been widely used to describe and understand the influence of the individual parameters, and also in relation to describing and understanding the effect of each of the parameters in terms of operating economics. In addition, the significance of expressing the cleaning parameters in a pie chart is to understand that the different parameters can substitute for each other, within certain limits or restrictions that stem from the nature of the product that has to be cleaned out of the tank.

The Sinner circle has also led to the definition of temperature, action, concentration and time (TACT). This definition has been extended still further to TACCT, where coverage (see Section 6.4.2) is also included. All these aspects have been used in the attempt to obtain a better understanding of the importance, distribution and control of these parameters, and their effect in ensuring that the process equipment is cleaned to the required standard.

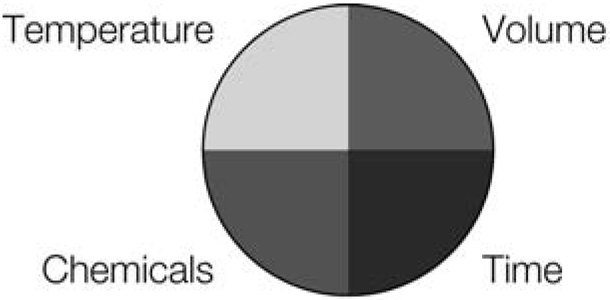


Fig. 6.2 Sinner's circle, illustrating the four basic cleaning parameters. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

6.4.2 *Mechanical forces: hydraulic energy and coverage*

The mechanical forces (e.g. action or impact) involved in in-tank cleaning processes are primarily a function of the total flow of the cleaning liquid, and of the means by which it is applied to the tank walls and internal structures. When simple distribution devices using high flow and lower pressure are used, the mechanical force or the impact or action applied is a result of the cascading flow of the cleaning liquids over the tank surfaces. Rotating types of device utilise the energy of the flow of the cleaning liquids to enhance the impact or action of the cleaning liquids as they are applied to the tank wall. With the use of greater mechanical force, the influence of other parameters can be reduced.

The tenacity of residual product, or fouling, is often used as a design factor for selecting the level of mechanical force or hydraulic energy required in the cleaning process. Experience has shown that biofilms can be removed by mechanical energy when it is applied to tank surfaces by rotating distribution devices.

Coverage is related to the total flow of the cleaning liquid, but coverage is also related to the selection of the particular distribution device in use in a particular tank, and the CIP supply system. It is essential to note that the design of the tank and – in particular – the selection and position of any internal structure influence the effect of the coverage and impact or action. The design of baffles, agitators and top- and side-mounted manhole covers, as well as nozzles on tank tops and sides, can create 'shadows' that impair the action and coverage on some parts of the tank surface. In addition, the position of the distribution device in the tank influences the coverage and action provided by the detergent. The 'shadow' effects and limitations in reach, or effective throw length, for each particular device have to be taken into account.

Direct coverage is the result of the jets or fans of cleaning liquid hitting the surfaces to be cleaned directly, whereas *indirect coverage* is provided either by a splash-back effect or by the effect of a cascading flow moving over the surfaces that cannot be covered directly. In the case of splash-back effects, the need to ensure sufficient splash-back makes it essential to have sufficient impact at the point where the jet impacts directly. In the case of indirect coverage provided by cascading flow, this can be obtained only by increasing the flow. This is normally done by installing additional spray devices.

The *flow and pressure* of the cleaning liquid provided by the CIP system are key parameters in ensuring that the mechanical forces, the coverage and the heat required for cleaning the tank are made available for the cleaning process. In addition, the flow is a very significant cost factor, both for the overall project cost and for the cost of the actual cleaning operations.

The project cost increases when high-flow cleaning devices require larger-diameter pipe, valves, connections and fasteners. The CIP plant required will also require larger tanks, heat exchange surfaces, pumps, pipes, valves and connections. As a result, operating costs increase because of:

- greater energy consumption for transporting larger volumes of liquid
- higher chemical usage due to higher volumes of cleaning liquid
- large quantities used in changeover phases between detergent and rinse water
- greater environmental impact

The effect of these cleaning parameters (i.e. mechanical forces, flow and coverage) is influenced by the design of the tank cleaning set-up as a whole. They must therefore be taken into account as critical design parameters for the CIP supply system and its control equipment.

6.4.3 *System parameters*

The system parameters are influenced by many factors, such as flowrate, time, coverage, chemistry and concentration, process parameters, mechanical or hydraulic energy, action or impact and temperature. Other considerations that can also affect these parameters include the operating parameters (see Figure 6.3), i.e. the distribution of the CIP parameters in systems and process parameters.

The requirement for both sets of parameters and all individual parameters must be specified at the design phase for each tank or process equipment. This applies regardless of whether the design is for an upgrade of an existing cleaning process or for a new installation. In addition, the effect that chemistry and concentration of the detergent, temperature and time of circulation have on cleaning a tank is also influenced by the overall design of the system. Dosing and heating equipment must be designed to comply with the overall requirements of the cleaning process.

6.4.4 *Operating parameters*

The difference in the effect that the individual operating parameters have on the cleaning of a tank can be seen by considering the pie charts (Figures 6.4, 6.5 and 6.6) for different types of cleaning device, i.e. static spray ball, rotary spray ball and rotary spray head, respectively.

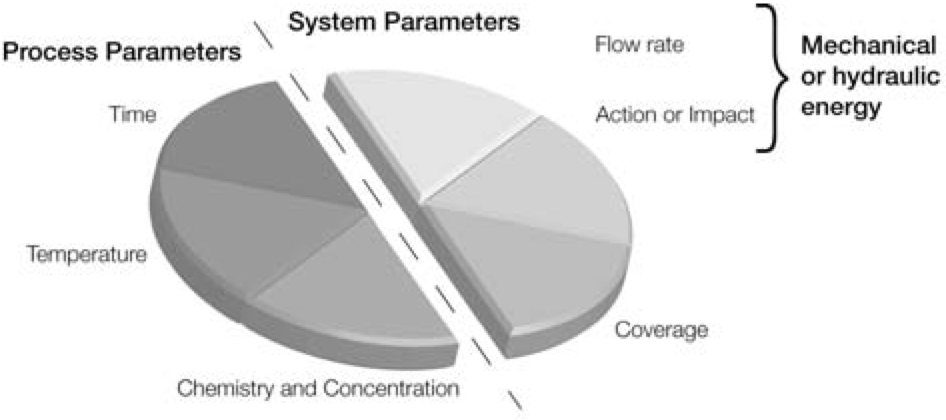


Fig. 6.3 Cleaning systems versus process control parameters. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

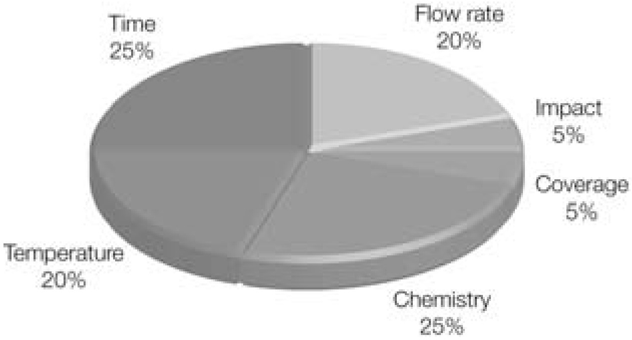


Fig. 6.4 Influence of the key parameters (see text) – static spray ball. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

The distribution is based upon empirical values collected from a range of different tank-cleaning processes in real-world situations.

The influence exerted by the different operating parameters determines the effectiveness and the cost of the tank-cleaning process as a whole. It is therefore the key to understanding, analysing and making decisions about which solution to select for each specific application, both in relation to ensuring a completely clean tank and in relation to the time spent and the quantities of utilities used. These different operating parameters are therefore prime determinants of tank-cleaning costs.

The introduction of more efficient rotary spray heads and rotary jet heads for tank cleaning has added two extremely important additional operating parameters: first, the physical impact of the cleaning liquid (impingement) on the interior of the vessel and all the fixtures

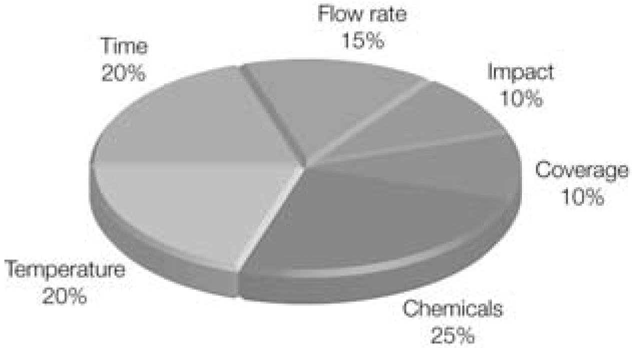


Fig. 6.5 Influence of the key parameters (see text) – rotary spray head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

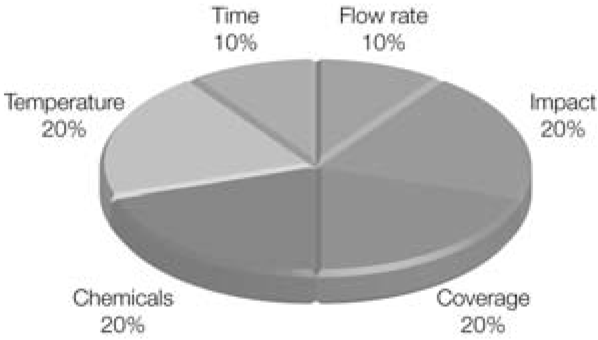


Fig. 6.6 Influence of the key parameters (see text) – rotary jet head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

installed there; and second, how much of the interior the spray or jet of cleaning liquid actually impacts on.

The greater the mechanical impact of the cleaning liquid, and the greater the proportion of the surface that the liquid actually impacts upon, the less liquid and fewer chemicals are needed, and the faster the cleaning process. These two new parameters both dramatically reduce direct CIP costs as well as the substantial indirect costs that result from production downtime.

Consequently, the TACT definition was extended to include coverage, and it is therefore more appropriate that the cleaning parameters should be more precise and defined as TACCT. The growing popularity of rotary spray and rotary jet methods for tank cleaning in the dairy industry came about precisely because operating margins are crucial.

6.4.5 *Distribution device parameters affecting cleaning performance*

There are several parameters for the design of tank-cleaning systems or distribution devices that must be taken into consideration, because they all influence the relative performance of the cleaning device. One key issue is the quality of the jet of cleaning liquid as it leaves the orifice of the nozzle of the distribution device, or the fan of cleaning liquid leaving the slot in the faster-rotating spray head.

Other factors that affect cleaning performance are reviewed in subsequent sections (Sections 6.4.6 and 6.5). These factors are also significant for the selection and positioning of the device in the tank, and play an important role in the total cost of ownership of the distribution device in question.

6.4.6 *Nozzle design*

The mechanical energy, impact, action or hydraulic energy that a jet of cleaning liquid can transfer to the tank surface depends on several factors, all of which can be summarised in the velocity at which the jet is travelling when it impacts on the surface. The *orifice coefficient* depicts the characteristics of the nozzle, and is dependent on several factors, including air entrainment, surface tension, sharp edges and pressure drop in the feed to the nozzle, the roundness of the hole, and the parallel bore as well as the surface finish.

There are distinct differences between the orifice coefficients available from different designs of nozzle, ranging from the holes in a static spray ball to a nozzle specifically designed to ensure a coherent jet of liquid.

6.4.7 *Difference in mechanical energy applied by static spray balls and rotary jet heads*

It is commonly known that the *Reynolds number* (*Re*) can be used to assess whether a flow of liquid is laminar or turbulent, and it is common practice to use 1.5 $\mathrm{m\ s^{-1}}$ as the design velocity in pipes to ensure that the flow in the pipe is turbulent. *Re* can also be used to assess whether the flow and quantity of the cleaning liquid supplied to a tank generate a flow that has turbulent characteristics.

The following information is not intended to be definitive or exhaustive, nor does it apply in every case. However, it does depict the considerable differences in the hydraulic energy that a rotating jet head and a static spray ball deliver in the cleaning process. It also highlights the fact that the absence of mechanical and hydraulic energy is the background for the distribution of the cleaning parameters and their relative size and distribution, and why chemistry and thermal energy play such a significant role in high volume/low impact static spray devices.

Common standards used for practical CIP design use a static spray ball with a nominal *Re* of 2100–2600. This positions the theoretical turbulence of the film flow from a static spray ball or its hydraulic energy only slightly above the laminar flow, and below the turbulent flow.

The equivalent calculation for a rotary jet head shows that the *Re* will be in the range of 30 000–70 000 in the impact area. The velocity at which the jet of liquid hits the tank wall influences the *Re*: high velocity in the impact area results in higher *Re* and in the transfer of more hydraulic energy from the jet to the tank wall.

6.5 Tank-cleaning technologies

When comparing different types of cleaning liquid distribution device with a view to selecting the type most appropriate for a specific installation, it is important to prepare and then apply a set of objective criteria for evaluating these devices.

Such criteria must be comparable to those used to evaluate the processing equipment in general, plus separate and specific criteria for cleaning devices. Specific issues should include:

- the hygienic design standards applied;
- construction details to meet the hygienic design standards for self-leaning capabilities;

- documentation for the self-cleaning capabilities of the materials, metals and polymers used in the construction;
- manufacturing standards and techniques, including casting quality, surface finish, welding specifications and the fastening of individual components.

Furthermore, the cleaning performance relative to the cleaning requirements for the particular tank or processing equipment must be ensured. The requirements listed above constitute the qualification requirement for the device (see Joppen, 2004; Moerman, 2005a, 2005b).

6.5.1 *Static spray balls*

Description

A static spray ball (Figure 6.7) is a ball-shaped sphere with a connecting pipe for input of the cleaning liquid, which is distributed through the holes drilled over the surface in a specific pattern. The static spray ball design is normally available in either thin-walled or thick-walled versions, with wall thicknesses of 1 mm and 2–3 mm respectively. The latter type is normally more durable. The wall thickness is also used to improve the orifice coefficient of the static spray ball.

Fig. 6.7 A static spray ball. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

The drilling patterns are described as either 360°, 180° downwards or 180° upwards. However, the drilling patterns can also be directional in cases where the aim is to ensure direct coverage of a certain part, component or connection to a tank. Static spray balls are made from two halves of a sphere, welded together after drilling. They are also available without drilled holes. The process involved in determining where the holes have to be drilled to ensure complete coverage of all the parts of the tank, component or connection can be very labour intensive and time consuming.

How they work

Spray ball cleaning devices are the simple, straightforward and cheap solution for cleaning the inner surfaces of tanks and similar vessels. The idea was first introduced in the food industry almost a century ago and has been considerably refined since, although it still has inescapable built-in limitations. Spray ball cleaning solutions provide a simple, low-cost way to distribute cleaning liquid onto the inner surfaces of the tank, and are available from many different manufacturers, and in a wide range of shapes, sizes and capacities.

In effect, the static spray ball design washes the inner surfaces of the tank, as a result of the small jets that emanate from each hole in the perforated ball always hitting the internal surfaces of the tank at the same place throughout the cleaning cycle (Figure 6.8). The distance between jets may only be 10 mm at the ball, but by the time these jets have travelled across the inside of the tank the pattern mesh has often opened up to more than 100 mm. For the tank surface in between these impact points, cleaning relies solely on a cascading flow to ensure the requisite coverage and agitation.

This, in turn, means that the desired cleaning effect can be achieved only by using large volumes of cleaning liquid, high chemical concentrations, high temperatures and extended cleaning time, or a suitable combination of these four important parameters.

Unfortunately, any particles circulated in the cleaning liquid can accumulate in the static spray ball. This can result in blockages that generate shadowing areas on the inner surface of the tank.

Rotary spray discs work on the same principle as spray balls, spraying cleaning liquid onto the upper inner surfaces of the tank so that this liquid then cascades down with a washing and rinsing effect.

Mounting

Static spray balls are normally mounted on a downpipe using a standard threaded connection or a pin/clip-on connection. Multiple static spray balls are often used to provide as much direct coverage as practically possible.

Applications

Spray ball cleaning devices are traditionally very popular as distribution devices for cleaning liquid, because they are simple to size, select and install. Because they require only limited investment, they have found their way into almost every application in the dairy industry. Static spray balls are most frequently used in tanks used for low-viscosity products.

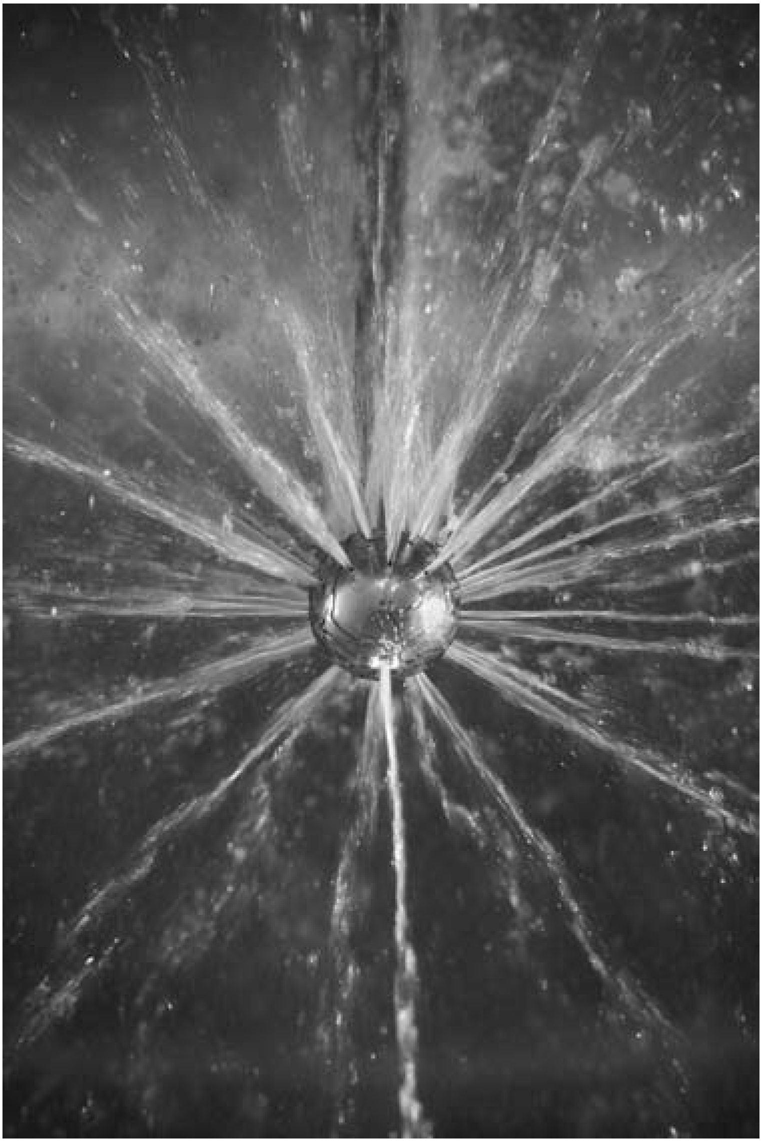


Fig. 6.8 The jetting and the flow of water and detergent through a static spray ball. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

With tanks that hold products that are more difficult to clean, other types of distribution device are normally used, because the limitations of static spray balls mean that the required cleaning standards are unobtainable.

Advantages

Traditional spray ball cleaning devices are normally considered to have certain advantages and benefits. As demands regarding the cleaning performance of the CIP systems and the distribution devices increase, more thorough studies and greater accumulated experience reveal that some of the perceived benefits of static spray balls do not, in fact, apply in real-world installations.

A static spray ball provides neither 360° coverage nor three-dimensional impact coverage. The significance of this has been identified in the inability of such devices to remove biofilm from tank surfaces, and to prevent the biofilm from building up again without the disadvantage of using additional detergent, temperature and time (see Figure 6.4). Nevertheless, the main advantages of the static spray ball are:

- no moving parts;
- low-maintenance operation;
- low initial purchase price, especially if the tank design is simple;
- low pressure effect;
- ease of installation.

Disadvantages

Disadvantages of the static spray ball include the following.

- Investment cost, including connections and tank penetrations, can be high, especially in installations involving multiple units that require large volumes of water and/or high chemical concentrations, in addition to requiring extended cycle times to obtain the desired cleaning effect.
- Particles from the cleaning liquid tend to accumulate inside the spray ball, causing shadow effects on the tank wall. These are difficult to predict or monitor.
- It is unable to deal with biofilms or pathogenic bacteria, such as *Salmonella* spp., *Listeria* spp. and *Campylobacter* spp.
- It offers limited flexibility regarding the types of product that can be cleaned.
- The operating costs are quite high.
- It lacks self-cleaning capability as regards the external surfaces.

6.5.2 *Rotary spray heads*

Description

Rotating spray devices (Figure 6.9) are available in many different designs, but basically the design consists of an inlet pipe that distributes cleaning liquid into a rotating unit. The rotating part can take various shapes or forms – for example, rotating rings with holes, rotating spheres with slots, or combinations of these. Discs or saucers are also used as a distribution device. Properly designed rotating spray devices ensures full direct coverage of the spray

Fig. 6.9 SaniMidget – rotary spray ball. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

pattern within a processing tank. All devices rely on using a reaction force to rotate the distribution part around the inlet pipe; however, the spray patterns within a tank range from 360° to 180° up and down as well as 270°. The bearing support for the distribution device is made up of a ball bearing, or a slide bearing with bearing surfaces, or by using different materials that can slide against each other.

There are several designs and construction parameters for rotating spray heads, which affect the ability of the spraying device to ensure a clean tank. Some examples are as follows.

- Stable rotation of the dynamic part – the spray pattern should allow for complete coverage of the surfaces of the tank when designing the device. Full coverage in the vertical upward direction to cover the entire top of the tank and the downpipe (i.e. the inlet to the tank) must be achieved, because these parts of the tank are the most difficult areas to clean.
- Materials used in construction must comply with both food processing and CIP requirements.
- The design must comply with hygienic design standards.
- The internal and external parts of the cleaning device, as well as the bearing construction, must be self-cleanable.
- It must be self-drainable.
- Wear characteristics of bearing surfaces – sliding or rotating.
- The design of the rotating spray device and the means of the distribution of the cleaning liquid influence the cleaning performance; stable rotation of the distribution device is an essential performance feature.

- The position and size of the slots must ensure that the rotation speed of the dynamic device does not exceed a certain limit, in order to ensure that the coverage and impact on the tank surface reach a maximum.

How they work

Rotary spray heads provide a tank cleaning solution that is significantly different from conventional static spray balls. A rotary spray device applies the cleaning liquid in a ‘fan’ pattern that is determined by the specific design of the head. This means that the spray of cleaning liquid hits all the inner surfaces of the tank in a controlled, consistent pattern. It is the combination of this pattern and the physical impact of the ‘fans’ or jets of cleaning liquid that removes any deposits in the tank, and does so more rapidly and with less water and chemicals than with a static spray ball (see also Figure 6.5).

The operating pressure for rotary spray heads is normally 0.2 MPa. The rotary devices are reaction driven, propelled by the flow of the cleaning liquid passing through the device and then out through slots cut into the head at a tangent (see Figure 6.10).

Mounting

Rotary spray heads are normally mounted on a downpipe using a standard threaded connection, pin/clip-on connection or welding connection.

Applications

Rotary spray heads are most frequently used in applications where the product is relatively easy to clean inside the tank. However, good cleaning performance is the crucial criterion.

Advantages

Rotary spray heads are normally considered to have the following advantages:

- reduced flowrate compared with static spray balls
- lower overall project and installation cost
- reduced cleaning time
- reduced load on the CIP plant
- reduced consumption of water, chemicals and energy
- lower operating costs
- performance can be monitored by measuring the flowrate and pressure
- improved cleaning performance
- better quality assurance for the products being processed.

Disadvantages

Disadvantages of rotary spray heads include the following.

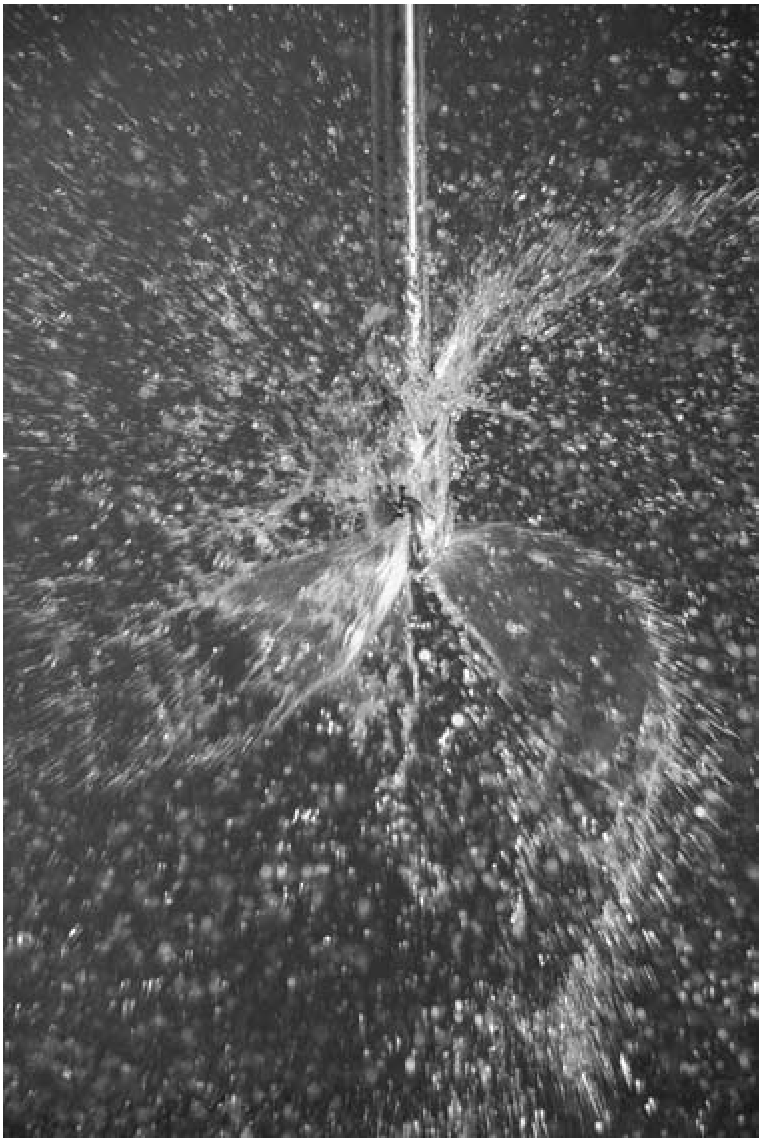


Fig. 6.10 The fans of cleaning liquid distributed by a SaniMidget – rotary spray head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

- Inadequate design impairs the rotation of the cleaning device and limits the coverage it provides.
- Poor design reduces the impact and the cleaning effect of the detergent solution.
- The use of moving parts means that the operating and maintenance schedules laid down by the manufacturers of these parts have to be complied with in order to ensure optimum cleaning performance.

Fig. 6.11 A rotary jet head distributing the cleaning liquid through four nozzles – SaniJet 25. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

6.5.3 *Rotary jet heads*

Description

Rotary jet heads (Figure 6.11) distribute the cleaning liquid onto the tank wall through a number of nozzles – normally between two and eight. These nozzles are mounted on a special hub that is made to rotate by the cleaning liquid passing through the turbine and by reduction gearing running through a condensing indexed cleaning pattern. This guarantees that all the internal surfaces of the tank get covered with cleaning liquid.

The nozzle hub and the body have different gear ratios, which ensures an indexing of the pattern mesh during a double rotation of the body and the nozzle hub. The rotations of both the body and the hub are driven by means of an external motor, running on either compressed air or electricity, or by means of a turbine drive that generates the rotation from the flow of the cleaning liquid passing through the rotating jet head to the nozzles.

How they work

The cleaning effect provided by rotary jet heads is the result of the jets of liquid from the nozzles rotating inside the tank in a carefully planned manner, so that the jet progresses over the entire surface in a successively denser pattern. Modern rotary jet heads use the cleaning liquid to drive the cleaning nozzles in a geared rotation around both the vertical and horizontal axes. In the first cycle, the nozzles lay out a coarse pattern on the tank surface; in the subsequent cycle they ensure that the spraying pattern is gradually denser, until a full pattern is reached – normally after eight cycles. A typical cleaning pattern for a vertical tank is illustrated in Plates 2 and 3.

The selected nozzle diameters determine the length of the jet in order to ensure optimum impact and flowrate at any required pressure, and an electronic rotation sensor monitors that there is full 360° coverage of the tank surface (see Figure 6.12).

The rotary jet head is particularly useful in more complicated tanks and vessels equipped with agitators, baffles and other internal fittings. This is because one single rotary jet head enables the cleaning jets to impact on the numerous angled surfaces from two different angles during rotation, thus eliminating issues related to shadow areas.

The gearing between the turbine shaft and the body of the cleaning machine is normally ~1000:1, which means that the body rotates at 2.5 revolutions min^{-1} (rpm) when the turbine shaft rotates at a speed of 2500 rpm. The flowrate, throw length, nozzle revolution and cleaning time required are determined by the nozzle size and pressure of the cleaning liquid. Normal operating pressure is ~0.5 MPa.

Mounting

Rotary jet heads are normally mounted on a downpipe using a standard threaded connection or a sanitary welding adaptor, and they can normally be mounted vertically downwards as well as vertically up. The vertical position is important for ensuring full drainability after CIP cleaning.

Applications

Rotary jet heads provide high-impact cleaning, and full coverage at a throw length of 2–16 m. They are normally used with cleaning liquid flowrates of 2–30 $\text{m}^3\ \text{h}^{-1}$, depending on the tank size, and at a pressure range of 0.3–0.8 MPa, with a recommended operating pressure of 0.5–0.6 MPa.

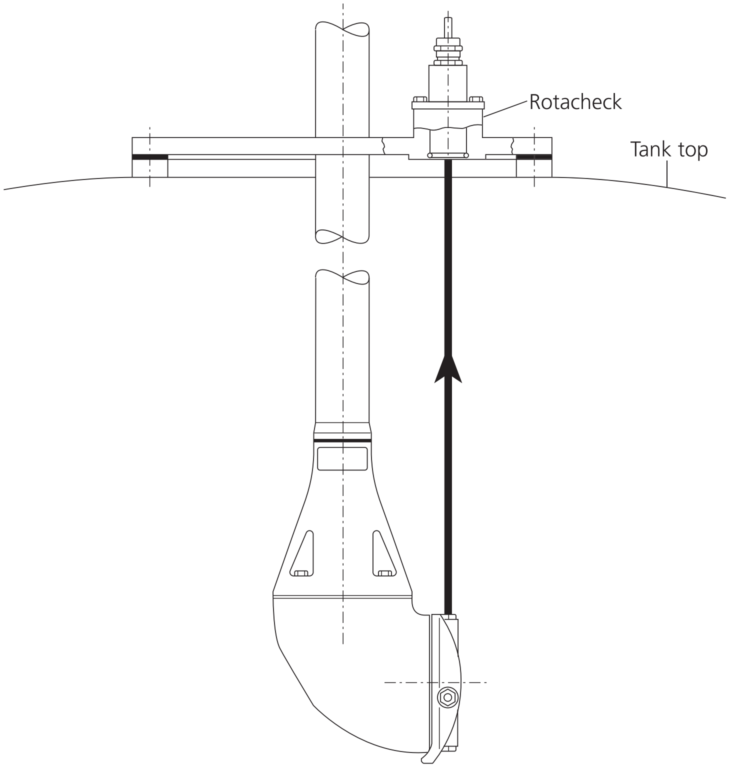


Fig. 6.12 Rotacheck sensor and rotary jet head TJ20G. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

Rotary jet heads are most frequently used in CIP cleaning applications where there is a requirement for complying with good cleaning practices to ensure hygienic cleaning standards with a high degree of reproducibility. These types of head are also widely used in CIP cleaning applications involving products that are difficult to clean.

In addition, the use of rotary jet heads will provide a cost-effective alternative to any other cleaning devices in almost any CIP application. In most cases the focus on monitoring is a result of a desire to improve the cleaning standard. Improving cleaning performance and reducing costs very often go hand in hand when using rotary jet heads (see Figure 6.13).

Advantages

The main advantages of rotary jet heads include:

- low flowrate requirement compared with static spray balls and rotary jet heads;
- low total installation cost, because low flowrates require pipes and valves with less demanding specifications;
- short cleaning time;

Fig. 6.13 TJ20G – rotary jet head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

- increased up-time;
- reduced processing cycle turnaround time, boosting productivity;
- lower overall operating costs, with low consumption of water, chemicals and energy as well as reduced load on the CIP plant;
- removal of any in-tank residues during the pre-rinsing cycle;
- less process residue remaining to contaminate detergents;
- less dilution of the cleaning agents in the CIP tanks;

- more effective cleaning, resulting in improved standards of hygiene and better product quality;
- full three-dimensional impact coverage;
- the ability to monitor cleaning operations;
- fewer cleaning heads needed, resulting in reduced project costs;
- design details focused on achieving better hygiene;
- self-cleaning, both externally and internally.

A typical illustration of the rotary jet head is shown in Figure 6.11 (see also Figure 6.6 regarding the parameters of the cleaning action).

Disadvantages

The disadvantages of the rotary jet head include the following.

- The need for higher pressure requires consideration in retrofit projects.
- The use of moving parts requires compliance with manufacturers' maintenance guidelines.
- The initial investment in cleaning heads is higher.

6.5.4 *Cleaning tanks that include internal fittings and other equipment*

In modern dairy installations, cleaning the tank itself is frequently only part of the production process. In most tanks there is also a wide range of process equipment. including mixers, agitators, baffles and sensors, fitted within the tank itself. In order to maintain the requisite levels of hygiene, this process equipment must be cleaned just as effectively as the tank walls, with only a minimum of extra time, consumables and energy inputs being expended to do so.

The configuration and positions of these mixers, agitators, baffles and sensors must be taken into consideration when positioning the tank-cleaning device within the tank.

Static spray balls and rotating spray heads have to be considered as spraying devices from a single point. If these types of device are selected for the in-tank cleaning applications, tanks that feature internal structures and components will require additional units to achieve the requisite cleaning effect (Figure 6.14).

By contrast, the jets from a rotary jet head must be considered as sprayed tangentially from a ball shape, and the radius of the ball shape is the radius from the vertical centreline of the rotary jet head to the centreline of the nozzle.

This means that the jets from the rotary jet head will be able to clean around an agitator shaft or clean on both sides of a baffle. However, this type of cleaning head cannot clean around, for example, an agitator shaft that has a diameter larger than the distance between the centreline of the rotary jet head and the jet (Figure 6.15).

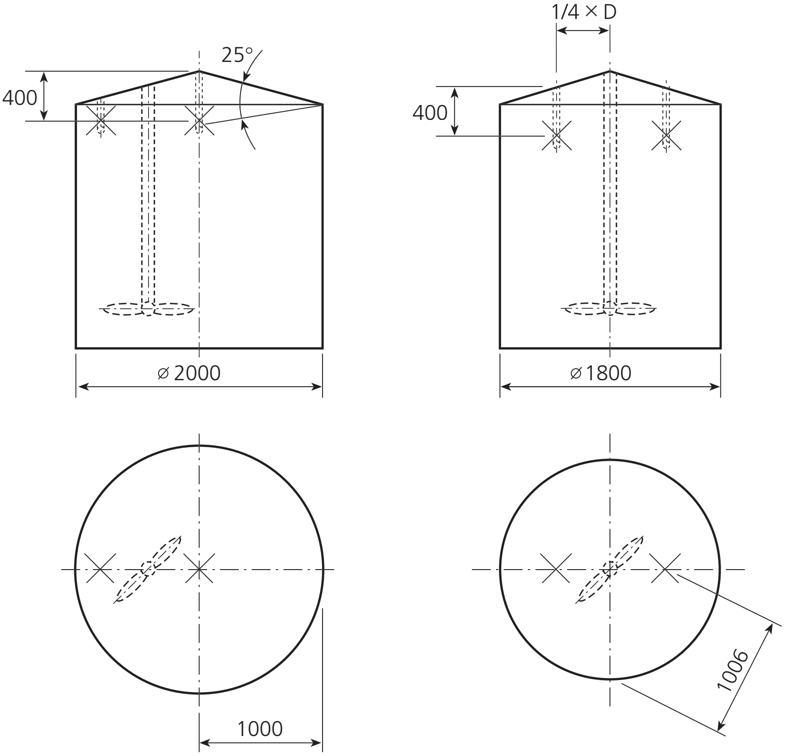


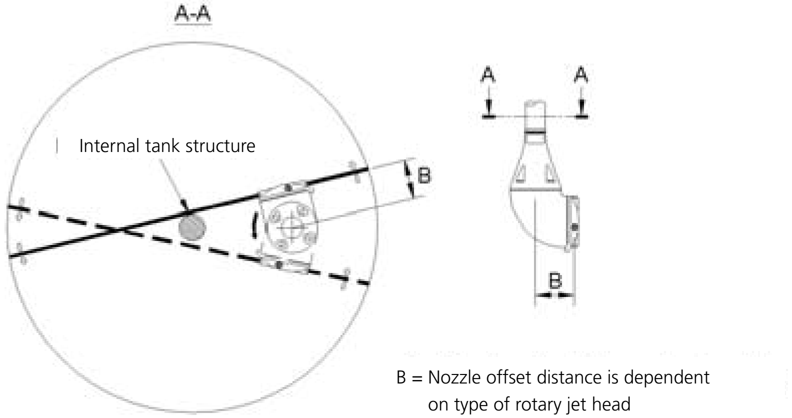
Fig. 6.14 The mounting position of the static spray ball. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

6.5.5 *Selection and sizing of tank-cleaning equipment*

Flowrate

The sizing and selection of a tank-cleaning unit is based primarily upon the flowrate required for the tank size and for the type of device selected. In addition, the flowrate required for a specific tank size is also governed by the shape, i.e. vertical or horizontal (see Figure 6.16). The flowrate requirement also takes into account whether there are product residues and detergent flocculates that have to be moved out of the tank via the liquid flow.

Static spray balls require larger flowrates than either rotary jet heads or rotary spray heads, and the flowrate differences are shown in Figures 6.17, 6.18 and 6.19. This difference in flowrate requirement is due to the difference in the impact that the device transfers to the wall of the tank.



B = Nozzle offset distance is dependent
on type of rotary jet head

Fig. 6.15 Illustration of the distance between the rotary jet head centreline and the nozzle location. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

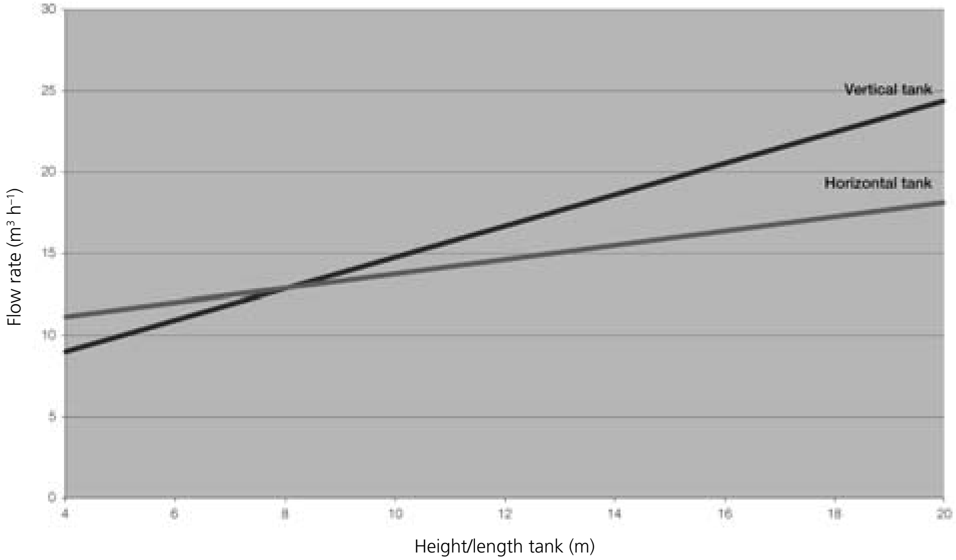


Fig. 6.16 Flow requirements for cleaning horizontal tanks versus vertical tanks. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

Effective cleaning distance

In addition to the sizing, the effective throw length of liquid is based upon the cleaning device. This is also known as the *effective cleaning radius* of the cleaning device. Effective throw length is the distance the jets or fans of cleaning liquid will travel and yet still hit the tank wall with sufficient force to ensure a cleaning effect on the tank wall. This is also termed the *impact effect* or *scrubbing effect*.

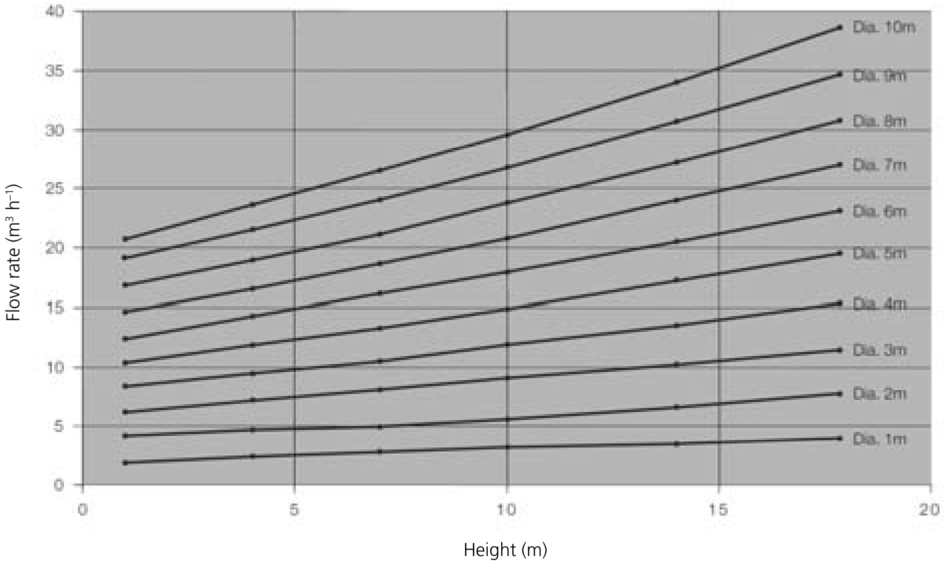


Fig. 6.17 Minimum flowrates required in vertical tanks using a static spray ball. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

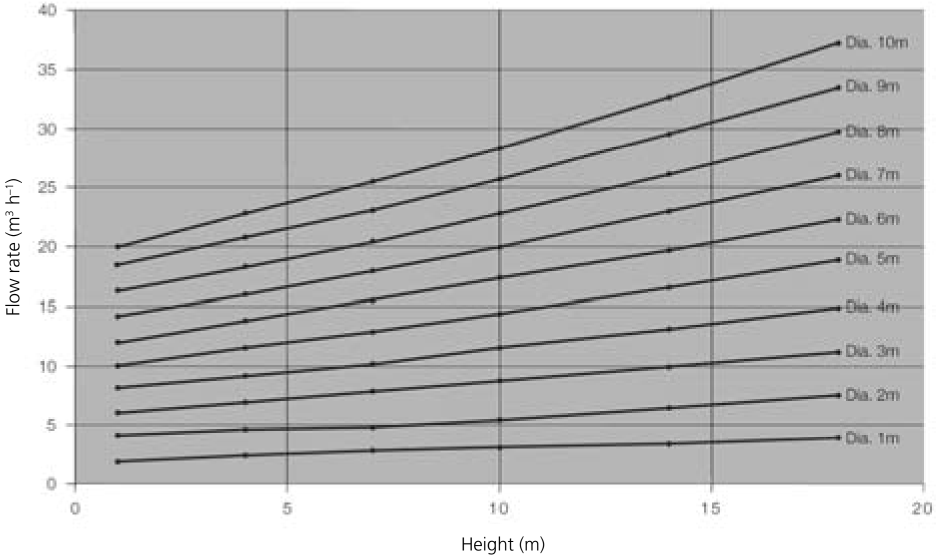


Fig. 6.18 Minimum flowrates required in vertical tanks using a rotary spray head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

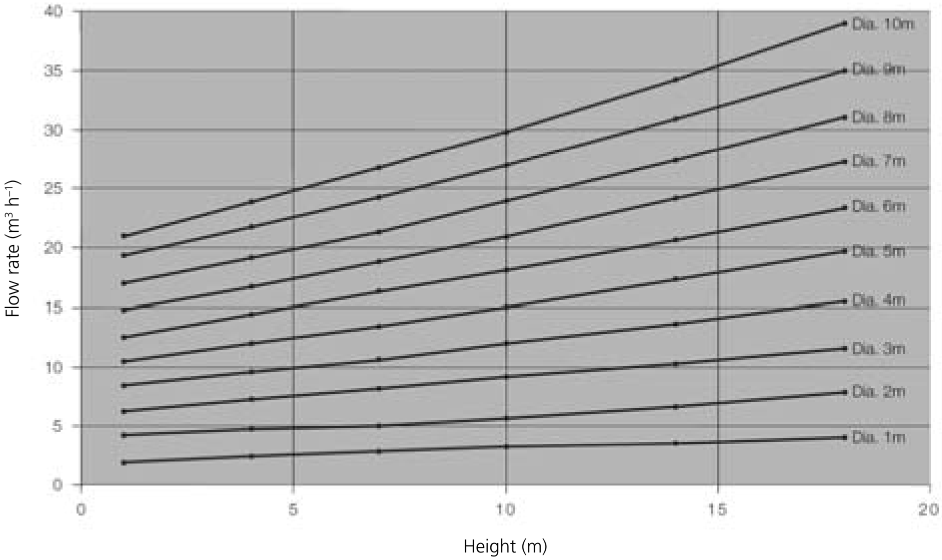


Fig. 6.19 Minimum flowrates required in vertical tanks using a rotary jet head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

The sizing of a tank-cleaning device should not be based upon the wetting distance of the jets or the fans. Wetting distance is the maximum distance the jets or fans will travel, but at the wetting distance there will be only very limited impact effect or scrubbing effect (Figure 6.20).

The effective cleaning radius is ideally related to the physical removal of a residue product given the specific process parameters, including flow, pressure, medium and medium temperature. This is not a definition of the complete cleaning process, but it can be related to

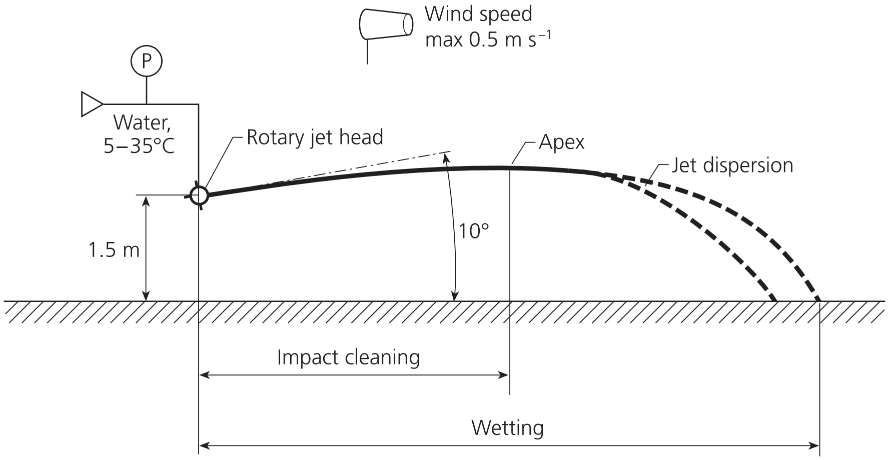


Fig. 6.20 Illustration of a rotary jet head impact cleaning distance. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

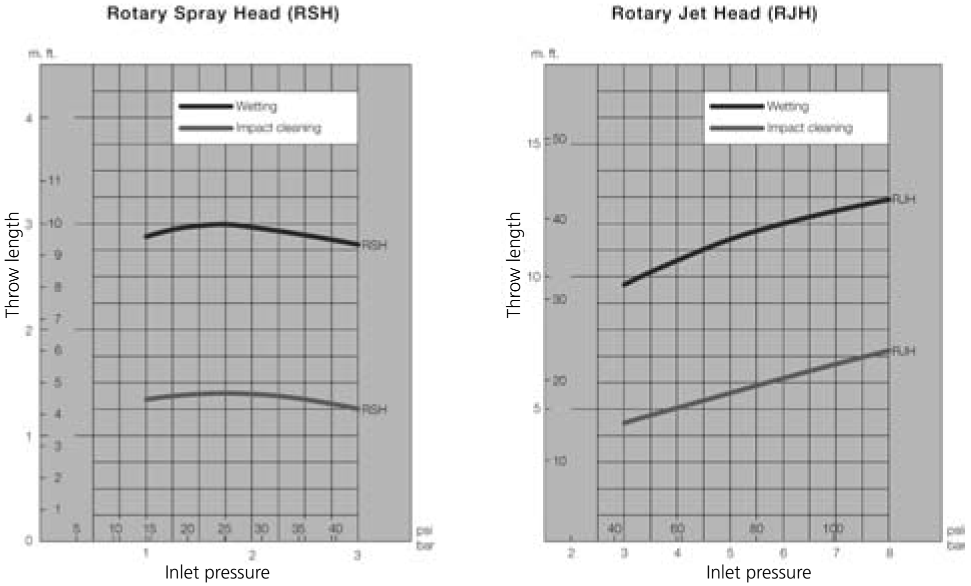
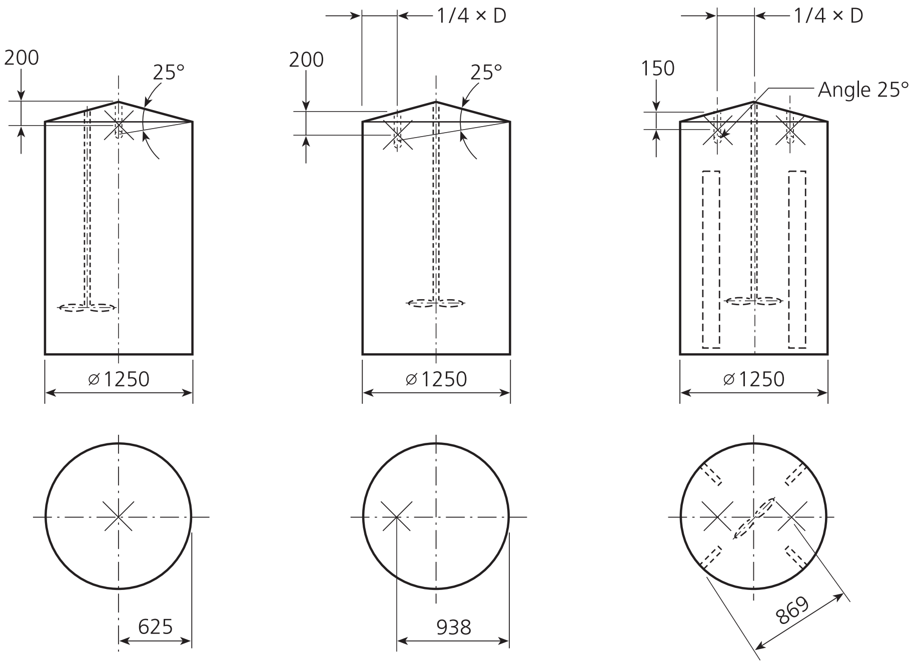


Fig. 6.21 Effective cleaning radiuses of a rotary spray head and a rotary jet head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.



Off centre agitator

Centre mounted agitator

Centre mounted agitator
with baffles

Fig. 6.22 Mounting position and cleaning radii of a cleaning device. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

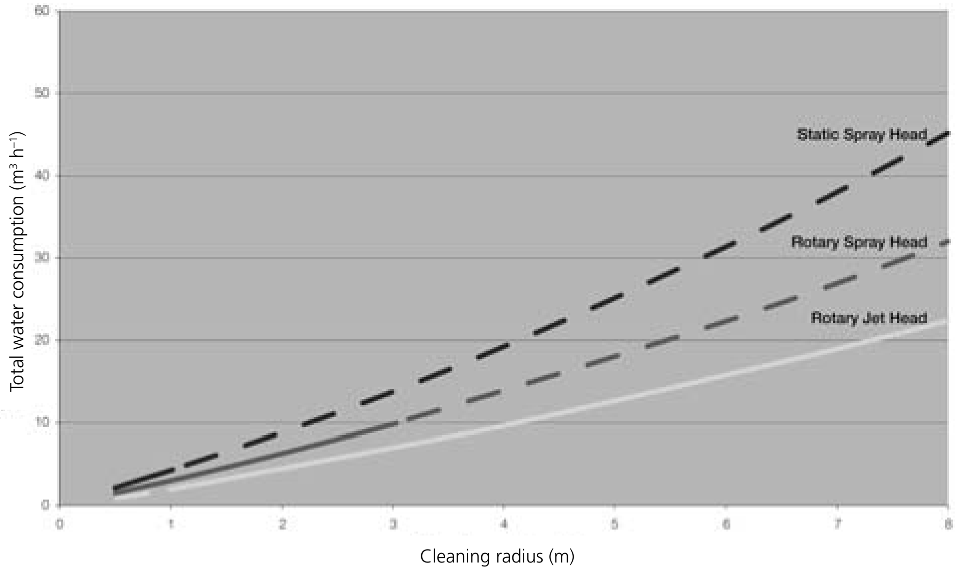


Fig. 6.23 Total water consumption and cleaning radii of a cleaning device. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

an effective pre-rinse or pre-cleaning step of a CIP programme, and these definitions should be provided by the manufacturer of the cleaning device (see Figures 6.21, 6.22 and 6.23).

The *traditional approach* to determining the specifications for the cleaning equipment required for any particular tank in a dairy processing facility normally involves the following aspects:

- considering the cleaning equipment once the size and shape of the tank have been decided, with the decision influenced by a range of external parameters that have no obvious links with hygiene considerations;
- a tendency to oversize the cleaning equipment installation ‘just in case’, and to build in a certain amount of flexibility in CIP set-ups in order to cope with future requirements;
- a certain amount of trial and error, because decision-making is often rooted in mechanical and engineering considerations and based on repeating existing set-ups that have a proven track record.

The *modern approach* to the selection of sizing CIP tank-cleaning equipment is based upon performance data, flowrate requirements and pressure and cleaning radius, as provided by the manufacturer of the cleaning device. The cleaning parameters for a given application – such as the effective cleaning radius, flow, pressure requirement and cycle times – should be the basis for the performance data provided by the manufacturer. The selection of a tank-cleaning machine should therefore always be based upon the cleaning duty in question, with tank design as a supplementary consideration.

Simulation software

The modern way to determine the specifications for CIP tank-cleaning systems is to use simulation software that avoids the limitations of the traditional trial-and-error approach. Such software makes it possible to ensure both correct installation and optimised cleaning operations right from the outset, based on each customer's specific process parameters. This is done by calculating the best possible positioning of the CIP cleaning device, accompanied by details of the location, piping and pressure/flow requirements, based on each customer's specific tank dimensions and equipment configurations.

The selection of tank-cleaning equipment is based upon an evaluation of the specifics of a particular cleaning assignment. The simulation takes this into consideration when evaluating the effect of the flow and volume as well as the shadow areas (see Plates 4 and 5).

Advanced software of this kind also provides an analysis of the cleaning shadows that can arise on account of internal structures and fittings, so that configuration of the cleaning device can take these into account. The advantages of simulation software include:

- verification of the tank-cleaning process parameters for system designs;
- documented operating parameters, including impact coverage;
- verification that the required cleaning performance can be achieved;
- avoiding investing in oversized installations;
- savings on installation costs;
- standardised tools for selecting tank-cleaning equipment;
- reduction of overall risk, better follow-through and easier allocation of responsibility, along with a greater degree of executive responsibility;
- reduction in the variability of CIP operating parameters;
- paving the way to a knowledge platform for CIP optimisation programmes.

6.5.6 *Upgrading tank-cleaning systems: total cost of ownership (TCO) and risk assessment approach*

Decisions about whether or not to upgrade existing tank-cleaning equipment have, traditionally, been required to consider the following aspects:

- mechanical performance of existing tank-cleaning equipment
- documented wear and tear that has already occurred
- expected service life
- up-front purchase costs of new equipment
- ease of maintenance
- cleaning performance
- cleaning cost

However, the most effective approaches to upgrade and retrofit decisions focus on a range of other analytical tools that factor in all the costs involved – both directly and indirectly. This wider conceptual focus no longer considers tank cleaning in isolation from issues that include plant-wide operating costs, the achievability of levels of hygiene whose consistency is documented, product quality, and product reputation.

Typical criteria that should form a viable basis for informed decisions about whether or not to upgrade existing equipment or to retrofit new equipment within an existing installation include:

- total cost of ownership (TCO) and financial payback;
- life cycle costs rather than initial purchase cost;
- energy profile, including carbon footprint;
- environmental impact, including waste generation and water consumption;
- risk assessment related to product quality and safety.

When CIP procedures become a management issue, it is important to have a series of appropriate tools available for assessing the criteria on the basis of which decisions have to be taken. Familiarity with *variation theory* thus becomes an essential element in the management of production processes.

Variation theory requires an in-depth understanding of a manufacturing process or system – i.e. *ceasing dependence on inspection*. Depending on inspection is like treating a symptom while the disease is killing you. The need for inspection results from excessive variability in the process. The disease is variability. Ceasing dependence on inspection means that the processes are understood so well that the quality can be predicted in terms of their outputs from upstream activities and measurements. To accomplish this objective, management must have a thorough understanding of the sources of variation in the processes, and then work towards reducing the variation (see Joiner & Gaudard, 1990).

These risks and variables in tank cleaning are addressed via application of the TACCT criteria in the cleaning method and the design of the tank.

6.5.7 *Risk assessment example*

The risk factors involved in such an assessment can normally be described as follows.

Residue type

The adherence of residue serves as a primary design criterion for chemistry or temperature considerations. The solubility of such residue is a primary design criterion for chemistry considerations. The location of residue affects accessibility as well as the effect generated by the energy applied by the cleaning device. Residue accumulates and continues to do so in discrete layers, bands, or areas related to tank operations. Residue includes adhered particulates. Residue in confined areas serves as a bacteriological growth site.

Tank design

Complex internal fittings interfere with fluid distribution. Increasing flowrates are required to address increased tank size or complexity. Tank hold-up volume dictates cleaning design flowrate.

Methodology

Materials plug orifices and affect fluid distribution. Plugging of orifices is not detected. Improper or inadequate pressure or flow affects distribution performance. Improper device position affects distribution. Control of liquid distribution is susceptible to supply system variations (other systems cutting in are energy losses between supply and distribution).

Monitorability

The directness of connection between process monitoring method, i.e. process analysis, to process performance and control. The responsiveness of the process to monitored parameters. Ease of data management for use in process innovation or improvement.

Repeatability

Sensitivity of tank-cleaning process to variations in production process. Number of variables involved in selected method. Detectability of out-of-specification (OOS) materials via process monitoring.

Cleaning materials

The number and type of cleaning chemicals creates its own set of process control requirements (complexity). The efficiency of cleaning chemistry is a corrective action process rather than innovative.

Table 6.1 Degree of variability when using different types of cleaning method.

	Manual (COP)		Automated (CIP)		
Function	Hand	Fill and boil	Static spray balls	Rotating spray balls	Rotating jet heads
Residues	High	Some	High	Some	Low
Tank internals	High	Some	High	Some	Low
Methodology	High	Some	High	Some	Some
Monitorability	High	Some	High	High	Low
Repeatability	High	Some	Some	Some	Low
Cleaning materials	High	Some	Some	Some	Low
Testing	High	Some	High	Some	Low

⟶

Decreasing order of ‘degree of variability’

Source: Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

Testing

Scope of process variables that must be addressed by testing method. Sensitivity of testing method. How representative the test results are as regards to process variables. It can be seen from Table 6.1 that the method of ranking with fewest sources of variability is the rotating jet head, whereas the method with the greatest number of variability sources is manual cleaning. The ‘boil and agitate’ method, although featuring fewer potential sources of variability, is also resource intensive and the least efficient of the methods available.

6.5.8 *Perspectives associated with CIP tank cleaning*

The efficiency of CIP tank-cleaning procedures is governed primarily by the design of the tank. In the dairy industry, the key to designing a tank for maximum cleaning effectiveness at lowest possible cost could be to turn the traditional design process on its head.

The best overall results in terms of hygiene standards, total cost of ownership (TCO), capital costs and operating costs can, in fact, be achieved by designing and configuring the tank installation around the cleaning requirements just as much as around the production requirements.

Every element of the fixtures and fittings inside the tank must be designed from the ground up with hygiene in mind. Even the most minute ‘weak link’ in the overall hygiene set-up can have catastrophic consequences for production. This is because the levels of hygiene available in the tank before production even begins can have a profound knock-on effect on both production costs and product quality.

Once the design of the CIP set-up for a tank has been focused on hygiene from the outset, the following can be considered the key design criteria.

- All tank design work must be carried out with hygiene requirements and cleaning procedures in mind.
- Each CIP tank-cleaning device must be selected for the particular tank and in the light of the specific production processes used.
- The CIP system as a whole must be designed to meet the particular process requirements associated with the CIP equipment selected for cleaning the tank, rather than merely considering basic size/volume/flow specifications.
- It must be possible to optimise all aspects of the infrastructure and support procedures in order to ensure that the CIP process can be carried out with maximum efficiency and minimum cost, while consistently maintaining documented hygiene standards. It must be possible to monitor the CIP system in order to have documented confirmation that the design requirements have been complied with.
- It must be possible to determine and verify the standard operating procedures for the specific cleaning device in the specific tank and within the specific operating context, as well as to lay down clear criteria for registering and evaluating any variance from these procedures and standards.
- It must be possible to determine the appropriate training requirements for both operation and maintenance of the equipment.

6.5.9 *Tank-cleaning devices*

The five main areas of focus when assessing whether or not tank-cleaning devices make a positive or negative contribution to overall hygiene standards are:

- surface finish – both internal and external;
- design, assembly and construction that take hygiene into consideration (for example, threads must be sealed off with O-rings);
- use of lubricants (these must be eliminated completely);
- self-draining capabilities;
- the ability to monitor and document cleaning performance.

The hygienic design of food production equipment has been an issue for several years. The European Hygienic Engineering and Design Group (EHEDG), in particular, is very active in establishing design guidelines as well as equipment approval via third-party certification, in order to assist both equipment suppliers and food manufacturers. EHEDG certification is based on an accredited third-party engineering review and testing programme.

Documented hygiene standards can be ensured by selecting process components and equipment that are certified as complying with relevant EHEDG criteria (see Figures 6.24 and 6.25).

Fig. 6.24 Toftejorg SaniJet 25 Rotary Jet Head. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

Fig. 6.25 Certification logo of the European Hygienic Engineering and Design Group. Reproduced with permission from the EHEDG (www.ehedg.org).

There are many aspects involved in considering effective cleaning of the tanks and vessels used in the dairy industry. The focus here is on the management and engineering aspects of making decisions on which solution and/or equipment is most suitable for any given context.

In the mindset required for modern dairy hygiene management, cleaning is an upfront, pre-emptive process with a high priority, rather than a utility function that is essentially just a practical afterthought to the design and engineering aspects of tank design. This new perspective includes a focus on where responsibility for decision-making about such equipment, i.e. its operational parameters, should lie.

This, in turn, raises the issue of which kinds of data and criteria should be made available to support such decision-making. A wide range of new measuring tools, technologies and analytical approaches are now available, which together move any discussion about the most suitable tank-cleaning device a long way from the realm of mere engineering and hands-on problem-solving.

There are significant tangible benefits to be obtained from using the most effective CIP procedures and cleaning devices. For example, it has been shown that the best rotary spray heads and rotary jet heads are capable of:

- reducing cleaning liquid flows by up to 50%;
- reducing water consumption by up to 75%;
- reducing cleaning time by up to 50%;
- reducing consumption of detergents and chemicals by up to 75%;
- making more time available for revenue-generating production.

These kinds of savings make CIP procedures and tank-cleaning devices a ‘mission-critical’ part of an overall process flow.

There are also wider management perspectives involved. Cost-effective CIP tank-cleaning procedures can provide management with:

- a more effective framework for dealing with overall operating costs;
- better supervision and control relating to tank-cleaning processes;
- greater influence on product quality;
- greater control in relation to environmental issues;
- an effective basis for managing quality assurance programmes;
- a way to minimise elements of risk in relation to the entire production set-up;
- a way to ensure that good results can be consistently reproduced;
- a way to provide full, accurate documentation as well as an audit trail that makes it possible to assign responsibility.

However, such benefits can be obtained only by fielding a comprehensive set of appropriate analytical tools, including a comprehensive, structured risk assessment programme. These are crucial as a basis for effective, well-informed management decisions that aim at tackling the root causes of any problems, and assigning clearly documented responsibility as a platform for remedial action. This is the only way in which tank-cleaning procedures can have a pre-emptive effect, as opposed to the traditional approach of merely dealing with symptoms once a problem already exists.

When CIP procedures become a management issue, it is important to have a series of appropriate tools for evaluating the criteria on which decisions have to be taken. Such tools can include assessments of:

- overall business risk
- product quality
- environmental impact
- traceability and documentation
- total cost of ownership
- water usage and effluent disposal
- total time spent on CIP procedures
- service management

6.5.10 *Cleaning validation*

An important new development in dairy industry tank-cleaning procedures lies outside the tanks themselves. In order to be able to maintain a seamless and wholly reliable quality assurance regime, there is now considerable focus on having full documentation – often called *validation* – that a tank has been cleaned according to specifications, and on a ‘right first time’ basis.

This means it is becoming increasingly important – often a *sine qua non* – for companies that process dairy products to provide completely reliable, end-to-end documentation of the cleaning processes and their consistent effectiveness over time. Such documentation is required for the extremely comprehensive quality assurance and hygiene management programmes now required in the food industry, and as part of an overall focus on traceability. As a result, sensors, software and IT systems that link into the company's overall control and monitoring systems are now often an important part of CIP systems used in the dairy industry.

Within a modern perspective on CIP procedures, the service and maintenance of all items of equipment is just as important as both the production processes and the cleaning procedures. It cannot be considered an 'afterthought' in the traditional mould. The structured management of service and maintenance keeps operating costs down as well as preventing expensive downtime. Effective training of the operators of CIP equipment is a key part in this. Even though rotary spray devices and rotary jet devices are normally highly automated, this greater degree of automation brings even greater importance to the role played by each of the relatively few operating and maintenance staff.

6.5.11 *Case study*

This real-world example illustrates how a strategic decision to save on water consumption on a new site under construction as well as at an existing site was implemented – with close cooperation between company management, the operating and engineering staff, and the manufacturer of the tank-cleaning equipment. The result was not only savings on water and effluent, but also reductions in CIP times, and consistent improvements in cleaning performance.

Before installation of any tank-cleaning devices at the new dairy under construction, extensive trials were carried out at an existing site. The appropriate rotary spray heads were sized and selected, and Toftejorg SaniMega rotary spray heads were tested to determine whether the flowrate could in fact be reduced from 40 $m^3 h^{-1}$ to 25 $m^3 h^{-1}$, as recommended by the manufacturer, Alfa Laval.

The objective of this test was to achieve good, reliable cleaning performance using the lower flowrate. However, it was also very important to make sure that the water savings would not result in any prolonging of the CIP tank cleaning cycle due to extended heat-up times.

The outcome of these trials proved that the silo and the outlet pipework were cleaned extremely well, using 40% less water. Heat-up times were unaffected, and were even reduced by 10% on some occasions. This reduction, even with a reduced flow of water, was attributed to the effect of improved coverage and the impact of the hot water across the internal surfaces of the tank. Following these trials, SaniMega rotating spray heads were installed in all the raw and finished milk silos involved in the project (see Figure 6.26).

Rotary jet heads were also tested, and later installed in the hot cream and cream-ageing tanks. The benefit of using rotary jet head technology in these particular cleaning applications was that it was necessary to use only one tank-cleaning device per tank – compared with two being required if static spray balls were to be used.

At the same time, the flowrate was reduced by 40%, which assisted product recovery. When running a short rinse just to recover the product left behind in the tank, less entrained water in the cream was achieved, and more products could be recovered. Another important,

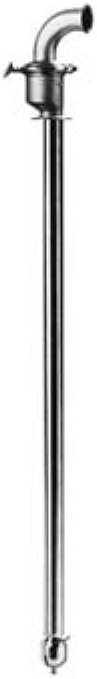


Fig. 6.26 SaniMega – rotary spray head silo-tank-cleaning device. Reproduced by permission of Alfa Laval Tank Equipment A/S, Ishoej, Denmark.

consistent result was that there were no problems related to tank hygiene, tank CIP issues or the reliability of the tank-cleaning devices.

The trials were conducted over a two-year period, and showed that the silo fitted with the SaniMega rotary jet head device had been cleaned much more effectively and economically than the other silos in the milk reception area.

In the dairy industry, there is a huge focus on minimising both utilities consumption and production downtime. The technologies behind rotary spray heads and rotary jet head technology have been proven to provide the solution.

6.6 Conclusions

Tank-cleaning procedures are traditionally part of a mindset that considers them primarily a question of just cleaning up after the previous production batch, ready for the next. The equipment used to implement these key procedures usually receives correspondingly limited attention, with the focus normally on individual items of equipment, without consideration of the wider overall context within which these operate.

The modern remit for CIP, however, involves moving away from this traditional engineering mindset. Seeing CIP procedures as the first step in any new production batch, rather than as the last step in the previous one, is part of a process of reassessing and rethinking the CIP procedures for the tanks and vessels used in the dairy industry.

This opens up important new perspectives and opportunities. It also involves executive-level responsibility for establishing and maintaining an overall mindset that considers decisions about CIP installation and their use as central to a set-up in which quality is substantially improved, costs are substantially reduced, and the element of risk is substantially curtailed.

Better management, monitoring and control of the overall production and cleaning set-ups, thereby maintaining high levels of control, traceability and executive accountability, provide a solid basis for successful tenders for new, high-quality/high-value production assignments. Ultimately, effective CIP design and management also make it feasible to enter into new strategic alliances and constellations with partners that demand exceptional standards of accountability and control.

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7 Design and Control of CIP Systems

D. Lloyd

7.1 Introduction

The design and control of cleaning-in-place (CIP) systems is – or should be – based upon a logical design process. Usually, in fact, it is based upon what funds are left. It is almost always the poor relation to the shiny filling machines and other expensive equipment. It is possible to suggest that the following principles should be applied, and these are further amplified in subsequent sections.

7.1.1 *Full recovery system: three tanks CIP*

This unit has hot and cold detergent tanks, and cleans a variety of tankers including milk, cream, beer, cider and general foods. A general illustration is shown in Figure 7.1.

Fig. 7.1 A general view of the CIP cleaning system.

Cleaning-in-Place: Dairy, Food and Beverage Operations Third Edition. Edited by Adnan Tamime

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7.1.2 *How much CIP?*

This involves establishing how many pieces of equipment are to be cleaned, and the time available. For example, if there are 10 h available to carry out CIP, there are 10 items to be cleaned, and each cleaning sequence will take 30 min, then cleaning time is 5 h plus a 20% contingency time, i.e. 1 h, and the overall cleaning time is 6 h. Therefore the available time of 10 h is adequate for one CIP system. If, for the same example, the cleaning time is 1.5 h, with the same contingency time, the total cleaning time is 18 h. But the time available is only 10 h, and therefore two CIP systems or channels are needed for cleaning the equipment.

In addition, it is also necessary to factor in future expansion, and to make arrangements in the design of the CIP system to cope with the cleaning cycles.

7.1.3 *Size of the CIP equipment*

The CIP detergent tanks should hold (i.e. the working volume) sufficient capacity for the largest volume in circuit plus 20%, plus any allowance for known future requirements (see Table 7.1).

The pre-rinse tanks, when fitted, should be large enough to hold the total volume of water required, and for the circuit with the largest and longest flow (see also Table 7.1). For example, in a CIP system required to clean large vessels and 7.65 cm diameter pipelines, the flowrate through a low-pressure spray ball may be 200 L min^{-1} with a total rinsing time of 3 min; the volume of water required is therefore 600 L. However, the pipeline may also be rinsed for 3 min, but at a flowrate of 550 L min^{-1} to give the correct velocity, and the total flow of water is 1650 L. Therefore the pre-rinse tank should be sized at 2000 L for the worst case plus some spare capacity.

7.1.4 *Hot or cold pre-rinse?*

If the product is light and free flowing, e.g. rinsing milk, then cold pre-rinse is adequate; whereas, if the product is thick and fatty, such as cream, yoghurt or other dairy products, a warm pre-rinse at 40°C will be sufficient to ‘melt’ the fat and deliver a much more effective pre-rinse, which will reduce the detergent usage and CIP time.

In a heated CIP system (which covers virtually all dairy applications), there is no harm in using warm pre-rinse on milk. Again, it will speed up the sequence, and reduce the detergent consumption.

7.1.5 *Choice of scavenge pump*

If the scavenge or return pump is ineffective, the CIP sequence will be poor, costly, and will not clean the vessel effectively. This pump must be capable of self-priming, and pumping through air. A typical and most effective machine is the liquid ring pump (see Figure 7.2); although expensive, it works perfectly.

Table 7.1 Advantages and disadvantages of type of pressure spray head for the efficiency of CIP cleaning.

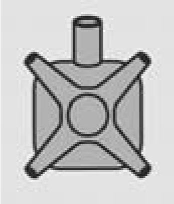
Type	Advantages	Disadvantages	Illustration
Low pressure	Low cost	High flowrate	
	Wide range available	High effluent and water load	
	Rapid heat input	No mechanical effect	
	No moving parts	Relies entirely on chemical cleaning	
	Low maintenance	Limited spray diameter	
	Copes well with internal obstructions	High pressures vaporise sprays	
	Flowrate integrates well with line cleaning.	Need multiple spray heads for larger vessels	
	Easy to confirm correct operation automatically	Need larger CIP pipework and scavenge pumps	
Medium pressure	Medium cost	Need multiple spray heads for larger vessels	
	Wide range available	Moving parts	
	Moderate heat input	Higher maintenance, and can jam	
	Medium flowrate	Need flow and motion instrumentation to automatically confirm operation	
	Lower effluent load	Internal obstructions break up jets	
	Lower CIP pipework diameters		
	Smaller scavenge pumps		
	Good spray diameter range		
	Some mechanical cleaning effect		
High pressure	Low flowrate	High cost	
	Lowest effluent load	Lower heat input rate	
	Removes heavy soil by high-pressure jets	Moving parts	
	Lowest CIP pipework diameters	Higher maintenance	
	Smaller scavenge pumps	Can jam	
	Excellent spray diameter range	Needs flow and motion instrumentation to automatically confirm operation	
	High-pressure mechanical cleaning effect	Internal obstructions break up jets	
	Single head can clean very large vessel	Longer cleaning sequence	

Fig. 7.2 An Alfa Laval MR200 liquid ring pump on a trolley.

7.1.6 *Choice of spray head*

The various choices of spray head are discussed in Section 7.4. In a brand new installation the use of high-pressure heads can reduce pipeline sizes and scavenge pump capacity. However, it is difficult or impossible to match the CIP pump for the spray head with that required for the pipe lines. So it is often the case that a high-power, low-flow pump is used for the spray heads, with a high-flow, low-pressure pump for the lines. It is unlikely that a pump can be found to cover both flow duties via a variable-speed drive.

On existing systems, where the outlet pipework is often cleaned with the tanks, to get the correct flow in the pipework (which may be up to 900 L min^{-1} for 10.16 cm diameter pipelines) the high-flow/low-pressure spray head is usually the best choice to match both duties.

Other considerations, such as water and effluent costs, are very important and favour the use of the higher-pressure spray heads. But the most important features of the cleaning system are that it works effectively and produces consistently clean plant as quickly as possible. All other considerations should be of secondary importance.

7.1.7 *Control units*

There is a huge range of control options on the market: from simple logic controllers for £100 or so to large PLCs with graphics and supervisory, control and acquisition of data

(SCADA) systems. The choice will depend on budget, plant standards, the need to link with other equipment, and the level of operator skills. Most dairy processors in the UK are still specifying basic simple systems as well as the complex versions. In fact, the majority of new CIP systems are basic in concept.

7.1.8 *Supermarkets*

Several of the large supermarkets impose strict CIP conditions on dairy producers. Some of the requirements are documented, and some emerge following visits from the various auditors. If a producer is required to adhere to the specific requirements of any customer, it is important to obtain and adhere to the standards of that particular customer.

7.2 Principles of chemical cleaning

This section is not intended as a scientific text, but as a general guide; for more detailed information refer to Chapter 4.

7.2.1 *Soil removal*

Soil is anything that should not be present on the surfaces of the processing equipment. However, visible soil, such as product residues – particularly in fermentation tanks/fermenters – hard water scale or foreign bodies can, as suggested by the name, be seen and often smelled. Non-visible soils include bacteria, spores and yeasts. Tainting can also be a problem. Sometimes non-visible soiling can be smelled and sometimes not.

7.2.2 *Soil removal parameters*

The main parameters of soil removal are time, temperature and concentration of detergent, along with the mechanical effect from the spray head, and these parameters can be varied pro rata (within limits) to change the cleaning regime. Some cleaning must be carried out above a certain temperature to facilitate removal of fatty products. If low-pressure spray heads are used, then there is no mechanical action.

It is relatively easy to vary time, temperature and concentration of the detergent, but varying mechanical action is not so straightforward. Cleaning efficiency does not improve above certain detergent concentration limits. There are settings for each cleaning application where time, temperature and concentration are optimised to give minimum cost and best cleaning efficiency. For most CIP users there is scope to make significant operating cost savings by carrying out an optimisation exercise.

7.2.3 *Chemical soil removal and disinfectants*

CIP detergents are used to dissolve the soil, and remove it from the equipment being cleaned. It is necessary that the soil removed be held in suspension and returned to the CIP system. Most detergents are more effective when used over 50°C, and with the correct choice of detergent and concentration there is very little improvement in cleaning performance over 70°C.

Caustic soda or *sodium hydroxide* is one of the best chemicals for soil removal. The alkali reacts with the fat in the soil, softens it, and removes it quickly. It is not good at removing staining or scaling. Caustic soda is typically used at 0.5–2 g 100 mL^{-1} for most applications, and at up to 4 g 100 mL^{-1} for very heavy soiling. Although caustic soda and other detergents are good at removing soil, they are not always good at holding it in suspension. To improve this, other chemicals and sequestrants are added to get over the shortcoming, and a typical illustration is shown in Figure 7.3.

Acid detergents, such as phosphoric and nitric acids, are used in detergent formulations. They can remove light soiling, and they are commonly used on raw milk applications. Acids are good at removing staining and scaling, and leave the plant bright and shiny after CIP. Equipment that is generally cleaned with caustic detergents is cleaned occasionally with acid to remove stains and brighten up the surfaces. The use of detergents with high concentrations of nitric acid must be considered with care, as it will often attack seals in pumps and valves if they are not made of a suitably resistant material. Nitric acid products are used on difficult stains, and sometimes when degreasing, brightening and preparing new installations.

Disinfectants, *sanitisers* or *chemical sterilants* are all terms describing chemicals used to ensure the quality of rinse water, and to kill any remaining microbial contamination prior to using the equipment. There are many different products and chemicals, but only two compounds are popularly used in CIP: these are sodium hypochlorite and peracetic acid (PAA). All disinfectants are very poor at soil removal, and small residues of soil will quickly neutralise their active ingredients and render them useless. The plant must therefore be clean to a high standard before they are applied.

Sodium hypochlorite is a very popular chemical because it is cheap and extremely effective. The active ingredient is chlorine, which is very good at killing micro-organisms. This is a bleach, and has a characteristic chlorine odour. It has shortcomings: it can be very corrosive on all steel surfaces at high concentrations, and it can release chlorine, which combines with moisture in the air to form hypochlorous acid. This acid attacks and pits all grades of stainless steel – and people! If acid is added to hypochlorite, chlorine gas is released, and it

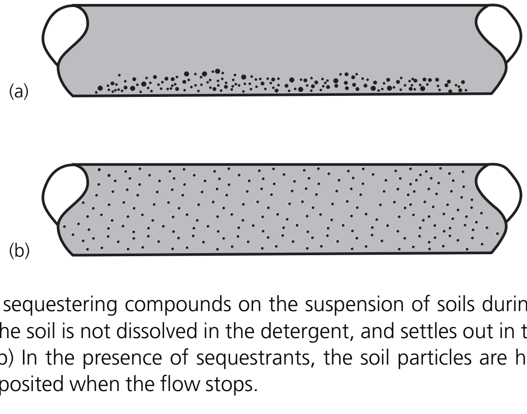


Fig. 7.3 The effect of sequestering compounds on the suspension of soils during the cleaning cycle. (a) Without sequestrants, the soil is not dissolved in the detergent, and settles out in the equipment when the detergent flow stops. (b) In the presence of sequestrants, the soil particles are held in suspension in the detergent, and are redeposited when the flow stops.

Fig. 7.4 The safety aspects of detergents in the CIP room.

can taint products even at very small concentrations. This means that, when hypochlorite is used as a disinfectant, it must be rinsed out afterwards to avoid these problems.

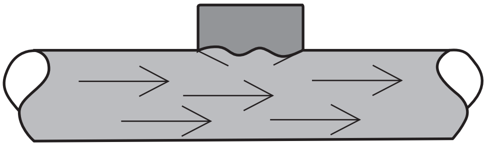
Peracetic acid has become popular over the last 15 years because, for most products, some sites are known to leave this product in the plant with no apparent adverse effects, and it does not have to be rinsed out afterwards. The PAA breaks down to harmless components. It is almost as good as hypochlorite for most microbial duties, and better on yeasts and spores. It is considerably more expensive, and has a very sharp, vinegary odour, which is extremely unpleasant when concentrated. PAA is unstable, and if kept for a long time in concentrated form breaks down and evolves oxygen. In a confined space it can create an oxygen-rich atmosphere, which is a fire risk – especially if welding is taking place nearby.

All of the chemicals are potentially hazardous to people, product(s) and the general working environment. It is vital that full product datasheets with all necessary COSHH and safety data are to hand, understood, and displayed near the storage and/or CIP facilities (see Figure 7.4). When handling concentrated product, appropriate safety equipment should be worn – especially chemical goggles and gloves – because these chemicals are very unforgiving on eyes. In addition, when used in CIP systems, the chemicals are often very hot, which adds another risk for the unwary. It is best to be aware of hose and safety shower locations before beginning any work on CIP and chemical facilities, even if you are just observing or measuring up.

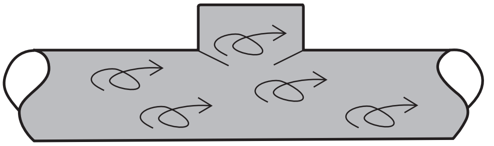
7.3 Application of CIP

7.3.1 *Pipeline cleaning*

The efficiency of cleaning of pipelines is influenced by the velocity of the chemical solution, which can affect its flow behaviour (i.e. laminar or turbulent; see also Figure 7.5). The



Laminar flow below 1.4 m s^{-1}



Turbulent flow above 1.5 m s^{-1}

Fig. 7.5 The flow behaviour of the cleaning solution, as influenced by velocity.

laminar flow does not give an effective cleaning (flowrate < 1.4 m s^{-1}), whereas the turbulent flowrate is between 1.5 and 2.1 m s^{-1}, and gives an effective cleaning. There is no gain in the efficiency of cleaning if the velocity exceeds 2.1 m s^{-1}.

7.3.2 *Vessel cleaning*

CIP application

CIP used to clean a vessel may be applied as high or low pressure (see Plate 6). In the former approach, the cleaning heads remove the soil by mechanical action (i.e. the force of the jet), and the surface of the vessel is contacted in a series of 'sweeps' as the jets rotates and spins. If a low-pressure cleaning head is used to clean a vessel, the efficiency of the cleaning system relies entirely on the chemical action to remove the soil, and there is little or no mechanical effect from the spray jets.

CIP return or scavenge

Poor scavenge is the cause of a large proportion of CIP problems. Effective vessel CIP demands that the cleaning solutions be quickly pumped away so that the surfaces of the vessel are presented to the CIP jets, and are not covered by a back-up of cleaning solutions (Figure 7.6). In addition, poor scavenge will cause excessive CIP times, excessive use of detergent, excessive effluent discharge, and excessive use of heating energy. The correct

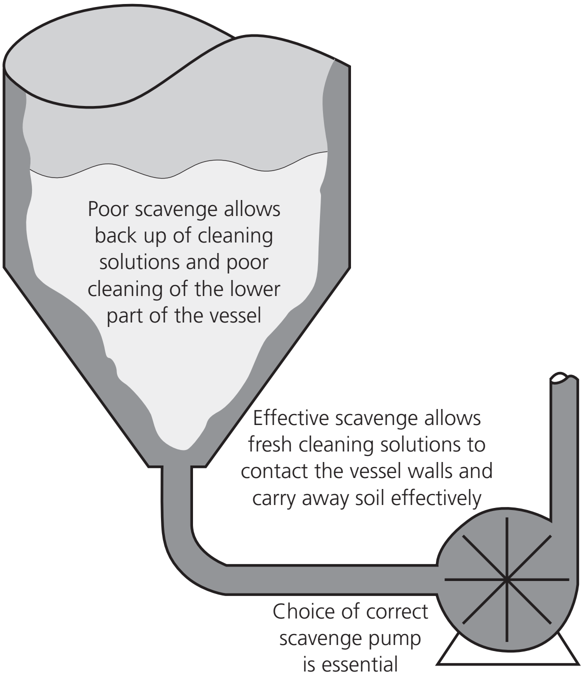


Fig. 7.6 The effect of scavenge in the CIP system on the efficiency of cleaning a vessel.

choice of a pump and correct design of CIP return pipelines are both vital to effective and efficient cleaning.

The correct choice of spray head, and balancing it with the flow requirements of downstream pipelines and equipment, is of great importance in correct CIP design. Table 7.1 summarises the main advantages and disadvantages of the effect of the type of pressure and spray heads used in a CIP system.

7.4 Types of CIP system

7.4.1 *Single-use system*

This type of system is also known as *single tank* or single use, and a water buffer tank is normally used. All stages of the cleaning process, which would normally include pre-rinse, detergent, final rinse and disinfecting rinse, are discharged to drain, i.e. after recirculation where appropriate (see Plate 7). Water make-up is controlled by level probes and automatic valves. Some recirculation of detergent solutions is achieved either by linking the return into the suction side of the CIP pump or by scavenge back to the buffer tank.

Heating of detergent solutions for hot CIP using total loss systems is usually by a plate or shell and tube heat exchanger. Some existing systems still use direct steam injection into the CIP flow, but this method is unlikely to be used on any new systems manufactured.

The addition of detergent or disinfectant chemicals to total loss CIP systems is from local containers or ring mains, pumped either into the suction side of the main CIP pump or into the buffer tank. Additions are normally made on a timed basis for disinfectants, whereas conductivity control is generally used to control detergent addition and strengths.

7.4.2 *Partial recovery system*

There are two configurations used for partial recovery CIP systems (Plate 8). There may or may not be a water buffer tank, and there is a recovery tank. For light soiling – which is the norm in dairy processing – the recovery tank holds and recovers the dilute detergent. In this application the system operates much as a full recovery system, but with the final rinses routed to drain, and the pre-rinses being fresh water. For very heavy soiling the detergent from the previous sequence is used as the pre-rinse for the next cleaning cycle. This application is not very common, but may be necessary in some dairy/food production applications. These systems have more in common with 'total loss' of water/cleaning solutions, except that there is some recovery of the detergent, which will reduce water and effluent costs. There are very few partial recovery CIP systems installed in the dairy/food industries.

7.4.3 *Full recovery system with heated rinse tank (optional)*

This type of CIP system is also known as *three-tank*, *reuse* or *full recovery* (see Plate 9). The three tanks are used as a water buffer tank, a detergent recovery tank, and a rinse recovery tank. At the start of a sequence, or by separate selection, the contents of the tank are recirculated through a 'make-up' loop circuit. Depending upon the design of the cleaning system, this may be a separate pumped loop or part of the main CIP supply pipework using the CIP pump. The loop contains conductivity and temperature probes, either with local controllers or with direct analogue outputs. When below a set point, the detergent is added and the heat is switched on, and at the required set point the full CIP programme proceeds. The conductivity and temperature probes must be in circuit, and the controller must be enabled throughout the detergent phase of the CIP cycle to ensure that the concentration of the detergent and temperature are maintained throughout. This system may lead to delays in CIP start-up, but does ensure that the detergent solutions are at the correct strength and temperature, and are available at the start of the detergent phase and throughout the cleaning cycle.

The water tank is used to establish a hydraulic loop before the pre-rinse cycle, possibly to purge the product and to supply final rinses, which may include disinfectant. The final rinse is not directed to drain after the cleaning cycle, but to the rinse recovery tank. This recovered rinse water is then used as a pre-rinse in the next CIP cycle, offering two advantages: (a) detergent residues in the water will lead to more effective pre-rinsing; and (b) water usage is minimised. The facility for automatic water addition should exist on all three tanks, by automatic valves and level probes.

A full recovery system can, in fact, operate with only two tanks in a similar way to that described above: the water is not present, and the fresh water supply is direct from the mains or from a centralised buffer tank into the CIP section. However, the provision of a water tank is strongly recommended, and it may not be possible for a direct connection to the mains supply. A typical full recovery sequence is shown in Table 7.2; it also includes an acid detergent stage, which is not usually selected for everyday cleaning operation(s).

7.5 Verification

CIP verification is a requirement of supermarket codes; in any case, it is good practice, and provides valuable information on the CIP performance as well as identifying any problems. Usually the return flowrate, temperature and detergent conductivity are monitored. Where possible, the CIP sequence selected is also automatically monitored, and an example of a Johnson Diversey 'Shurlogger' printout is shown in Figure 7.7.

7.6 Control systems

There is a huge choice of control systems available on the market; the features currently available and their application will be briefly reviewed.

7.6.1 *Schematic illustration*

A typical schematic illustration of control systems for CIP is shown in Plate 7, and the various features of the control system are summarised in Table 7.3.

Table 7.2 The sequence of operations of a full recovery CIP system.

Cycle	Description	Comments
1	Purge out product with air or water	Usually done only on cream or other higher-value products
2	Establish a return flow using cold water	Check with the return flow probe
3	Pre-rinse to drain	From pre-rinse tank; effective pre-rinse is the key to successful CIP systems
4	Purge out the pre-rinse water with dilute detergent	Measure the return detergent strength with the return conductivity probe
5	Return dilute detergent to tank and recirculate for a set time	At least 10 min at temperature and conductivity set points
6	Monitor temperature and conductivity of the detergent	Hold sequence on if they fall below set points
7	Recover the detergent	On time or to high level of tank
8	Intermediate rinse to recovery tank	
9	Inject acid	Control by conductivity probe
10	Circulate the acid solution	
11	Intermediate rinse to drain	
12	Inject disinfectant	Usually peracetic acid, which is controlled by a small flow meter or timed
13	Recirculate disinfectant	
14	Scavenge or drain down	If sodium hypochlorite is used as disinfectant, then it is necessary to carry out a final rinse to drain

7.6.2 *Instrumentation*

Detailed control will vary, depending on standards and/or budgets. Figure 7.8 shows conductivity and temperature probes, and a flow switch. All the instruments required to conform to the latest supermarket codes of practice, including their functions in a CIP system, are listed in Table 7.4, except for the valve feedback switches.

7.6.3 *Mechanical components*

Instruments for the full recovery heated detergent and pre-rinse system fitted with three tanks are listed in Table 7.5; however, the quantities will vary for the other models.

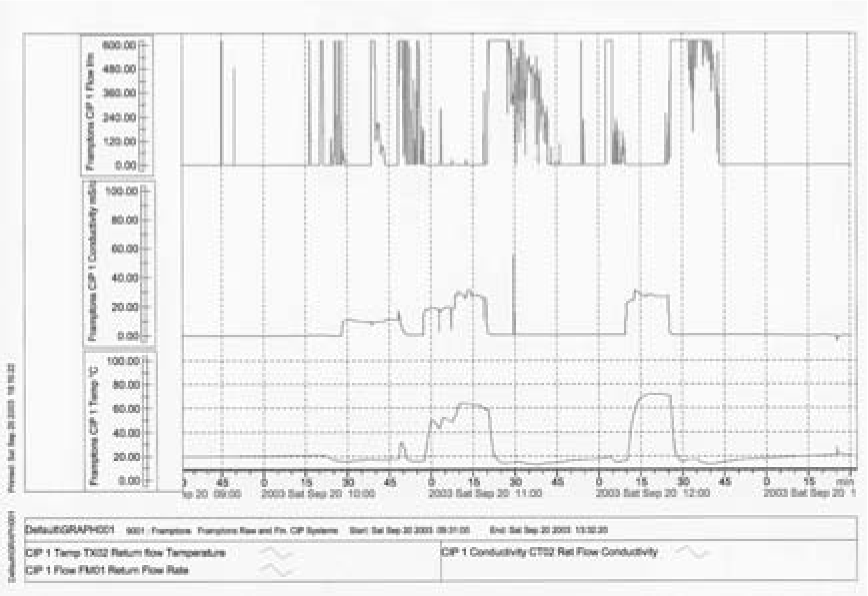


Fig. 7.7 Johnson Diversey 'Shurlogger' display of CIP.

7.7 Design information

All the data in this section are intended for quick outline reference, and for information only. However, it is recommended that detailed calculations be made for specific applications.

7.7.1 *Pipeline capacities*

Any CIP installation may have pipelines of different lengths and diameters; Table 7.6 lists approximate capacities.

7.7.2 *Detergent tank capacities*

Table 7.7 lists pipeline cleaning capacities for different detergent tank capacities.

When calculating the capacity of detergent tanks, we suggest that at least 25% spare capacity be included to allow for future development.

Table 7.3 A summary of the features of the control system for CIP.

Description	Standard
Case	Usually stainless steel to IP 55 as a minimum
Operator display	Push buttons and indicators or colour LCD display
	Touch-sensitive screens are now economically available
Communication	RS232 C, RS422, Profibus, Control Net, Device Net and other protocols
Computer link	Ethernet
Connectivity	Ethernet via computer
	Some systems can be programmed to send data automatically via e-mail. Connectivity via the Internet is possible with some systems
Direct dial-in	This achieved via special modems in the PLC racks
Storage	PLCs are not very good at storage
	This is best done by separate computer, or some storage can be in HMIs
Processor capacity	Not now a limitation on CIP design; even the smaller PLCs have plenty of memory capacity
Analogue inputs 4–20 mA	For temperature, conductivity and flowrates
Costs	Costs of smaller and medium-sized PLCs are now comparatively very low
	A small logic controller with 8 inputs and 6 outputs can be purchased for under £100

7.7.3 *Cleaning velocity*

See Table 7.8. For optimum cleaning of pipelines, a flow velocity between 1.5 and 2.1 m s^{-1} is recommended. It has been shown that for flows over 2.1 m s^{-1} there is no discernible increase in cleaning efficiency, and below 1.5 m s^{-1} flow becomes laminar, with poor soil removal. Lower flow velocities use less energy, and are used only for clean liquids, such as bright beer and bright cider. However, medium flow velocities are used for cleaning milk, beer and fruit juice processing plants; high flow velocities are used for cleaning cream, yoghurt, soups, pickle and other viscous foods installations.

Fig. 7.8 Typical conductivity/temperature probe and flow switch.

7.7.4 *Pressure drop*

Table 7.9 gives an overview of pressure drop in pipelines in MPa 100 m^{-1} of pipeline. To these figures should be added losses for fittings, bends, tees, spray heads, and head difference and so on. This is not generally an exact science

The approximate dimensions (in cm) of the CIP installation shown in Figure 7.9 are listed in Table 7.10.

Table 7.4 Instruments required in a CIP system, and their requirements to conform to supermarkets' code of practice.

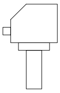
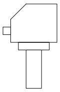
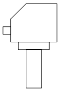
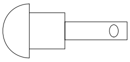
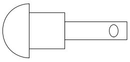
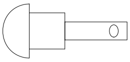
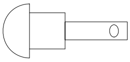
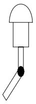
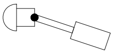
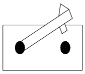
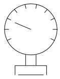
Description	Number required	Duty	Illustration
Supply flow transmitter	1	Monitors CIP supply flow for different routes Gives warning of blockages or open ends	
Return flow transmitter	1	Monitors CIP return flow for different routes Gives warning of low flow and poor scavenge	
Water supply flow meter	1	Measures the total water usage per cycle This is very important information when optimising performance and assessing the environmental impact of the system.	
Conductivity transmitter: detergent make-up	1	Controls the strength of the detergent in the tank	
Conductivity transmitter: return line	1	Used to return the detergent to the tanks. Stops the CIP timer if the conductivity drops Used to monitor acid and disinfectant injected (if applicable)	
Temperature transmitter: detergent make-up	1	Controls the temperature of the detergent in the tank	
Temperature transmitter: return line	1	Stops the CIP timer if the temperature drops	
Return flow switch	1	Used to confirm that there is a CIP circuit at the start of CIP sequences	
Level probes	6	Used to control the filling and emptying of the CIP tanks Flags a fatal alarm if the detergent tank empties during recirculation	
Valve feedback switches	28	Used to confirm the position of each valve, i.e. open or closed	
Pressure gauge	1	Confirms correct supply pressure, and indicates any filter blockages	

Table 7.5 Instruments required in a full recovery and heated detergent CIP system.

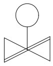
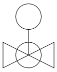
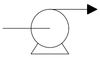
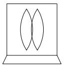
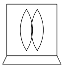
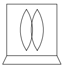
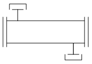
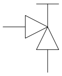
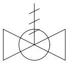
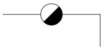
Description	Number required	Duty	Illustration
Butterfly valve	14	Routes CIP solutions	
Ball valve	1	Steam on/off valve	
Supply pump with stainless cowl	1	Pumps CIP solutions	
Detergent dosing pump, e.g. pneumatic diaphragm	1	Doses detergent into the tank On some systems, this is a valve	
Disinfectant dosing pump, e.g. pneumatic diaphragm	1	Doses disinfectant into the supply line	
Acid dosing pump, e.g. pneumatic diaphragm (optional)	1	Doses acid into the supply line	
Heat exchanger: shell and tube design	1	Heats up detergent and pre-rinse solutions	
Sample valves	4	Samples tank solutions and supply line solution	
Antisyphon valves (one standard, one optional)	2	Prevents contents of acid or disinfectant containers from being drawn into the suction line	
Steam trap	1	Separates steam and condensate from the heat exchanger	

Table 7.6 Pipeline capacities.

Outside diameter of pipeline (cm)	Capacity (L m^{-1})
2.54	0.5
3.81	1.1
5.08	2.0
6.35	3.2
7.62	4.6
10.16	7.9
15.24	18.0
20.32	31.0
25.40	50.0
30.48	72.0

Table 7.7 Pipeline cleaning capacities (in metres of pipeline) for different detergent tank capacities.

Tank capacity (L)	Outside diameter of pipeline (cm)									
	2.54	3.81	5.08	6.35	7.62	10.16	15.24	20.32	25.40	30.48
1000	2000	909	500	313	217	127	56	32	20	14
2000	4000	1818	1000	625	435	253	111	65	40	28
3000	6000	2727	1500	938	652	380	167	97	60	42
4000	8000	3636	2000	1250	870	506	222	129	80	56
5000	10000	4545	2500	1536	1087	633	278	161	100	69

Table 7.8 Pipeline cleaning capacities (in metres of pipeline) for different cleaning velocities.

Cleaning velocity (m s^{-1})	Outside diameter of pipeline (cm)									
	2.54	3.81	5.08	6.35	7.62	10.16	15.24	20.32	25.40	30.48
1.5	45	99	180	288	414	711	1620	2790	4500	6480
1.6	48	106	192	307	442	758	1728	2976	4800	6912
1.7	51	112	204	326	469	806	1836	3162	5100	7344
1.8	54	119	216	346	497	853	1944	3348	5400	7776
1.9	57	125	228	365	524	901	2052	3534	5700	8208
2.0	60	132	240	384	552	948	2160	3720	6000	8640
2.1	63	139	252	403	580	995	2268	3906	6300	9072

Table 7.9 Pressure drop in pipelines (MPa 100 m^{-1} of pipeline).

	Outside diameter of pipeline (cm)					
Flowrate (L m^{-1})	2.54	3.81	5.08	6.35	7.62	10.16
90	0.47	0.07	0.017	0.006	0.002	
270		0.49	0.11	0.043	0.017	0.004
450			0.28	0.01	0.04	0.01
540			0.38	0.16	0.058	0.015
630			0.50	0.19	0.073	0.018

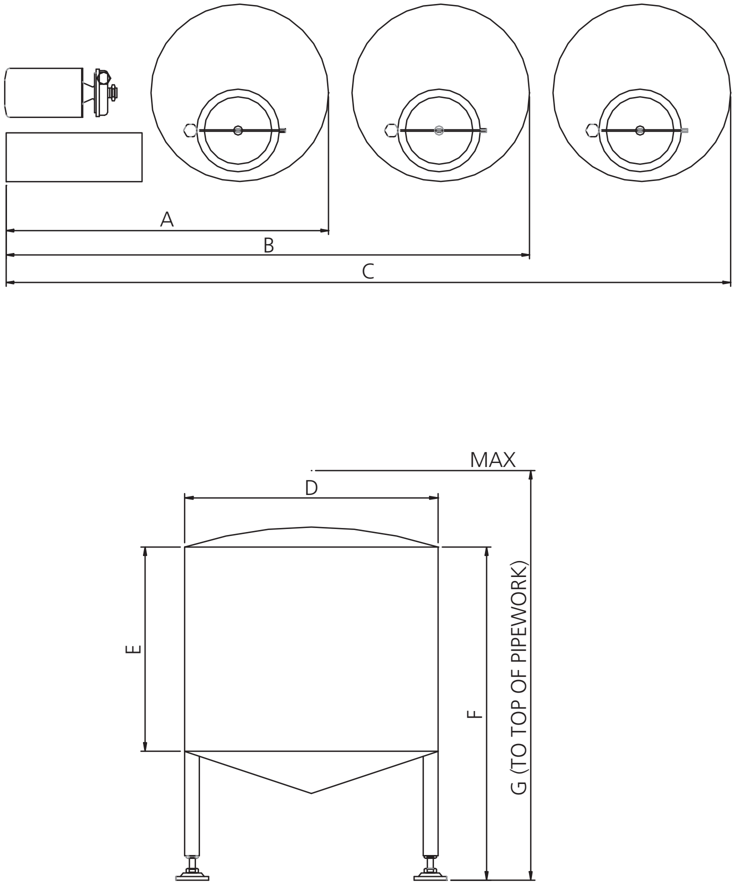


Fig. 7.9 Illustrations of CIP tank dimensions (see Table 7.10).

Table 7.10 Approximate dimensions (cm) of the CIP plant shown in Figure 7.9.

Capacity (L)	A	B	C	D	E	F	G
1500	230	350	470	120	150	220	260
2000	242	375	507	132	150	220	260
3000	250	390	530	140	200	270	310
4000	270	430	590	160	200	270	310
5000	290	470	650	180	200	270	310

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Lloyd, D. (2003) *The CIP Handbook*, D.B. Lloyd Ltd, Stroud.

8 Assessment of Cleaning Efficiency

K. Asteriadou and P. Fryer

8.1 Introduction

It is important to establish the definition of ‘clean’ in equipment and pipelines, and then determine the actions needed to maintain that situation. The assessment of cleaning efficiency is the means by which the performance of any cleaning system is validated, verified and monitored. The process of assessment requires:

- setting of standards
- reliable methods of performance measurement
- recording and reporting of results
- interpretation of results

Assessment should always lead to appropriate action when defects in the system have been identified, and an algorithmic sequence for an optimal cleaning method is shown in Figure 8.1.

The aim of this chapter is to give guidance, and offer suggestions on techniques and methods for assessing the effectiveness of cleaning in dairy, food and beverage production lines. Many researchers have developed new methodologies or amended existing ones (Grasshof, 1994). Others have emphasised the need to deliver techniques that can be applied in the industry – both in-line and off-line – with reliability, ease of use, and accuracy (Hasting, 2002, 2005; see also Cramer, 2006). Usually cleaning is poorly understood, and can take up a lot of production time.

8.2 Validation

Validation should ensure that the information supporting the cleaning process is correct. It is the method that determines which is the right cleaning process. It takes place before implementation and after alterations (e.g. new products, new product formulation, line changes and process parameters).

After setting up the new or altered process, validation will usually include:

- Monitoring of process parameters (e.g. temperature, flow, velocity), and ensuring that they remain within the limits required.
- Bioluminescent ATP assay. Adenosine triphosphate (ATP) is the chemical compound in which energy is stored in all living cells. In the ATP-luminometric test, the firefly enzyme (luciferase), in the presence of its substrate, luciferin, oxygen and magne-

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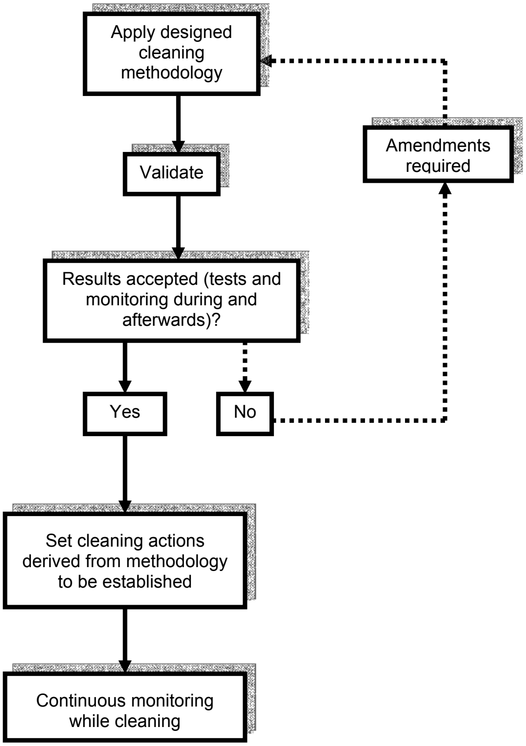

Fig. 8.1 Algorithmic representation of an optimal cleaning method.

sium ions, catalyses conversion of the chemical energy of ATP into light through an oxidation–reduction reaction. The quantity of light generated is directly proportional to the amount of ATP present: thus the light units can be used to estimate the biomass of cells in a sample. With state-of-the-art equipment and highly purified reagents it is possible to detect amounts of ATP corresponding to approximately 100 bacterial cells (Hygiena International Ltd, http://www.hygiena.net/tech_library-article-01a.html). ATP is present in all living organisms, and is also present in large quantities in a variety of foodstuffs as non-microbial ATP; it may also be present as free ATP. Thus the presence of ATP in both food debris and viable micro-organisms allows the dual detection of these sources of contamination using the technique of ATP bioluminescence described above (Hawronskyj & Holah, 1997).

- Direct microbial counts in rinse waters.
- Redox reactions (e.g. persulphate technology, Thonhauser GmbH). This is usually carried out on a line or piece of equipment that has just been cleaned with the already applied kind of cleaning. The line is filled with an aqueous solution of a specified concentration of a persulphate and potassium permanganate ($KMnO_4$) of known pH and temperature (usually the one applied for cleaning). Oxidation–reduction reactions occur in which the organic substance is oxidised and the potassium permanganate ($KMnO_4$) is reduced. The $KMnO_4$ is continually re-oxidised by the persulphate, which is a stronger oxidising

agent. The various oxidation states of the permanganate impart different colours to the solution. The colour reading leads to quantification of the organic matter.
- Visual inspection of open lines after cleaning is finished (see Section 8.2.1).

8.2.1 *Preliminary examination*

Properly cleansed plant, such as tankers, silos, pipelines and valves, should smell clean and fresh, with no trace of stale or sour odours, or excessive odours of chlorine or other disinfecting agents.

Sour smells may be due to:

- inadequate cleaning, particularly in the pre-rinse cycle;
- a small pocket of infection or area of deposit on the surface.

Stale smells may be due to:

- inadequate cleaning, for example due to low detergent strength or temperature;
- poor-quality final rinse water, for example due to insufficient chlorination;
- a vessel (or plant) allowed to stand for more than 24 h between cleaning and reusing.

*Excessive smell*s of chlorine or other disinfecting agents may be due to overdosing, which may in turn lead to taints developing in the product. It is also a considerable waste of money.

8.2.2 *Visual examination*

The standards for stainless steel surfaces of plant, vessels, pipelines and valves are typically that they should be bright, and free from residual moisture, surface film, scale, milk solids, scum and loose debris (such as gritty or powdery deposits).

A torch should be used to illuminate surfaces. Stainless steel should be bright and shiny, with no signs of dulling or deposits. (Note that it will be necessary to enter road tankers, or other vessels having a manhole fitted on top, to carry out an efficient inspection.) The surface of the metal should be cool to the touch. The position, colour, thickness and nature of any deposits should be noted. Any pooling of water on the bottom of a vessel should also be noted, and a sample should be taken for a pH test. A clean, sterile, plastic spatula should be used to gently scrape the metal to remove deposits, which may then be placed in a sterile sample pot for further examination or demonstration.

The condition of gaskets should be noted with regard to deposits such as milk solids, dirt or other foreign matter, and for integrity of the surface. Where possible, the gasket should be removed and the undersurface and metal in contact with the surface of the gasket examined for product solids, dirt or other foreign matter.

When inspecting tankers, particular attention should be paid to the plug cock valves, blank ends and dead ends (especially where these have been wrongly fitted at right angles to the flow of CIP fluids), vacuum release valves, manhole lids, hoses and interceptor bowls.

If the vessel or pipelines are still warm to the touch after cleaning, an insufficient final rinse may be the cause.

Hard deposits are comparatively thin and difficult to remove, and may cover the surface completely; a typical such deposit is scale, which is a combination of milk solids and hard water salts, and the time, strength, pressure and suitability of the detergent should be checked.

Soft and bulky deposits are usually localised, and consist of fat, non-diluted solids and some detergent residues. They often have an offensive smell, and they tend to occur where the CIP spray does not reach, because:

- the spray device is partly blocked;
- the wrong type of spray device has been fitted, e.g. one single arm instead of a T-spray, or the CIP inlet is offset to one side of the roof of the tank instead of in the more normal vertical/central mounting;
- the wrong type for that CIP system has been fitted, e.g. the spray holes are too small and/or the pressure is too high, causing atomisation of CIP fluids; alternatively, the holes may be too large and/or the pressure too low, so the spray does not reach the ends of the vessel;
- a rotary spray head has become seized, or is not spinning;
- the spray device has been damaged; or
- there is a varying delivery pressure caused by a blocked filter on the CIP delivery line, or poor recovery from the CIP circuit so the feed pump is starved.

Transparent and gelatinous deposits may be impossible to see from the manhole, and indeed the tank surfaces may look clean and shining. Even at close quarters the deposits may not be detectable unless the surface of the metal is scraped, thus removing the material, which is brownish in bulk, and odourless. This type of deposit is extremely slippery, and care should be taken when walking inside a tank. Factors leading to its formation and accumulation may be connected with other faults in the CIP, such as incorrect temperature, pressure and strength of detergent.

'Scum' and froth deposits, particularly in a 'bathtub' ring towards the top of the road tanker, indicate:

- flooding of the tank during cleaning due to inadequate scavenging;
- inadequate pre-rinsing;
- a faulty joint, allowing air to be sucked in; or
- detergent strength that is too high.

Gritty or soft powdery deposits may be due to hard water scale, or to metal particles from the CIP tanks/pipelines.

Deposits in plate heat exchangers may be hard scale or soft gelatinous matter resulting from long pasteurising runs and consequent 'bake-on', or from inadequate cleaning. Deposits towards the bottom of the plates in the raw milk regeneration section (i.e. prior to clarification or filtration) of the plate heat exchanger may be due to high levels of visible dirt in incoming raw milk supplies. Similar deposits in the heating section may indicate inefficient clarification or filtration.

More detailed information and guidance regarding the design and application of spray cleaning devices will be found in Chapters 6 and 7, and for further information regarding the cleaning of plate heat exchangers, see the *Pasteurizing Plant Manual* (Green, 1983). However, water remaining in the vessel may be due to:

- inadequate scavenging;
- inadequate venting;
- an airlock in the CIP circuit;
- insufficient slope on the tanker bay roadway, so that water does not drain to the outlet; or
- a deformed bottom to the vessel.

If the pH of the water is high (alkaline) – or acid, if an acidic detergent is used – the final rinse cycle is inadequate.

8.2.3 *Action following an unsatisfactory preliminary examination*

Adverse reports following routine visual inspection usually require straightforward remedial action. Damaged and worn parts should be replaced. It may be necessary to change from an alkaline to an acidic treatment to remove scale. Many systems recommend the periodic change from one to the other at routine intervals. Parts of filling equipment that are normally cleaned in place may periodically require stripping down and manual cleaning.

The above tests should ensure that the design of line and process is correct, and that continuous monitoring and verification protocols can be set up.

8.3 Verification

Verification determines whether the process agreed after validation performs in the right way. It is a continuous process, similar to monitoring, but with a lower frequency. Verification refers to the whole process, whereas monitoring evaluates specific points in the process.

These aspects are usually non-intrusive, and take place after the production or cleaning run. The methods used (microbiological and chemical) to monitor the efficacy of cleaning are detailed below.

8.3.1 *Surfaces*

Swabs are preferred for quantitative results, because contact slides give poor transfer of organisms from the test surface to the slide. Swabs should also be used to test for the presence of specific bacteria, and for less accessible areas.

Rodac (replicate organisms detection and counting) or contact plates are surface contact plates containing agar. They are recommended for the detection of micro-organisms on non-porous surfaces. With the use of Rodac plates, a facility can monitor sanitation levels with before-and-after colony counts (Merck, 2002, http://service.merck.de/microbiology/tedisdata/prods/4976–1_07084_0001.html; Pharmacal, http://www.pharmacal.com/Monitoring.htm).

8.3.2 *Flush/rinse material*

- Direct counts of first production batch and rinse water.
- Water analysis for disinfection/sanitation: e.g. pH, total suspended solids (TSS), hardness (see subsequent section and Chapter 3).

8.3.3 *Water quality*

In general, water makes up more than 97–99 g 100 g^{-1} of the cleaning solution, and should get serious attention. In most cases the water used for making up the cleaning solution is also used for intermediate and final rinses. Special attention should be given to both its microbiological and its inorganic content.

Soluble iron and manganese salts in concentrations above 0.3 mg L^{-1} will cause coloured deposits on equipment surfaces. If the hardness of the water is high (> 60 mg Ca^{2+} L^{-1}), the water might be less suitable for cleaning purposes, and partial softening should be considered. Scale formation can be reduced by adding acid in order to lower the pH. However, excessive acid results in corrosion of metals.

Water softeners should not be used after chlorination as this might cause taint problems. However, water, as a product ingredient or cleaning agent, must be monitored by testing for total viable counts, and for total and faecal coliforms and *Escherichia coli*, at a point as close as possible to its entry to the plant.

In the United Kingdom, water supply regulations allow specific maximum limits (DETR, 2000), as shown in Table 8.1.

8.3.4 *In-process material*

- Dip slides.
- Protein on surface. For example, the Pro-tect M test from Biotrace is a rapid, sensitive and easy-to-use protein detection test that can be used to measure cleaning efficiency. It

Table 8.1 UK water supply regulations; maximum limits.

Microbiological criteria for potable water	Maximum concentration (colony forming units, cfu)
Total coliforms	0 cfu 100 mL−1
Faecal coliforms	0 cfu 100 mL−1
Faecal enterococci	0 cfu 100 mL−1
Sulphite-reducing clostridia	<1 cfu 20 mL−1
Total viable count	No abnormal change observed within testing period (2 successive years)

is based on the colour change after a reaction takes place with residual protein. If protein is present, the test will change from green to purple. Its use as a method for validating cleaning has been regularly demonstrated (Patrick & Bayliss, 1997; Tebbutt, 1999).

It is very common to find that there are specific points in a line at which sampling should be carried out within the verification process. Some examples are:

- bypasses;
- critical valves, e.g. flow diversion valves between pasteurised and unpasteurised products;
- dead ends and T-pieces;
- drain valves;
- filler heads;
- following heating or holding areas (e.g. in pasteurisers or sterilisers);
- inside the tanks;
- recirculation pipes; and
- sampling valves.

Samples can also be taken after each cleaning step to test for organic material residues and bacterial counts. The frequency of sampling is discussed in more detail in the next section.

8.4 Frequency of assessment/sampling

8.4.1 *Equipment/surfaces*

The frequency of assessment or sampling will be determined by the way in which the cleaning systems are programmed but, in general terms, a product sample should be taken every time a clean vessel or other plant is used for production.

Storage vessels (silos, balance tanks, finished milk tanks) should be inspected regularly – weekly, if possible.

Plate heat exchangers in the industry tend to be inspected on a six-monthly basis, as the opening of plate packs is time consuming, and can be very costly if gaskets are damaged.

Pipelines should not be dismantled unless serious problems, indicating a probable fault in such lines, are encountered.

Other processing equipment, e.g. clarifiers, homogenisers or pumps, should also be inspected, for example when opened for maintenance, unless a hygiene problem is suspected, in which case they should be inspected immediately.

Fillers, which are cleaned entirely in place, with no requirement to dismantle and reassemble, should be examined at the end of each cleaning cycle, paying particular attention to all accessible areas. Where some dismantling, manual cleaning and reassembly are required, the inspection process is a necessary step before reassembly. Care must be taken to avoid recontamination during reassembly.

Inspection of *miscellaneous equipment* should, in general, include checks to ensure that blank ends are fitted to broken pipework, and that all pipeline joints are tightened up after the cleaning cycle has been completed.

The condition of *flexible hoses* in the reception area should be checked daily. Blank ends should be fitted to hoses when they are not in use, and correct storage facilities should be provided for flexible hoses when not in use.

Storage vessels may have difficult-to-clean areas such as agitators, thermometer probes, sample points and under the internal lip of the manhole; particular attention must be paid to such areas.

8.4.2 *Product*

Method of sampling

Where possible, a filled package, ex-line, should be taken, otherwise samples must be taken aseptically. The value of the test result is only as good as the sample. Sampling from tankers and other vessels without a sample valve should be by use of a sterile dipper. Where sample valves are installed, care should be taken to ensure that they are either cleaned in place, or cleaned manually each time the vessel is cleaned. When sampling, care must be taken to sterilise the valve thoroughly before use, either by swabbing with industrial methylated spirits, or by using a proprietary disinfectant spray. (Note that the purple methylated spirits sold for domestic use contains pyridine and is unsuitable for sterilisation.) A quantity of milk should be run to waste before the sample is taken. The hands of the person taking the sample should be clean and dry.

Heat-treated product

The first heat-treated product through the plant after start-up should be sampled and subjected to the presumptive microbial test. Suitable sample points include the cooler exit from the pasteuriser, finished product tanks, filler bowls (into laboratory-sterilised bottles from the filling heads on the bottling lines), the first packages filled (which are usually discarded to avoid contamination with water) and the first saleable ('commercial') packages. The use of selective or differential methods can be useful in troubleshooting: such methods include spore counts, thermoduric counts, psychrotrophic counts (either 7 days at 5°C or 25 h at 21°C), and confirmatory tests for coliforms.

Packed product

The product filler is the most serious potential source of contamination. This is because the filler cleaning system may well include a degree of manual cleaning, with problems associated with recontamination after cleaning and during reassembly. The first packed product through any filler after cleaning should be sampled and subjected to microbial tests, where the results should again show that coliforms are absent in 1 mL.

Where the filler is required to fill aseptically, as in the case of ultra-heat-treated (UHT) milk for example, a high degree of plant cleaning efficiency is required, followed by sterilisation, generally with steam. Sterility after this process would normally be assessed by subjecting a sample of the first packed product to a colony count test. The details of the test are given in MAFF (1992). The number of colonies should be fewer than 10 cfu mL^{-1}.

Swabbing and rinsing methods

The visual inspection of road tankers, storage vessels and plant may be supported routinely by a programme of swabs and rinses, as appropriate. The areas chosen for swabbing may be either a representative sample of the tankers, vessels and pipelines in regular use, or specific areas that are suspected not to have been cleaned effectively.

The purpose of swabbing is to collect physically any bacterial contaminants from the surfaces under examination and determine the level of that contamination. After use, the swab is placed in a quantity of sterile diluent. The determination is carried out by the plate count method for total viable count, and by a coliform test.

The disadvantage of swabbing is that the area swabbed tends to be an area that is comparatively easy to clean. The source of the problem may be an inaccessible pocket of infection, so a satisfactory swab result should be interpreted with caution if product test results are unsatisfactory.

Rinses are used for the same purpose in situations where a quantity of sterile diluent can be introduced to one end of the plant and collected aseptically at the other, having come into contact with the same surfaces as the product. This method is suitable for carton-filling machines, for example.

8.5 Monitoring

Monitoring refers to the regular measurements taken on the cleaning process that serve as indicators of whether the process is in a state of control. This might include process parameters already measured (or not), such as total organic carbon (TOC), or conductivity.

The monitoring methods can be either invasive or non-invasive. Effectiveness must be monitored according to a sampling plan that specifies methods, sampling frequencies and target values. It should be fairly easy, and provide results fast.

For surfaces of equipment, sampling may include:

- ATP – rapid, relatively simple to use;
- microbial swabs;
- visual inspection;
- test strips for residual protein testing (linking to allergens).

This last applies to the detection of allergens as well. The most common allergens are considered to be milk, egg, peanut, shellfish, tree nuts, fish, soy and wheat. The test methods available for allergens are:

- ATP – to detect proteins or organic materials;
- ELISA – the detection range of proteins is between 2.5 and 25 mg L^{-1}; and
- redox reactions – as an oxidation process of all organic residuals.

Monitoring of fluids includes:

- in-line monitoring of (a) pressure drop, (b) temperature differences, (c) turbidity, (d) conductivity (also to determine the sequence of the cleaning steps), and (e) daily continuous recording of time, pressure, flowrates, temperature and conductivity;
- environment monitoring (e.g. wet areas on or next to key process equipment, e.g. fillers, drain valves, suspected leaking areas).

The aim of monitoring is to ensure that cleaning procedures minimise and control the risks of product and line contamination, and that contamination does not increase downstream or between operation periods. Monitoring is strongly linked to verification, since it indicates whether the process is within the standards set beforehand.

8.5.1 *Results from system monitoring*

All sensors, such as the Pt100 for temperature monitoring shown in Figure 8.2, should be replaced when inaccurate, and immediate steps need to be taken to remedy circuits that fail to reach the recommended processing conditions. All measurement devices should be installed hygienically without forming dead ends.

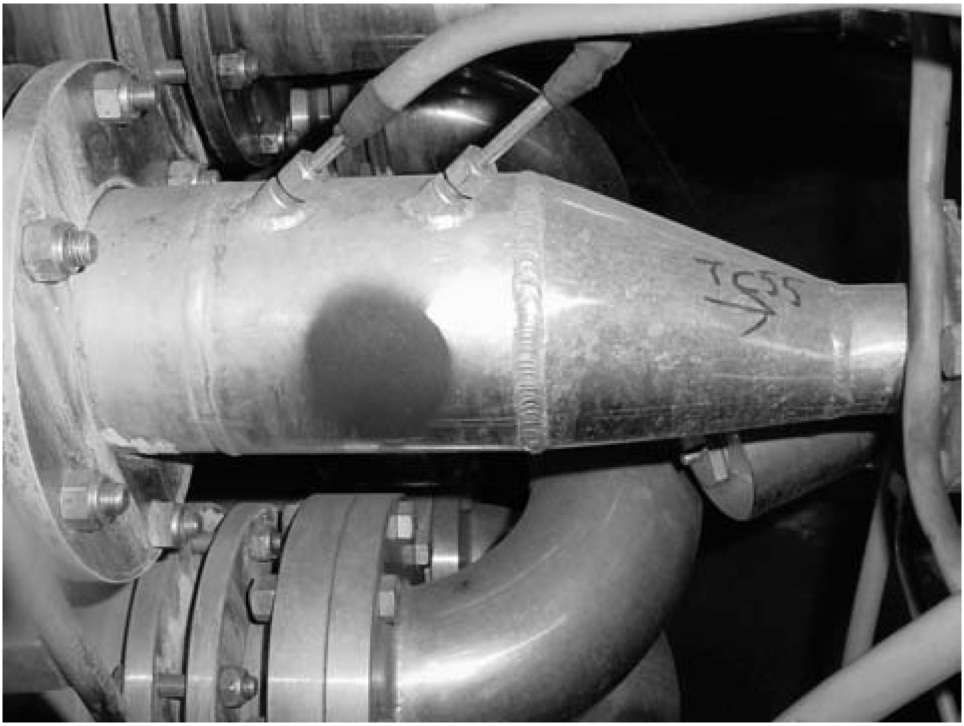

Fig. 8.2 Double Pt100 thermocouple positioned at a tube outlet. Reproduced by permission of Unilever R&D, Bedford, UK.

Unsatisfactory results obtained from detergent strength tests should be investigated to identify any fault in the automatic dosing equipment, or operator error if dosing is carried out manually. Daily visual inspection of detergent samples should indicate the point at which the detergent should be discarded and replaced owing to contamination with product residues. Detergent in continuous use in the raw milk reception area may require replacement on a weekly basis. Care should be taken to avoid over-chlorination of the water supply, which, if used for a final rinse, may lead to the development of a taint in the milk that enters the tank after cleaning. Faults in automatic dosing equipment should be remedied quickly.

The use of caustic soda alone as a detergent can cause problems in areas of high water hardness. Under strongly alkaline conditions carbonate salts are precipitated from solution, which may result in a whitish film being left on the surface of plant after the cleaning cycle. The addition of ethylenediaminetetra-acetic acid (EDTA), sodium gluconate or sodium hexametaphosphate (Calgon) to the formulation acts as an efficient sequesterant, helping to keep the carbonate in solution.

8.5.2 *Interpreting results and taking action*

The meaningful interpretation of results depends on keeping clear and accurate records. Laboratory staff should keep systematic records of all tests carried out. Each entry should show the date and time of sampling, and the origin and type of the sample should be clearly identified. Results should be entered clearly and neatly in ink, and those that are out of line should be readily identifiable. Out-of-line results should be reported daily to dairy management, so that prompt action can be taken. The design of report forms should be simple and straightforward so that results can be followed in logical sequence. The target or standard for each test should be clearly indicated.

Out-of-line product test results may be related to an inadequately cleaned tanker. The results should be interpreted in the light of the visual appearance of the tanker on arrival, the temperature of the product, and the length of time it has been in transit.

Accommodation tanker supplies should be accompanied by a consignment note that, among other parameters, indicates that the tanker has been inspected before dispatch, and cleaned and sterilised if necessary.

With results rapidly available, an investigation can take place while the operator's memory of events is still clear. The problem can be identified and a course of action agreed before the next cleaning cycle takes place. In the event of a significant increase in the total viable count, the storage vessel should be inspected visually and, if necessary, swabs should be taken. Spray devices (e.g. balls and/or rotating jets) should be checked to ensure that they are in place and working correctly. Further checks of detergent strength, temperature and pressure should also be made.

Positive results in a presumptive coliform test taken from a batch of first pasteurised product will point to likely post-pasteurisation contamination. Samples that are clear at the cooler exit of the pasteurising plant but positive in the finished product tank will indicate that the problem is likely to be tank cleaning. Samples that are clear before the filler but positive in the packed product will point to a filler cleaning problem.

For these reasons, should a high spore count be found in the finished product, the spore count of the raw materials should be checked and compared with the finished product. A large increase suggests that the CIP system should be thoroughly investigated and an acid clean instigated as soon as possible in the case of dairy plants.

Thermoduric non-spore formers are probably not of any great significance with regard to keeping quality: they usually originate from raw milk, and are not active milk-souring organisms. However, they do contribute to the total viable count.

The presence of psychrotrophic organisms is another indication of post-pasteurisation contamination, as these organisms are destroyed by pasteurisation. They can be a significant problem in chilled products, and also in liquid milk now that a prolonged shelf-life is the norm. Their presence in high numbers is an indication that the CIP system should be thoroughly investigated. Single out-of-line results may not be significant, but each result should be noted and investigated. A trend of several out-of-line results is likely to be significant, and care should be taken to pinpoint the problem.

When carrying out an investigation into cleaning efficiency that involves the taking of swabs, it should be borne in mind that a comparatively small pocket (i.e. dead end, see Figure 8.3) of trapped product may lead to high residence times under conditions that favour microbial growth. If these are not properly cleaned, the new product passing from that point could end up heavily contaminated (Asteriadou *et al*., 2006). Unfortunately, these dead ends are usually inaccessible, making surface and product sampling an unsuitable method. This is why the cleaning process should be well designed in order to ensure that those areas are not a risk (Asteriadou *et al*., 2007).

Investigation is likely to indicate that action is necessary in one or more aspects of the cleaning process. The temperature, strength and pressure of detergent may require adjustment. There may be insufficient circulation time. Scale and other debris may be found, and dead

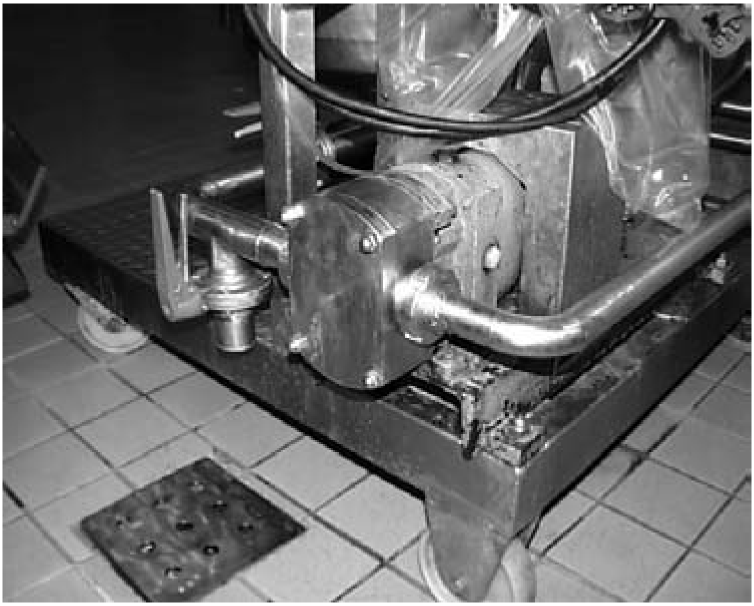

Fig. 8.3 Dead end formed by a sampling/draining valve. Reproduced by permission of Unilever R&D, Bedford, UK.

ends of pipework may not have been included in the circuit. Recontamination may be taking place during manual assembly, and some parts may not be receiving an adequate clean.

8.6 The commercial benefits of assessment

The routine assessment of cleaning efficiency achieves the following commercial objectives:

- economic cleaning, with costs under control;
- early warning of possible product failure;
- high product quality, with confidence based on consistently good results from the plant cleaning programme;
- longer production runs;
- longer equipment life, and reduced maintenance expenses.

8.7 Conclusions

Cleaning of equipment is a necessary step in food and beverage production. It can be costly of both money and time, but it is there to ensure safe products and long-lasting lines and equipment. Therefore it is crucial to perform it in the most suitable way, depending on the product and the line needs, and consequently it becomes essential to operate it with efficiency. In order to succeed in this it is important to establish a set of assessment criteria that will include validation, verification and monitoring of the cleaning process. Correct application of these and their various steps leads to a clean line as required according to the standards initially set.

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9 Management of CIP Operations

K.J. Burgess

9.1 Background to cleaning-in-place (CIP)

CIP is in widespread use in the dairy and brewing industries. Similar cleaning approaches are used in the wider chemical processing industries, giving us an important insight into the nature of CIP operations.

The context is industries where the focus is liquid processing, and several unit operations are linked by connected pipework. In such arrangements it is not practical to disconnect and disassemble equipment, thus leading to the CIP concept, where equipment and connecting pipelines are cleaned in their process configuration. This fundamental difference from manual cleaning means that CIP brings a number of advantages and challenges that need to be considered in the operation of CIP systems. The advantages can be summarised as follows.

- Usually, a better standard of cleaning is achieved, because stronger and higher levels of chemicals and temperature can be used.
- Chemical and water consumption are less than with manual systems
- In some CIP systems, the chemicals and water can usually be recovered.
- CIP systems are usually highly automated, and therefore more efficient and reliable than manual cleaning.

The challenges associated with CIP systems are as follows.

- Because the plant is not disassembled in CIP, it is not possible to physically ‘see’ whether the plant is clean or not.
- Because cleaning chemicals follow the same path as product flows, there is an increased risk of cleaning chemicals contaminating product.
- Because water and chemicals are reused, there are increased opportunities for cross-contamination.
- The use of strong chemicals, concentrations and temperatures gives rise to greater risks to human health and safety.
- There is a much higher capital cost associated with CIP.

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9.2 Some CIP operation basics

9.2.1 *CIP parameters*

The key parameters for CIP are no different from those important to manual cleaning, i.e. three sources of energy, together with a length of time:

- chemical
- physical/mechanical
- heat energy
- time

In setting up a CIP programme, it is important to understand which of the energy sources is the most effective in achieving the desired clean, and what is the best balance of energy input and time.

9.2.2 *People involved*

The operation of CIP systems involves many different groups of people from different disciplines:

- design engineers
- plant manufacturers
- automation/control suppliers
- detergent suppliers
- operations managers
- engineering managers
- technical and quality assurance and control staff
- health and safety, and environment managers

Each of these groups of people will have different perspectives in terms of the priorities for the CIP operation, and it is important that senior management exercises the appropriate judgement to ensure the correct balance between achieving quality standards, productivity and running costs, and health and safety standards.

9.2.3 *Key steps in CIP implementation*

As with any other manufacturing process, there are a series of steps involved in leading up to the successful operation of a CIP system:

- design
- construction
- installation
- operation

Earlier chapters have dealt with important design and construction issues, but it is important to remember that the CIP installation is defined as much by its plant hardware as by the computer software controlling it. The installation step includes the crucial phase of *commissioning*, where the plant and its control system are tested to ensure that all of the component parts are working effectively, and that the combination of components and management systems is interfacing correctly.

With the increasing complexity of larger CIP systems, it is particularly important that the commissioning process includes a *challenge* to the computer control system. Computer control systems include many lines of software programming, and this represents a large opportunity for error. The CIP control software should therefore be challenged in a series of *simulation exercises* where it is subjected to potentially incorrect operator instructions to test its integrity.

9.2.4 *A typical CIP sequence*

A typical CIP sequence will comprise the following cycles:

- pre-rinse
- detergent circulation
- intermediate rinse
- additional detergent circulation (optional)
- additional intermediate rinse (optional)
- disinfectant rinse (optional)
- drain

The *pre-rinse cycle* removes the loose soil: the more of this that can be removed with a simple rinse, the less the need for the chemical, mechanical and thermal energy needed later. Rinsing efficiency can be improved through 'burst rinsing' of vessels, and through ensuring that draining surfaces are on a slope.

The *detergent circulation* is usually the key stage in removing residual soil. Circulation time is typically 10–30 min, but the choice of detergent very much depends on the particular cleaning situation. Examples of detergent/temperature solutions are shown in Table 9.1.

An *intermediate rinse* is then required if a further detergent circulation is going to be used. This is particularly the case if an acid treatment is necessary, usually when scale removal is required.

Table 9.1 Some examples of detergent/temperature solutions in a CIP system.

Type of detergent	Temperature (°C)	CIP operation
Nitric acid	60	Tanks, pipelines
Caustic soda	50–80	Tankers, tanks, filling machines
	70–90	Pasteurisers
	90–130	UHT

A *final rinse* is then required to remove detergent residues, and apply a disinfectant if necessary. An example of the latter compound would be peracetic acid, and the final rinse can be recovered and used as a pre-rinse in the next cycle. Depending on the chemical used, or the nature of the operation, e.g. organic product manufacture, a further rinse with potable water may be required. At the end of the CIP sequence, the plant then needs to be *drained*.

9.2.5 *Cleaning the CIP system*

One key element in the management of CIP operations that is often overlooked is the cleaning of the CIP system itself. This is essential to prevent build-up of residue on the tank walls, which can lead to microbiological contamination of the objects being cleaned. With a fully automated system, a CIP cleaning cycle should form part of the full service routine, and in this cleaning cycle provision should be made to:

- drain 50% of the detergent solution to remove any sediment from the tank;
- drain all other tanks in the CIP system;
- recirculate the detergent via in-built spray devices in sequence through the detergent tank(s), recovered water tank and final rinse tank;
- rinse all the tanks in sequence with clean water; and
- make up all the tanks to the correct volume/concentration and temperature.

Where a fully automated system is not installed, safe access must be provided to enable the tanks to be drained and, at the very least, hosed out.

The frequency of this operation is somewhat dependent on soil levels within the detergent, and on the hardness of the water used within the system, but, even where detergent contamination with product residue is low, the minimum frequency for the set to be cleaned should be every two months.

9.3 Chemicals and chemical suppliers

Selection of detergent and disinfectants on the basis of their cleaning and bactericidal effects has been covered in Chapter 4. It is important, in the context of managing CIP operations, that the role of the detergent supplier be understood as a major driver of success. The practice of making up detergent blends on site is not to be recommended, because it is highly unlikely that the expertise will be available on site to achieve the right cleaning effect in a safe manner.

Dairies operating CIP systems therefore need to identify a reliable supplier or suppliers of CIP chemicals. In addition to the issues important in any supplier, i.e. the customer relationship, there are some particular questions that should be asked:

- Is there a comprehensive level of technical support available on a 24 h 7 day basis?
- Does the supplier carry out a full survey of the site and CIP systems before recommending chemicals to be used?
- Does the supplier provide usage, handling and safety training for all of its products?

9.4 Troubleshooting CIP

Each CIP operation must be designed and operated on the basis of its particular characteristics. Nevertheless, there are several generic factors that can positively or negatively impact on CIP effectiveness. When troubleshooting a problematic CIP operation, it is good practice to check for the presence of positive factors that contribute to successful CIP, and for the presence of negative factors that will give rise to impaired performance.

9.4.1 *Positive factors*

The CIP outcome will generally be satisfactory when the following are met:

- correct water quality standard (softness, hygienic quality);
- correct temperature;
- correct flow and turbulence conditions;
- correct concentrations of detergent and disinfectant;
- correct detergent circulation times;
- detergent tanks emptied and cleaned regularly to minimise product residues; and
- planned valve maintenance that includes all CIP valves as well as mainstream process valves.

9.4.2 *Negative factors*

While it is obvious that CIP effectiveness can be adversely affected by inadequate time exposure, there are also various potential ways in which the application of the three different energy inputs can be misapplied:

- Chemical
  - insufficient rinsing of residual soil
  - detergent concentration too low
  - detergent concentration too high
  - insufficient rinsing of detergent residues
  - wrong choice of detergent/disinfectant
  - inconsistent dosing and concentration control
  - insufficient clean-up of reused detergent
  - insufficient cleaning of the CIP set
  - incorrect water hardness/treatment
- Mechanical
  - spray devices blocked up or operating with an incorrect delivery pressure
  - poor drainage
  - ‘deadlegs’ (where CIP flows cannot reach)
  - poor scavenging
  - low flowrate/pressure
- Thermal
  - temperature too low (insufficient cleaning effect)
  - temperature too high (baking-on of residues, seal damage)

9.5 CIP and operational goals

The considerations listed above should help to give a broad background to some of the factors involved in CIP system and problem-solving. The remainder of this chapter is focused on how CIP fits in with the operational goals of management and quality, safety, productivity, and review and improvement(s).

9.6 CIP management and quality

9.6.1 *Quality management system issues*

CIP operations are part of a wider manufacturing operation, which has the objective of supplying a product to a customer. CIP operations therefore need to be included within the scope of a *site quality management system*, which would be expected to include the following categories:

- procedures and work instructions
- training
- maintenance of equipment and control systems
- calibration of sensors and instruments
- monitoring and control processes
- documentation and records

More specifically, it is recommended that a CIP manual be assembled to document the following:

- the original specification for the CIP set
- record of commissioning trials/checks
- CIP flow directions
- cleaning sequences and circuits
- engineering drawing
- maintenance and calibration schedules
- quality checks and record proformas
- a record of all subsequent changes
- records of CIP reviews and verifications

9.6.2 *CIP and due diligence*

From a legal perspective, the above arrangements are likely to provide a good basis for a sound due diligence defence under the Food Safety Act 1990 (Anonymous, 1990). However, because of the importance of the four key CIP parameters (temperature, flow, chemical storage and time), a successful due diligence approach should include records of all of these aspects. A new CIP system should therefore provide for the automatic recording of these key four parameters.

A due diligence approach would also be expected to include a series of inspections of the CIP operation on a regular basis. Such inspection programmes should include:

- Daily
  - detergent and disinfectant concentration
  - temperature (on a chart recorder)
  - water hardness
  - visual inspection for leaks
  - confirmation that all CIP sequences have been completed in full and to time
  - confirmation of record keeping
  - trend monitoring of hygienic status of surfaces and products
- Weekly
  - integrity of in-line strainers/sieves
  - integrity of detergent and water storage tanks
  - integrity of rinse water
  - cleaning effectiveness (visual, swabs, product)

Less frequent inspections would be part of CIP reviews and verifications described in Section 9.9.1.

9.6.3 *CIP and hazard analysis and critical control point (HACCP) system*

A HACCP system consists of seven principles that, when applied in practice and in full, provide for ensuring the safety of food (Pierson & Corlett, 1992; Corlett, 1992; WHO, 1993; FAO, 1995; Anonymous, 1997; Mortimore & Wallace, 1998; Mayes & Mortimore, 2001). The seven principles of HACCP are as follows:

- conduct a hazard analysis
- determine critical control points (CCPs)
- establish critical limits for CCPs
- establish a system to monitor CCPs
- establish corrective actions
- establish procedures to verify the system is working
- establish documentation.

A CIP programme is a process in the same way that product manufacture is a process. The HACCP approach should therefore be applied to each cycle of the CIP sequence listed in Section 9.2.4. The most important output of this analysis is the identification of CCPs where critical limits are established, and appropriate corrective actions identified. On a day-to-day basis the most important critical limits for CIP are most likely to be related to the four key CIP parameters, i.e. flow, temperature, chemical strength and time. However, the monitoring process also needs to include attention to the routeing of water and cleaning fluids, and to the effectiveness of valve maintenance. A full HACCP analysis is sometimes seen as an onerous task, but every CIP set-up is different and warrants an individual perspective.

9.6.4 *Some aspects of good practice*

As mentioned elsewhere, the particular nature of CIP operations can give rise to challenges, but it is important to recognise that there are best practices that can be applied to minimise the adverse impact of these challenges. The most important of these are:

- total separation of CIP systems for raw and post-heat treatment equipment;
- visual identification of all pipework, to define the nature and flow of raw product, heat-treated product, and detergents;
- elimination of direct interfaces between product and CIP lines; where unavoidable, separation between CIP fluids and product should be achieved by one of the following:
  - double-seat valves,
  - block and bleed valve arrangement,
  - physical brakes, such as flow plates or key pieces, or
  - provision of at least two valve seats between product and CIP;
- using alarms and interlocks with the controls software to:
  - lock out items that have not been cleaned within agreed time periods,
  - ensure a CIP programme cannot be started if product is present, or the route is not complete,
  - lock out routes if the CIP programme has not been completed.

The loss of any such interlock during a clean should stop the clean and either alarm or shut down the CIP sets.

9.7 CIP management and safety

CIP operations have the potential to be hazardous because of the elevated chemical concentrations and temperatures in use compared with manual cleaning. In addition, there are risks arising from the movement and transfer of cleaning materials within the factory.

9.7.1 *Health and safety issues with CIP*

The Health and Safety Executive's six priorities for health and safety in the dairy industry (HSE, 2002) are all relevant to CIP operations, and need particular focus as they account for over 80% of all injuries. These priorities are:

- exposure to cleaning chemicals
- slips and trips
- falls
- machinery
- handling
- transport

Of these priorities, exposure to cleaning chemicals is a major safety issue that requires appropriate actions and precautions to be taken to ensure that employees' exposure to the chemicals used is adequately controlled.

Exposure to cleaning chemicals

Although chemicals, and particularly disinfectants, used in the dairy industry are especially selected so that potential residues left on surfaces do not taint the products or are harmful to the consumer, many affect the skin, eyes or respiratory system, and can be harmful if ingested in sufficient quantity. Because of this, a Control of Substances Hazardous to Health assessment (COSHH, 2005) should be undertaken to identify the purpose of the chemicals (steam, if used, should be included, as it can be very hazardous), and eliminate their use where appropriate, e.g. where cleaning alone is adequate or heat can be used safely.

A COSHH assessment (COSHH, 2005) should include:

- a list of all chemicals/hazardous substances to be used;
- their hazards;
- provision of measures to control operator exposure, covering safe storage, chemical compatibility, working concentrations and safe dilution procedures, application procedures and equipment, any air monitoring or health surveillance requirements, information and training requirements, provision of washing facilities, and an emergency action plan (e.g. for spillage).

The following measures should be considered when establishing safe chemical-handling practices.

Handling concentrates and dilution procedures

Full-strength concentrates are seldom used for cleaning purposes. Working concentrations should not exceed manufacturers' specifications, as overdosing increases the risk to operatives, in addition to being wasteful, and may damage plant and equipment. Where costs allow, purchasing disinfectants in their dilute form or in pre-pack quantities for direct dilution are the safer options. Where these are not available, or incur prohibitive costs, diluting concentrates by auto-metering or positive displacement using drum pumps are considered safer than gravity feed from taps. The latter may jam or be displaced, resulting in uncontrolled leakage into the workroom. Diluting concentrates by manually tipping drums or carboys is extremely poor practice; it will inevitably cause a spill risk, and should not be carried out.

Chemical penetration and contamination of personal protective equipment (PPE)

It is important to ensure that garment, glove and boot selections have the ability to resist penetration by the chemical concerned. Manufacturers and suppliers have duties to supply this information. However, although some types of PPE provide very high levels of protection, breakthrough will eventually occur, so none provides 100% protection. Also, skin exposure may occur when removing used PPE. Suitable disposable gloves offer an easy management system to eliminate hand exposure from gloves that become contaminated inside. Advice and information for operators is necessary to ensure that the PPE provides the protection needed.

Maintenance

Chemical application equipment should be regularly maintained. Exposure control equipment should be kept in efficient working order and good repair. PPE/RPE (respiratory protective equipment) should be examined and, where appropriate, tested at suitable intervals. Gloves should be inspected visually every time they are used. Disposable gloves should be used only once if chemicals are handled.

Information and training

Operators should be informed about the hazards of the chemicals they work with, and the risks created by exposure to these chemicals. They should be instructed in the precautions to take and in how to use the control measures provided, and should also be informed of the results of any air monitoring and health surveillance.

Washing facilities

In many cases, skin is the principal organ that may be exposed to chemicals, so ready access to washing facilities is essential. Employers should ensure that suitable facilities are provided to allow operatives to clean themselves after using chemicals, and before eating and drinking. PPE should be cleaned after use and stored separately from everyday work wear. Where showers or eye-sprays are provided, they should be frequently flushed through, and account should be taken of a potential risk from *Legionella* spp., for which appropriate precautions should be taken.

Emergency procedures

Emergency procedures should be in place, particularly when larger quantities of concentrated chemicals are being handled. Emergency washing facilities (e.g. showers, eye-wash stations) should be available, including measures to irrigate eyes in the event of splashes.

The material safety data sheet should be consulted to obtain the appropriate method for handling spillages: this may include, for example, spill trays or absorbent granules. Environmental issues should be considered in the event of a spillage, particularly to prevent spillages from running into storm or surface drains.

Slips and trips

The precautions to be taken against slip and trip hazards due to wet conditions and obstacles, for example, are as follows.

- Take measures to avoid spillage and leakage onto floors, stairs and walkways.
- Make sure a system for cleaning spillages is in place, and is followed.
- Try to schedule floor cleaning when work is not in progress, or has finished for the day.
- After cleaning, dry floors as much as possible.

- Remove obstructions in walkways regularly.
- Maintain floors and stairs in good condition.

Falls

Some typical examples of falls are from ladders and on stairs. Therefore: (a) eliminate the need to work at height and, if not possible, always provide a safe means of access, a safe working area, and the correct equipment for the job; and (b) check the condition of stairways regularly.

Machinery

During maintenance and cleaning, machines such as conveyor belts have to be made safe. Example measures are as follows.

- Ensure guards are kept in place, regularly examined and properly maintained, and encourage people to report faults.
- Train employees, and provide them with information about the hazards of the machinery they are working on or will be cleaning.
- Ensure relevant parts of plant and equipment are appropriately isolated from hot or hazardous materials where access is required.

Handling

Consider safe manual transfer and dilution of heavy or awkward to handle chemicals. Measures to be considered are as follows.

- Eliminate unnecessary manual handling by assessing each job, and providing a mechanical alternative wherever possible.
- Avoid awkward or heavy lifting.
- Provide training to employees in the correct lifting techniques to use.

Transport

Lorry movement and lift trucks within a factory have to be made safe and, when considering tanker movements, security and access during cleaning, the safety measures include the following.

- Ensure safe access when fitting/connecting spray balls.
- Ensure tanks are cooled appropriately to prevent implosion.
- Segregate pedestrians and vehicular traffic, wherever possible.
- Eliminate the need to reverse vehicles, and where this is not possible set up a safe system for reversing.

9.7.2 *Safe CIP*

Safe CIP requires a good management system to help identify problem areas, decide what to do, act on decisions, and check the steps have been effective. A good system should involve:

- planning to make sure the correct cleaning regime is chosen, by whom, when it should be used, what contaminants will be present, and how spillages will be cleaned up;
- organising work and consulting with staff to ensure the planning stage is implemented;
- control to ensure working practices and processes are carried out correctly;
- monitoring and reviewing to identify improvements that can be made to the system;
- effective communication at all levels;
- effective training and supervision to match the individual, the environment and the equipment.

9.8 CIP management and productivity

The economic operation of CIP systems is a significant proportion of overall operation costs, and it must therefore be analysed and monitored frequently in order to maintain cost-effectiveness. However, CIP productivity is determined primarily through close control of CIP cost inputs, through making best use of product recovery options, through the best reuse of water and detergent chemicals, and through freeing up time for increased production capacity. The latter two aspects have been covered in Chapter 8, so the discussion here will focus on CIP cost inputs and product recovery.

9.8.1 *CIP cost inputs*

Cost inputs to a CIP operation have been estimated as follows (Sharp, 1985):

- labour and supervision 41%
- detergents 8%
- energy 12%
- water and effluent 21%
- maintenance 7%
- other costs 11%

The relative importance of the various costs will obviously vary over time, and with the nature and complexity of the particular operation. However, it is apparent from this breakdown that the most significant advantage can be made in the areas of automation and water/effluent reduction. It is for this reason that modern CIP operations are almost totally automated, with minimum need for human attention. At the same time, this places even more reliance on the effectiveness of the valves and control software, and that is why the good practice measures listed in Section 9.6.4 are so important.

Management of CIP cleaning costs is the key driver for managing CIP productivity, and while the above breakdown is a useful guide for a starting point, it is important to establish a specific cost per unit clean for each particular CIP operation. The list of costs to be included in the unit cleaning cost is actually somewhat broader than those listed above, as seen in the following:

- detergents
- product wastage
- water
- effluent treatment
- steam
- electricity
- labour
- maintenance
- loss of production capacity
- capital cost of plant and equipment

The regular monitoring of these costs provides the basis for the ongoing control of cost of the CIP operation.

9.8.2 *Product recovery*

The nature of CIP operations is such that a quantity of product will be lost as deposits on heat exchanger surfaces and as scale. This element of product wastage is unavoidable, but there are several instances where product remains intact in pipelines and equipment at the end of a production run and before a CIP programme starts. Effective recovery of this residual product can make a difference between achieving a site product wastage level of a good practice figure of 0.7%, and a poor one of up to 4% (Pankakoski, 1990).

Product reclaim

Silos and tanks

The time taken for milk, and particularly more viscous products such as cream and yoghurt, to drain from vertical surfaces can be quite significant, so it is important to allow for sufficient drainage time after tanks have emptied. Silos and tanks should therefore be set up to drain individually, usually by using a low-level probe as a trip, and then allowing a time lag to allow drainage below the probe, from the tank walls, and from associated outlet pipework. Switching between tanks can result in product loss if valves are not closed in the right sequence, allowing product to backfill from a subsequent tank. The frequency of tank cleaning should also be minimised to reduce product loss, consistent with achieving required hygiene standards.

Pipelines

Recovery of product from pipelines is often more difficult because they are often interlinked, and not all available for reclaim at the same time. However, product recovery from lines is far easier when lines are designed to slope and self-drain.

Reclaim tanks

If product is to be reclaimed for reuse, then the hygienic design and operation of reclaim pipework and tanks is vital. It is also important to ensure that sufficient residual space is available in reclaim tanks, since there is no point in going to the trouble of a reclaim process if the reclaim tank is full.

Product purging

Even when arrangements have been made for product reclaim, there will still be residual product left in the plant and pipelines. The most common means of purging product for possible reclaim is water purging. However, in a small number of cases air purging has proved an option with small-diameter lines; in larger diameters the air tends to tunnel through the product and simply go through the middle. Purging with 'pigs' has also been successful with more viscous products such as cream and yoghurt. In this case, a solid piece (known as the pig) is pushed through the line to force residual product out. Pigging has limited use because of the constraints of plant items such as valves and bends, and many pigging installations have resulted in hygiene problems.

Maximising product recovery in the water purging process requires achieving a sharp interface between the product being purged and the water, so that the product recovered is not over-diluted (Pankakoski, 1990). Traditionally, this has been achieved using sensors, such as conductivity and turbidity meters, to detect the interface and initiate the appropriate valve changes. However, these are not universal solutions, since conductivity is not very sensitive to products containing significant amounts of milk fat, and turbidity sensors tend to get fouled and give false readings. Nevertheless, a good approach to water purging is to meter the water being used for the purge. In this way, the exact volume of water needed to give the sharpest interface can be delivered every time.

Product scheduling

There is no doubt that product recovery can be improved by optimising the order in which products are processed. This can be achieved by minimising product changeovers, and by minimising changeover times between products. For example, switching from skimmed through semi-skimmed to whole milk can be achieved faster than the reverse sequence.

9.9 CIP management review and improvement

9.9.1 *CIP review*

The nature of CIP routes is that they will certainly change quite often as new products and/or processes or capacities are added to the capability of the system. In addition to the incremental changes, there will be changes when a totally new process and associated CIP are added to an existing system. Such changes require a review of the CIP operation at two levels, as follows.

CIP change review

This level of review is appropriate where an incremental change has been made to the CIP system. This verification process should review the following:

- the nature of the change and the reason for it;
- any change to clean durations;
- any changes to flowrates through the system;
- water clarity at end of pre-rinse;
- detergent changes;
- any changes to temperature of detergent;
- any change to concentration of detergent;
- visual inspection, where possible;
- ATP assessment of items and final rinses;
- hygienic assessment of product.

CIP verification

This level of review is appropriate when a significant change has been made to the CIP system, and the whole basis of the effectiveness of the CIP system is under question. This review should also be undertaken on existing systems on an approximately annual basis. However, this level of verification is more comprehensive than that set out for the CIP change review, and comprises three sets of checks: before the CIP is running, during the CIP operation itself, and after the CIP has finished.

Checks before the CIP operation are:

- walking the route to ensure the whole route can be traced;
- checking the route for poor design, e.g. deadlegs, changes in pipework diameters;
- ensuring that all product/CIP interfaces are separated;
- checking that all valves are identified and tagged;
- checking all spray devices and in-line sieves for integrity;
- checking the contents of CIP tanks for signs of product contamination;
- checking that probes and instruments are tagged and on the site calibration schedule;
- checking line diagrams to ensure that details of tanks and routes are up to date;
- checking that all cleaning parameters are being recorded; and
- checking the chemicals in use and verifying that they are suitable and approved.

Checks during the CIP operation are:

- pre-rinse (i.e. checking the route for leaks, recording and verifying the flowrate, and checking that rinse water going to drain is clear of product);

- detergent circulation (e.g. recording time above temperature and concentration set points, recording temperature from probe in return line, recording detergent concentration in return line, and recording flowrate);
- post detergent rinse (taking sample at end of rinse from return leg and test to ensure no residual detergent or product, and testing rinse for hygienic status);
- disinfectant rinse (e.g. taking a sample in middle of recirculation and analysing, checking time of disinfectant recirculation and, if thermal disinfection, checking time that temperature is above minimum set point).

Checks after the CIP operations are:

- recording the length of the clean;
- recording the number and reason of any CIP alarms – investigate and remediate;
- visually inspecting cleaned items – tank interiors, split lines, swabbing of cleaned surfaces;
- reviewing the software record of the clean.

9.9.2 *CIP improvement*

There are several areas where the effectiveness of CIP operations can continue to improve over current performance, which can be summarised as follows:

- reuse of water and chemicals;
- optimising CIP programmes to ensure that the required cleaning standard is achieved with minimum inputs;
- efficiency of spray devices;
- efficiency of product removal/recovery before CIP;
- efficiency of interface management.

It is important for dairy operators to keep aware of developments in these areas, to ensure that CIP performance improves in line with technology.

Improvement in CIP operation can also come about through the application of new sets of management techniques, such as those found in lean manufacturing and six sigma programmes (George, 2003). Such approaches to improvement have no specific relation to CIP itself, but can result in delighting customers with speed and quality, improving processes, ensuring decisions are based on data and facts, and working together for maximum gain. All of these can benefit the effectiveness of the CIP operation.

9.10 Conclusions

This chapter has set out some of the practical challenges associated with the operation of CIP processes.

As in most fields of activity, planning and preparation for CIP operation are key in delivering a successful outcome. Similarly, effective CIP operation requires input from many different disciplines in design, implementation and operation. The successful management of CIP operation is very largely dependent on the appropriate responsibilities and accountabilities being assigned between technical, operational and engineering personnel.

Once established, CIP operations must be managed in accordance with the important organisational goals of achieving quality, safety and productivity. Because it is inevitable that changes will be made to process pipework, valves, control systems, etc., it is vital that a CIP change review is carried out on a regular basis to ensure that the CIP operation is still delivering against its original objectives.

Where significant changes have been made to a CIP operation, then a more fundamental review is required to ensure ongoing effectiveness. This is achieved through the CIP verification process, which can also be used as the basis for auditing a CIP operation from a third-party perspective.

Finally, there are always opportunities for improving the quality, safety and productivity of the CIP operation, and it is important that these are reviewed and captured on a regular basis.

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10 Membrane Filtration

C.E. Askew, S. te Poele and F. Skou

10.1 Introduction

Membrane filtration installations are used to separate an initial liquid product feed stream into one or two separate streams, which are known as *permeate* and *retentate* (the latter is sometimes called the *concentrate*). This separation is based mainly upon a pressure-driven process, although there are other types of membrane filtration installation, electro-dialysis systems for example, which are driven by voltage and will not be discussed in this chapter. This chapter contains limited references to the literature, but the bibliography indicates useful sources for those wishing for further reading on membrane filtration.

Separation or filtration is carried out by passing the feed stream through equipment comprising numerous membrane filter elements configured in loops/modules, with the trans-membrane pressure (TMP) across the membrane forcing the smallest molecules in the feed into the permeate side of the membrane. A very rough analogy would be to compare it to the production of filtered coffee, where you have the liquid coffee as permeate, and the coffee grounds as retentate. However, retentate in membrane filtration processes is still a liquid, but with a higher total solids level than when it entered as the feedstock.

As the filtration processing continues, the membrane and pores gradually become filled with the feed components that are being retained, and consequently production efficiency begins to decline. Therefore the membranes will need to be cleaned to remove this fouling (soiling) in order to restore production capacity.

10.2 Membrane filtration processes

Depending on the membrane separation properties (structure and material), components can be rejected by the membrane, which is a perm-selective barrier (te Poele, 2005). When particles of diameter >100 nm have to be retained, the membrane process is called *microfiltration*. To separate macromolecules with molecular weights in the range 10^4–10^6 *ultrafiltration* membranes are used. Using *nanofiltration*, low-molecular-weight components and divalent ions can be separated; furthermore, monovalent ions can be rejected by reverse osmosis. Going from microfiltration to ultrafiltration, nanofiltration and reverse osmosis, the hydrodynamic pressure increases, and consequently higher pressure differences are needed (Mulder, 1996). Typical values of pore sizes and applied pressures, extracted from Doyen (2003), are presented in Table 10.1.

Figure 10.1 illustrates the use of membrane filtration systems in the food industry and other industrial applications, and Figures 10.2 and 10.3 show the pressure applied (bar),

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Table 10.1 Membrane filtration processes.

Membrane process	Pore size	Pressure range (MP[a])
Microfiltration	0.1–20 µm	0.01–0.3
Ultrafiltration	2–100 nm	0.02–0.8
Nanofiltration	< 2 nm	0.5–2
Reverse osmosis	Dense[a]	1–10

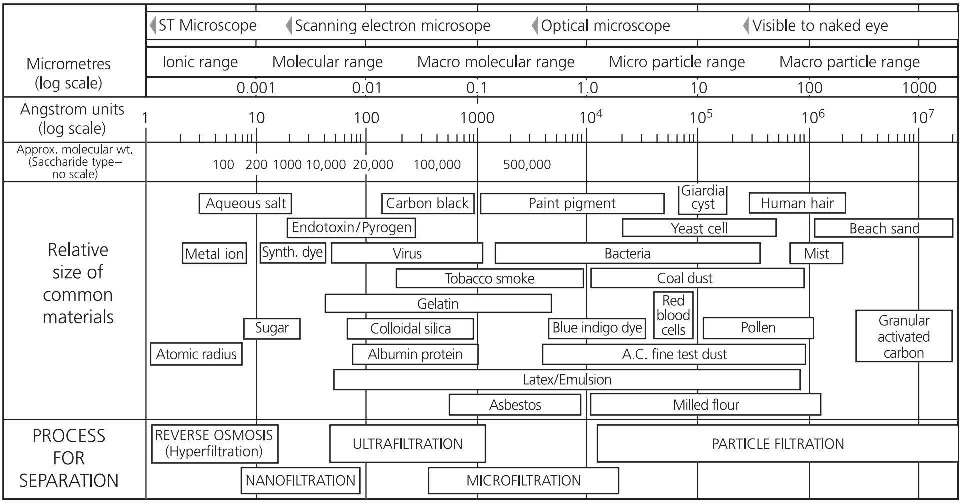
[a]Membrane structure is so dense that no pores can be distinguished.
Source: After Doyen (2003).

membrane pore size (µm), and cut-off (i.e. based on the molecular weight of the smallest molecule that will not pass through the membrane) for the different membrane filtration systems that may be used during the processing of milk and milk-related products.

10.3 Membrane process design

In general, for membrane process design, the following aspects should be considered (te Poele, 2005):

- choice of membrane material
- membrane module
- mode of operation



Note: 1 Micron (1×10^{-4} metres) = 4×10^{-1} inches (0.00004 inches)
1 Angstrom unit = 10^{-10} metres = 10^{-4} micrometres (microns)

Fig. 10.1 Spectrum of membrane filtration in the food and other industries. Reproduced by permission of GE Water & Process Technologies, Morgantown, USA.

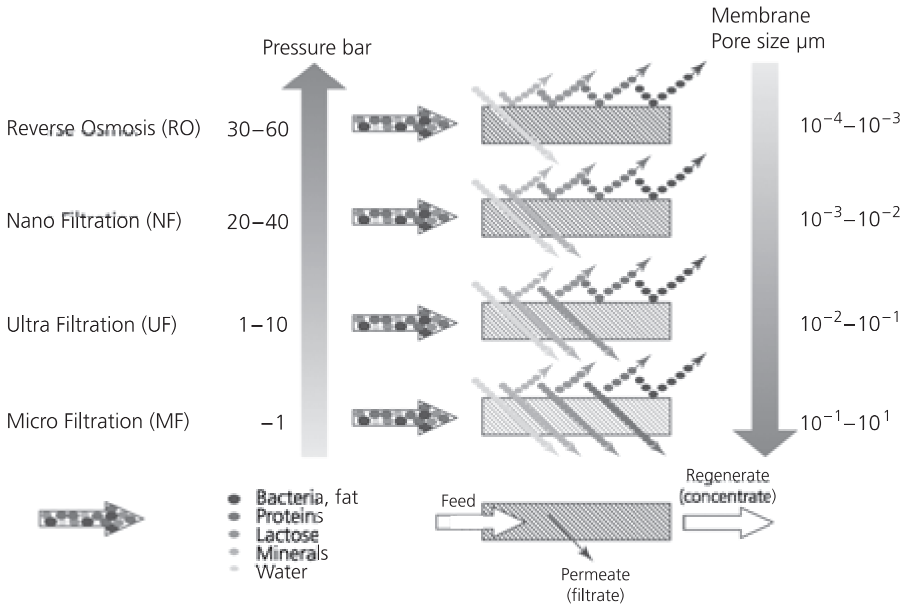


Fig. 10.2 Operational parameters for membrane filtration systems used in the dairy industry. Reproduced by permission of Tetra Pak A/B, Lund, Sweden.

Inorganic materials, such as ceramics, have a high chemical and temperature resistance, but are much more expensive than polymeric materials; more detailed information will be given in Section 10.3.1.

The average lifetime for a new membrane is approximately 2 years, provided the operating parameters specified by the membrane supplier have not been exceeded (see Section 10.5). The total cost of operation for a membrane installation includes energy, water, chemicals, down-time (when plant is not in production – normally used for cleaning purposes) and maintenance (replacement of membrane elements).

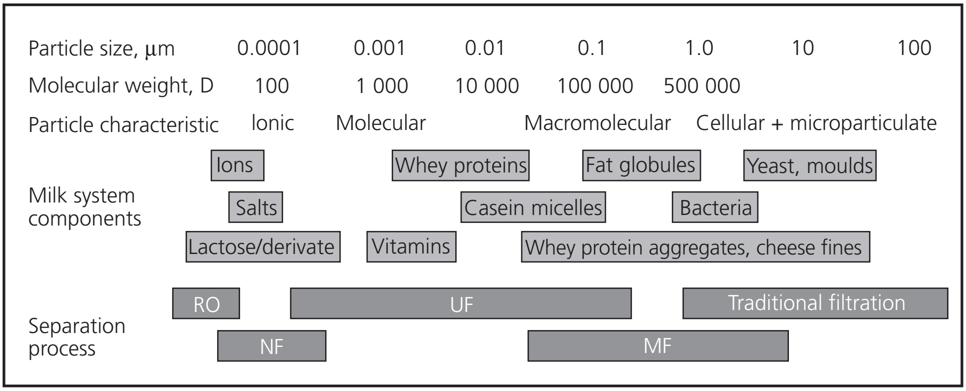


Fig. 10.3 Spectrum of application of membrane separation processes in the dairy industry. Reproduced by permission of Tetra Pak A/B, Lund, Sweden.

10.3.1 *Membrane material*

There are many different types of material used to make membrane filters; some types of membrane are made from one material alone, whereas many other types are composed of different materials. The main membrane chemical types are:

- acrylonitrile (AN);
- cellulose acetate (CA);
- ceramic, coated with either zirconium (Zr), aluminium (Al), or titanium oxide (TiO_2);
- polysulphone (PS);
- polysulphone with a thin polyamide coating (thin film composite – TFC);
- polyethersulphone (PES);
- polyvinyldifluoride (PVDF);
- polypropylene (PP); and
- polytetrafluorethylene (PTFE).

It is important for both equipment manufactures and dairy/food processors that they use the correct type of membrane, which will be dependent upon the type of installation and product (feedstock) being processed; these considerations will be discussed in Section 10.6.2.

All these classes of membrane material exhibit different tolerances to both chemical and physical parameters, such as pH, temperature and detergents (e.g. chlorine and surfactants). These characteristics are very important when it comes to choosing the chemical cleaning regime in order to remove all membrane fouling (soiling) that has built up during production. In addition, if incorrect detergents are used, then costly irreversible damage to the membranes may be the possible result.

10.3.2 *Membrane module design*

Membranes are manufactured to different configurations depending on the type and density of the membrane material in the module. The most frequently used configurations are:

- plate and frame
- spirally wound
- tubular
- hollow fibre

The first two configurations are more suited to high-fouling food processing applications, whereas spirally wound and hollow fibre systems are distinctly different designs permitting much higher packing density of membrane per module.

In *plate-and-frame* systems the feed flow passes up through the ‘open channel’ between the plates mounted in the compressed frame. As the feed passes across the membrane surface, low-molecular-weight components pass through the membrane, leaving the module as permeate. High-molecular-weight components, which do not pass through the membrane, leave the module as retentate. The packing density of such modules is about 100–400 m^2 m^{-3}.

In order to increase the packing density, *spiral-wound* modules were developed: to all intents and purposes these are a plate-and-frame system wrapped around a central collection pipe. Modules are created from flat sheets of membrane glued back to back on three sides, forming an envelope around a porous support material. Spacer material is required on both sides, i.e. feed and permeate, to enable flow to take place in both of these channels. The polyethylene mesh spacer also promotes turbulence within the feed channels. The open end of the membrane envelope is attached around a perforated tube, which provides a route for permeate to flow out. The membrane is rolled up around the centre tube, forming a cylindrical element. These membrane modules are designed primarily for use in cross-flow installations, with the feed running parallel to the membrane surface.

In this case, the feed flow enters at one face of the spiral element, and passes longitudinally through the element along the feed channel created by the feed spacer lying between the membranes. As the feed passes across the membrane surface, low-molecular-weight components pass through the membrane and flow in a spiral, rotating to the centre of the element and leaving the module through the centre collecting tube as permeate. High-molecular-weight components, which do not pass through the membrane, continue the longitudinal flow via the 'feed spacer', leaving at the other end of the spiral element as retentate. The packing density of this module is 300–1000 $m^2\ m^{-3}$. Because of the hydraulic design of such modules, many 'dead' areas are obtained, which are hard to clean. Figure 10.4 shows a schematic illustration of a spiral-wound element configuration. An excellent animation illustrating the flows through both plate and frame and spiral-wound elements can be viewed on the Alfa Laval website (www.alfalaval.com).

Tubular membranes are not self-supporting: therefore they are placed inside a rigid porous tube. Here the feed solution always enters through the centre of the tube, with the permeate flowing through the supporting tube into the membrane housing. The packing density of a tubular module is rather low: < 300 $m^2\ m^{-3}$.

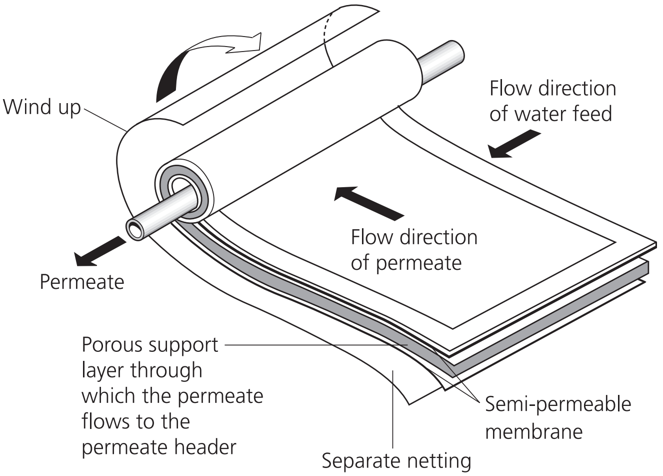


Fig. 10.4 Schematic illustration of a spiral-wound membrane element configuration. Reproduced by permission of Micro-Membrane Systems Ltd, Bath, UK.

Hollow fibre modules have good process control and membrane cleaning conditions, and are therefore suitable for feed streams with a high fouling tendency. The free ends of the fibres are bonded together with agents such as epoxy resins or polyurethanes to provide an absolute barrier between the feed side and filtrate side, and are then assembled together as a module to be inserted into the housing. The feed can be applied to either flow through the lumen of the fibres, i.e. inside-out, or enter the fibre from the outside, i.e. outside-in, according to individual manufacturers' designs. A disadvantage of outside-in filtration systems is that channelling may occur. The difference between capillary and hollow fibre modules lies in the values of the packing density, which are about 600–1200 $m^2\ m^{-3}$ and 30 000 $m^2\ m^{-3}$ respectively. Hollow fibre modules are often used when the feed stream is relatively clean. Figure 10.5(a) shows a membrane in cross-flow with feed to the inside of the lumen; the permeate collection is from the top port with the bottom port valved off. Figure 10.5(b) shows a membrane in cross-flow, with the feed to the outside of the lumen and permeate collection taking place from the top and bottom of the membrane module. The bottom (outlet) port for the retentate out is through a throttling valve that will be partially closed, to exert back-pressure in the system; otherwise there will be no driving force for the membrane to permeate.

10.3.3 *Methods of operation*

There are two modes of operation: dead-end and cross-flow filtration.

Traditional or conventional filtration processes are also called *dead-end filtration*. This type of filtration is normally used for separation of suspended particles larger than 10 μm, whereas membrane filtration separates substances of molecular sizes less than 10 μm. Dead-end filtration is a mode of operation in which there is only one feed stream (i.e. the retentate) and one outlet stream (the filtrate or permeate).

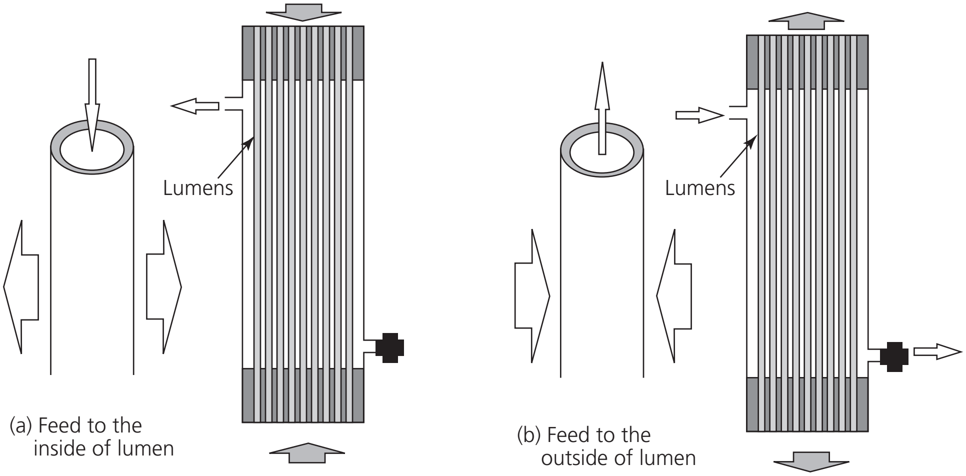


Fig. 10.5 Schematic illustration of hollow fibre membrane feed variation configurations. Reproduced by permission of Micro-Membrane Systems Ltd, Bath, UK.

Cross-flow filtration is defined as the flow of fluid across a membrane and parallel to its surface, which inhibits the formation of deposits. This system of filtration has one entry port (for the feed) and two outlet ports (one for the retentate and the other for the permeate).

In membrane filtration the use of pressure is essential as the driving force for separation, and a cross-flow or tangential flow design is followed. The feed solution runs parallel to the membrane surface, and the permeate flows through the filtration membrane. Filtration must be carried out in an enclosed system. The pressure put on the membranes (also known as *modules*) forces the retentate to be concentrated, while the permeate is forced through the pores of the membrane into the permeate side or outlet. The concentration factor defines the reduction in the feed volume of the processed material(s).

Filtration may be carried out as either a single batch or a continuous production process. The *single batch process* continually concentrates the same feed product until the required level is achieved, and is more commonly used in laboratories and pilot plants for small batches.

The *continuous production process* is the most widely used method for membrane processing. Basically, the membrane filtration plant consists of a series of single-batch processes linked together to form one continuous process. This provides customers with a constant feed product that can be processed for up to 20 h without stopping (depending upon the product feedstock and the efficiency of the installation) in order to remove retentate soiling from the membrane (i.e. cleaning).

Although each of the various types of installation has been explained as a separate entity, in many cases one or more membrane processes work together in order to progressively concentrate different products, as shown in Figure 10.6.

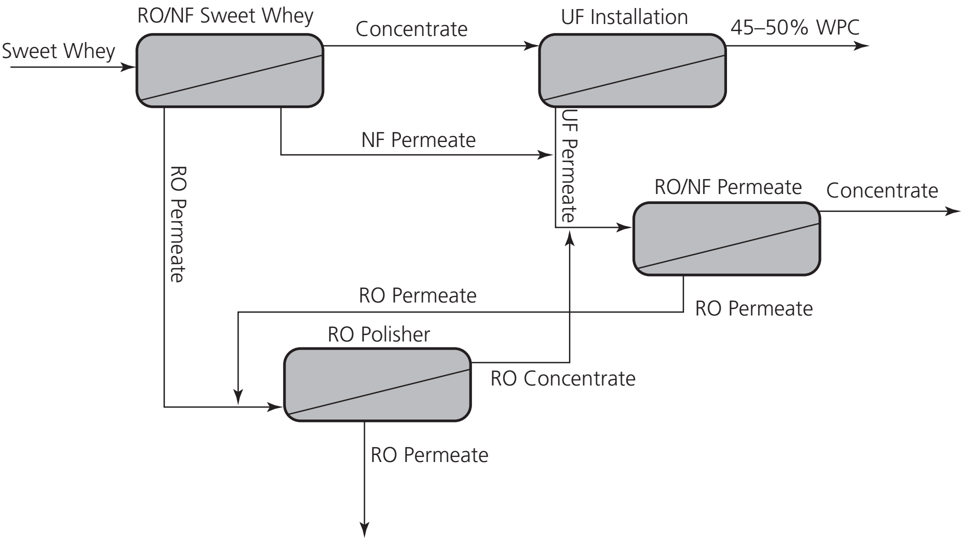


Fig. 10.6 Schematic of a membrane processing system for concentrating dairy components in whey. Reproduced by permission of JohnsonDiversey Ltd, Northampton, UK.

10.4 Membrane filtration in dairies

There are numerous different types of membrane filtration installation available, but the installations described in this section, which probably represent 90–95% of the membrane installation market, are of most interest to the dairy sector, as represented by Figure 10.7, which shows a typical on-site installation (Cheryan, 1998; Nielsen, 2000; Anonymous, 2003).

10.4.1 *Microfiltration (MF)*

The membranes in these installations are usually made from a ceramic material as the base, with an α-alumina coating creating the pores, which are tolerant of high pH (13) and temperature (80°C). Ceramic MF plants are typically tubular flow in configuration. Polymer types (see Section 10.3.1) are used in tubular, hollow fibre, plate and frame and spiral-wound membrane installations.

MF membranes are capable of retaining particles of about 0.01 μm or larger as the retentate or concentrate. Smaller particles (e.g. salts, sugars and proteins) pass through the membrane as permeate. Operating pressure for microfiltration is usually 0.01–0.3 MPa (0.1–3.0 bar). The most common applications for MF installations are:

- removal of bacteria from milk for nitrate-free cheeses (cold sterilisation);
- removal of bacteria from milk (extended shelf-life (ESL) milk);
- removal of bacteria from whey (cold sterilisation);
- removal of bacteria from brine (cold sterilisation);

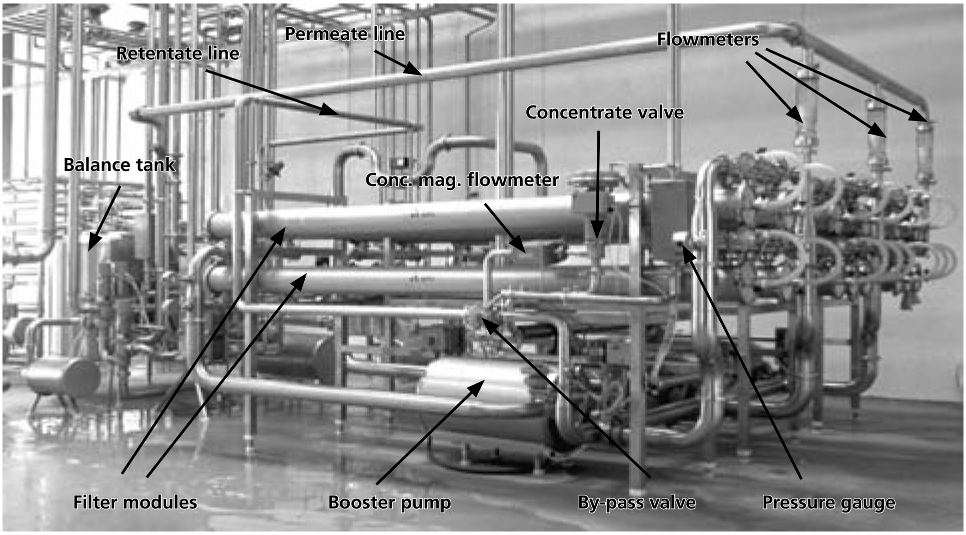


Fig. 10.7 A spiral-wound UF membrane filtration system. Reproduced by permission of APV Invensys, Silkeborg, Denmark.

- removal of fat from whey intended for whey protein concentrate (WPC) and for protein fractionation;
- fractionation of proteins;
- cold sterilisation of fruit juices and cider; and
- cold sterilisation of beer.

10.4.2 *Ultrafiltration (UF)*

These membranes are generally spiral-wound polysulphone/polyethersulphone, and are not as chemically tolerant as the MF membranes. However, they are still able to withstand chlorine and pH 11.5 cleaning regimes, but at lower temperatures than those used for MF; however, high-temperature-stable polyethersulphone UF membranes, which can be cleaned at pH 13.0 and 75°C, are becoming more common. Other UF plant configurations are hollow fibre, plate and frame or tubular flow, each of which has its own temperature and pH tolerances.

The UF membranes are capable of retaining particles of about 0.005 μm (i.e. molecular weight (MW) >1000 dalton (Da)) or larger as the retentate or concentrate. Low-MW substances (e.g. salts, sugars) pass through the membrane as permeate, and the operating pressure for UF is usually 0.05–1.0 MPa (0.5–10.0 bar). The most common applications for UF installations are:

- concentration of the proteins in milk or in acid and sweet whey;
- protein standardisation of milk for cheese making;
- protein standardisation of milk for yoghurt manufacture; and
- clarification of antibiotics in the pharmaceutical industry.

10.4.3 *Diafiltration (DF)*

This is an additional facility for UF installations, which does not occur with other pressure-driven filtration applications. This filtration method is a modification to a UF installation, where water is added to the 'feed product' (i.e. retentate) of each loop during filtration. This helps to progressively wash out increasing amounts of the milk components (e.g. salts and lactose) from each subsequent loop, resulting in a 'purer' retentate. Some examples include:

- removal of lactose and calcium phosphate from whey products;
- removal of minerals from other products, such as milk; and
- removal of cephalosporin from fermentation broth.

10.4.4 *Nanofiltration (NF)*

The membranes used are generally spiral-wound thin film composite (TFC) of polyamide-coated polyethersulphone with a smaller pore size than UF membranes. NF membranes are less resistant to chemical attack, with pH tolerances of typically 10.0–11.5. Basically, they are the same as reverse osmosis (RO) TFC membranes, but they have a larger pore size (0.0001–0.001 μm).

Depending upon the pore size of membrane being used, NF either retains divalent particles (e.g. $MgSO_4$) as the ‘retentate’, or allows them to pass through into the permeate. Water, monovalent ions and low-molecular-weight organic compounds (e.g. < 250 Da) pass through the membrane as permeate. The operating pressure for NF is usually 0.8–4.0 MPa (8–40 bar). The most common applications for NF installations are:

- concentration of the proteins in milk or in acid and sweet whey;
- concentration of UF permeate;
- partial desalination of UF permeate; and
- caustic detergent recovery from waste cleaning-in-place (CIP) solutions.

10.4.5 *Reverse osmosis (RO)*

As in NF installations, RO membranes are made predominantly of polyamide-coated polysulphone (pH 11.5 tolerant). There are also other membrane types, for example cellulose acetate (mainly plate and frame installations), whose pH tolerance is only 7.5–8.5. The low pH and temperature recommendations for cleaning this type of membrane make it among the most difficult category to clean, because higher pH levels will hydrolyse the cellulose acetate bonds. Currently, there are high pH tolerant polyethersulphone RO membranes available on the market, for which more active chemicals can be used to clean them more efficiently.

RO membranes allow only water to pass through them as permeate. Salts and dissolved organic substances with a higher MW of 50 Da are kept back as retentate, almost without exception. The operating pressure used for RO usually varies between 2 and 6 MPa (20–60 bar), and in some cases can be over 8 MPa (80 bar). The most common applications for RO installations are:

- dewatering or concentrating of whey;
- dewatering or concentrating of UF permeate;
- dewatering or concentrating of whole milk;
- concentration of white water rinses (white water is the thin milky solution in the separation phase between milk and water prior to CIP);
- concentration of wine or vinegar;
- production of pure water from condensate;
- production of drinking water from underground and seawater; and
- de-alcoholisation of beer (i.e. production of low-alcohol beers).

Figure 10.8 shows how the different milk components could be fractionated or concentrated using the various membrane filtration systems considered in this section.

10.5 Damage that can occur to membranes

As stated elsewhere, it is very important to appreciate and understand that most membranes are sensitive to both chemical and temperature damage. Membranes may also be damaged by adverse mechanical stresses, and the following examples (the list is not comprehensive), which are not in any order of preference, illustrate when damage to membranes could be experienced:

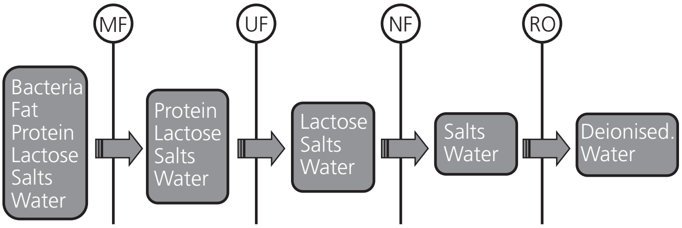


Fig. 10.8 Fractionation of milk components using different membrane filtration systems. Reproduced by permission of JohnsonDiversey Ltd, Northampton, UK.

- The use of phosphoric acid on ceramic membranes, as the phosphate ion can replace the aluminium oxide and make the membrane brittle.
- The use of chlorine on non-chlorine-stable membranes, as the active chlorine oxidises the polyamide layer.
- The use of high-caustic detergents on cellulose acetate (CA) membranes, as high causticity destroys the cellulose paper layer.
- Excessively high cleaning temperatures will distort the membrane, negatively altering its flow characteristics and performance.
- Changes in flow and pressure across the membrane surfaces can reduce permeability.
- Water used for cleaning not to the correct specification – for example, silicate, iron and other contaminants – can irreversibly damage/block membrane pores.
- If booster pump pressure is too high, it forces soil into the membrane pores, and if it exceeds the maximum recommended pressure drop across the membranes, can lead to telescoping and channelling of spiral-wound membranes.
- Leaking valves reduce rinse volume during the cleaning cycle, which results in low pressure, reduced permeability and poor cleaning.
- Continuous poor cleaning does not open the membrane pores, resulting in reduced production capacity, and cleaning efficiency drops over time.
- Excessively high temperatures and pH during cleaning and/or product processing will cause irreversible damage to the membranes resulting in costly replacement..
- Higher than recommended temperatures on RO membranes reduces production capacity, as the membrane becomes tighter.
- Higher than recommended pH can destroy the paper layer, resulting in the collapse of the membrane.

Figure 10.9 shows a micrograph of a UF-membrane installation used for concentrating milk, which developed blisters caused by the use of excessive heat.

To avoid costly mistakes always check the detergent product(s) information sheet(s) to ensure their compatibility with the specific type of membrane being cleaned.

When designing the cleaning product, surfactants are chosen to provide the highest production capacity on the membrane plant after cleaning. Surfactants that give the highest capacity on UF-polysulphone membranes, for example, might not be suitable for NF and RO membranes. The TFC polyamide coating, due to possible stress cracking of the polymeric membrane layer or an irreversible change in permeation, may result from using a non-suitable product.

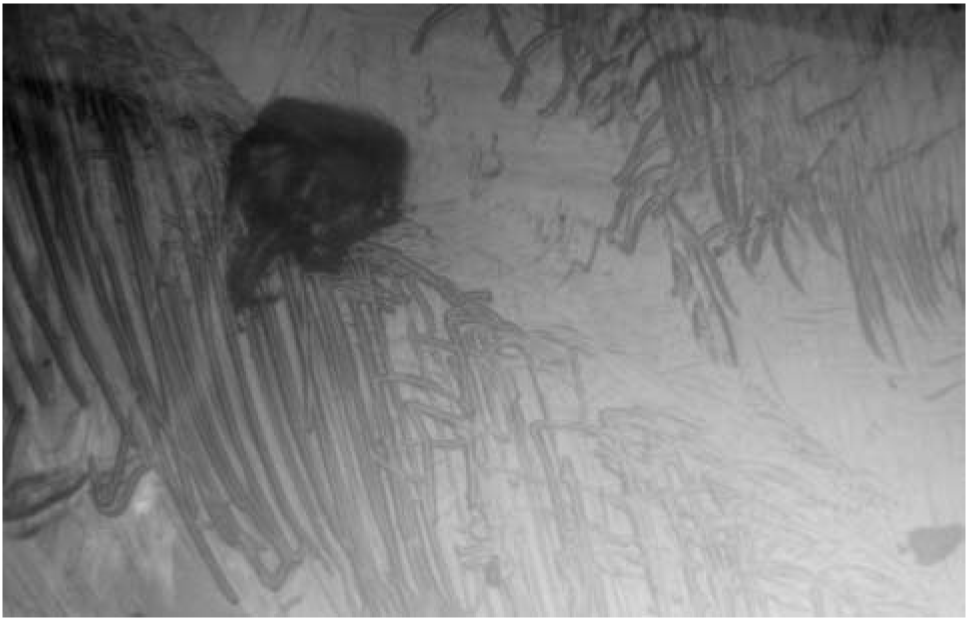


Fig. 10.9 Micrograph showing heat blisters on a UF membrane. Reproduced by permission of JohnsonDiversey Ltd, Northampton, UK.

10.6 How do membranes become fouled or soiled?

Membrane installations are designed to process specified products at correct flowrates, pressures, temperatures and times, with the correct types of membrane. The correct/recommended pressure drop across the membranes must be maintained at all times to enable the installation to concentrate the product efficiently with the correct permeation rate through the membranes. If the applied pressure and flowrate are not correctly balanced, major membrane fouling can occur very quickly.

10.6.1 *Concentration polarisation and membrane fouling*

During filtration, retained components accumulate at the membrane surface: as a consequence, the resistance to filtration will increase (te Poele, 2005). This phenomenon can be described in terms of concentration polarisation and membrane fouling. *Concentration fouling* occurs as the resulting concentration build-up generates a diffusive back-flow to the bulk of the feed solution. According to Song & Elimelech (1995), the accumulation of solutes includes colloids and particles, and concentration polarisation is inherent in all membrane processes. The phenomenon occurs rapidly, and is a reversible process. In addition *membrane fouling* occurs: this is defined as deposition of solutes inside the pores of the membrane or at the membrane surface. The fouling phenomenon takes place on a longer time scale, and can be either reversible or irreversible.

The different modes of fouling mechanism can be summarised as layer formation, pore blocking and adsorption, depending on the chemical and physical properties of the retained components. The concentration of the accumulated solutes may become so high that a gel layer can be formed, which exerts gel layer resistance. This happens mainly when the feed solution contains proteins (Mulder, 1996).

Membrane fouling can be classified into inorganic or mineral fouling, organic fouling, particles and biofouling, depending on the nature of the components involved (Flemming, 1995). Since most of the feed in dairy applications consists of organic matter, organic fouling is considered the major component of membrane soiling/fouling.

Particulate fouling is a persistent problem in all membrane filtration processes: it refers to the deposition of suspended matter, colloids and micro-organisms on the membrane surface. Scaling occurs mainly in reverse osmosis (RO) and nanofiltration (NF) applications: it refers to the deposition of hard scale on the membrane surface because the solubility of soluble salts has been exceeded (Boerlage, 2001).

Laîné *et al*. (2002) reviewed a few studies that have been reported on irreversible fouling due to inorganic compounds, such as aluminium, silica and iron, especially under specific conditions of concentration, temperature and pH. They also reported that precipitation of iron, manganese and carbonate has often been observed as the cause of membrane fouling, especially when an oxidant such as chlorine is applied during membrane cleaning. In addition, Boom & Borre (2001) found that oxidation of soluble manganese, Mn^{2+}, to solid manganese oxide, MnO_2, may occur when oxide is released by, for example, sodium hypochlorite (NaOCl), which is often used as a cleaning agent. The formed manganese oxide particles resulted in pore blocking of ultrafiltration membranes.

According to the ‘classical’ theory based on reversed osmosis (Flemming, 1995), biofouling is described as attachment of micro-organisms at the membrane surface, where they secrete slimy substances – the so-called *extracellular polysaccharide substances* (EPS). Furthermore, the micro-organisms are embedded within the EPS structure, which forms a three-dimensional tight matrix, i.e. a biofilm.

10.6.2 *Membrane fouling in dairy processing*

In addition, as with any manufacturing process in food and beverage applications, fouling of equipment surfaces and substrates is inevitable during and after processing the product has stopped. If a customer operates the installation for longer than the specified equipment manufacturer’s recommended processing times, for example, major fouling or even membrane damage could occur. Figure 10.10 represents a theoretical membrane system to illustrate flowrate mass balancing through a typical three-loop system.

Tanks and silos that are used to store products to be processed must be clean and disinfected before receiving product for membrane processing. Failure to meet these requirements can cause major microbiological problems in the membrane pores. For low pH tolerant membranes, it is hard to resolve some microbial problems.

For milk products, if the correct product specification is not followed prior to processing, major fouling problems can and will occur. For example, the age of stored milk products has to be exact, otherwise denaturing of milk proteins will occur in the installation, and have a similar effect as glue on membranes. However, if the correct temperatures are not maintained during processing and cleaning, damage to membranes will occur. This is especially the case if excessive temperature denatures the soil, which will then become very difficult to

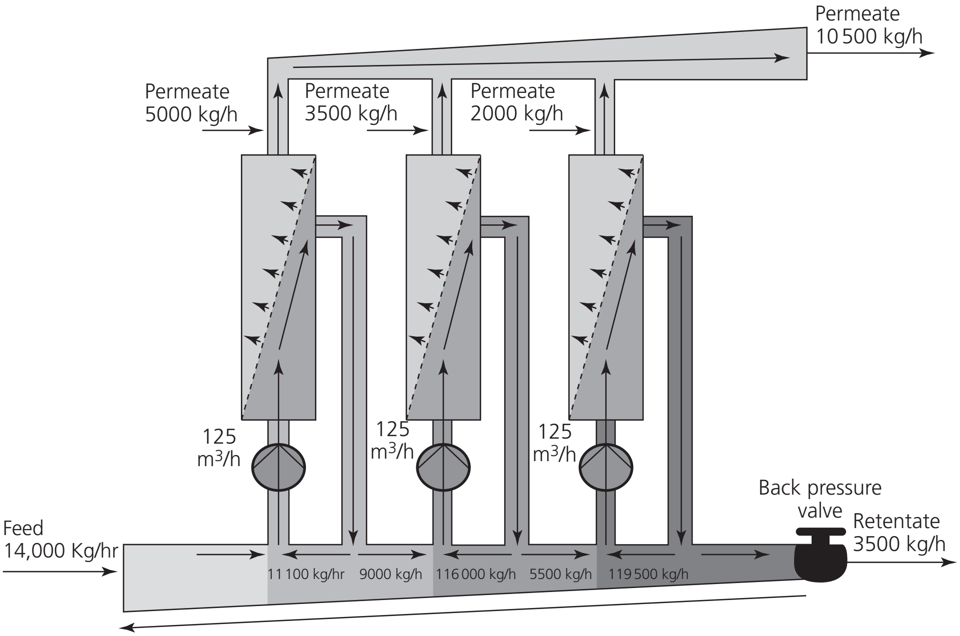


Fig. 10.10 Mass-balanced flowrates in a membrane installation. Reproduced by permission of DSS Silkeborg A/S, Silkeborg, Denmark.

remove from the membranes. Figure 10.11 illustrates an opened RO membrane, and shows whey protein that has been deposited in the pores.

10.6.3 *Fouling control*

Controlling the quality of the water used for cleaning is therefore an important requirement in eliminating potential colloidal and particulate fouling caused by silicates or iron, as this type of soiling is irreversible. It is recommended that feedwater for RO equipment must be maintained at an acceptable silt density index (SDI) level to ensure economic and efficient operation (ZyzaTech Water Systems, Inc., 1997)

The SDI test is used to determine the fouling potential of water feeding a membrane filtration process such as an RO system. This test is defined by its specific procedure (ASTM D-4189), which should be referenced for a more detailed description of the procedure. SDI tests on the raw supply water should be part of every feasibility study for an RO system, and it is recommended that a record of SDI values and filters be kept, to observe changes over time. It is also good practice to run an SDI test periodically to make sure no changes have occurred to the source water.

Measured values reflect the rate at which a 0.45 μm membrane filter will plug with particulate material when feed water is flowing through it. The ASTM chose the 0.45 μm filter because it is more likely to clog from colloidal matter than from hard particles such as sand or scale. SDI testing is commonly used as an ‘early alert’ to ensure that particulates in feed water do not plug the micropores in RO membranes.

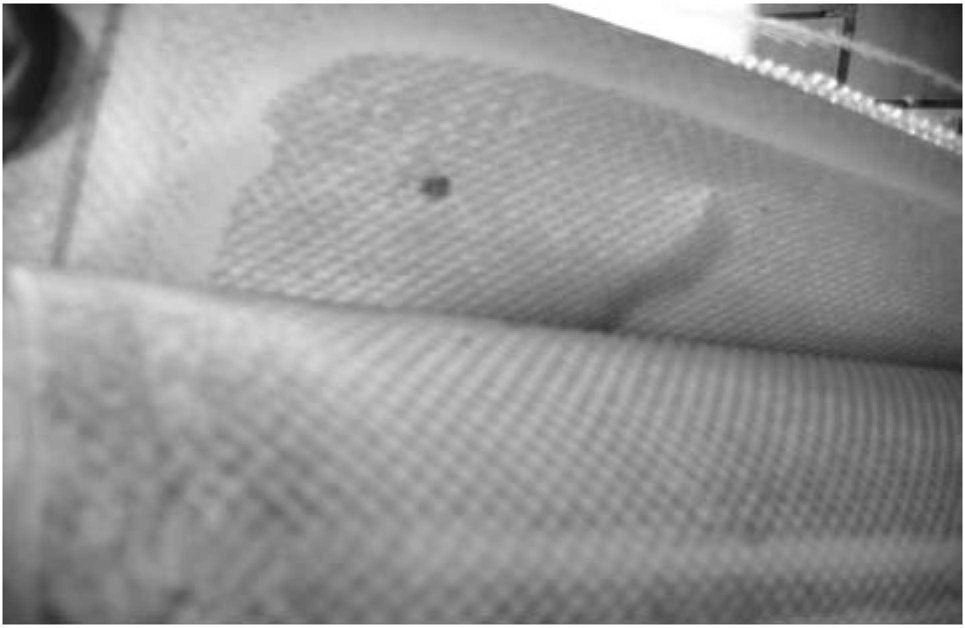


Fig. 10.11 An opened RO membrane element, showing whey protein deposits. Reproduced by permission of JohnsonDiversey Ltd, Northampton, UK.

Depending upon the type of fouling, it may be possible to change the cleaning programme to a milder procedure, which may have the added benefit of significantly extending the life of the membrane, e.g. replacing chlorine with enzymes for protein soiling on UF.

In many filtration systems, RO permeate, e.g. water, is a by-product, and is more often than not stored for reuse. This good-quality water should be used whenever possible for cleaning and rinsing all membrane filtration systems. Tanks used to store RO permeate (i.e. water) should be the subject of a regular cleaning and disinfection programme to eliminate any possibility of microbiological contamination occurring in the membrane elements.

10.7 Cleaning membrane filtration installations

10.7.1 *Background*

Stainless steel equipment is generally considered tolerant to almost any detergent or detergent disinfectant commonly used to remove the soil; however, there are limitations. For example, stainless steel can be damaged by peracetic acid (PAA) in water containing very high chloride levels when not rinsed out properly, resulting in pitting-type corrosion, which cannot be rectified. Stainless steel has a smooth surface, and it is relatively easy to remove surface soiling by using alkaline and/or acidic products.

Membranes, by contrast, have a porous surface, where soil can be easily deposited. Most types of membrane are less tolerant to chemicals, high temperature and pH than stainless steel, or other materials. Spiral-wound membranes have a ‘net’ structure, which, coupled with pH and temperature limitations, makes it much more difficult to clean than most other food and beverage applications. In addition, membrane materials are made from various types of polymer, which are prone to stress cracking if they come into contact with certain categories of surfactant. Also, surfactants can change the polarity of the membrane pores, which can modify the permeability of the membrane and alter the specification of the product being processed.

Furthermore, other components in the installation (spacers, support plates and glues) have their own chemical, pH and temperature tolerances, which need to be considered. It is important, therefore, to use only formulated products containing chemicals that are proven not to damage any materials of construction of the membrane system.

10.7.2 *Cleaning fouled membranes*

Membrane chemical cleaning is usually carried out in several steps, as follows.

- Back flush (BF) or forward flush (FF) is applied to remove the reversible fouling.
- Chemical cleaning solutions are introduced to the membrane surface by either BF or FF, when the membranes are often soaked for a defined time. To introduce mechanical energy, the cleaning solution can be pumped along the membrane surface, so that shear stress is introduced to the boundary layer of the membrane surface.
- The last step is to flush the membranes with permeate, mains water or ultra-pure water to remove residues of fouling and detergent.

The global parameters that influence the cleaning performance are:

- chemical reaction
- contact time
- temperature
- mechanical energy
- foulants, i.e. amount and type
- surface, i.e. type and roughness

The first four parameters can be changed, depending on the fouling present and the cleaning agent. Temperature is an important parameter, and its effect on chemical cleaning can be exponential. Most chemical cleaning is performed between 30°C and 50°C, depending on the specific membrane module limitations (te Poele, 2005).

The quality of the water that is used for chemical cleaning and, more specifically, its hardness are of importance for the concentration of cleaning agents. Soluble salts can disrupt the cleaning performance, and often a higher concentration is needed. Important such components are metal ions such as manganese, iron and aluminium. These components could very easily form oxides and hydroxides during alkaline cleaning. However, fouling of the membrane by pore blocking can be caused by the formation of metal oxides and hydroxide complexes (Boom, 2001), which can only be removed by acid cleaning. In the case of manganese hydroxide formation even acid cleaning is very difficult, because manganese can easily transform into other configurations of oxide and hydroxide complexes. The formed pyrolusite is extremely difficult to remove.

When cleaning membranes, there are many factors to take into consideration, some of which have already been discussed elsewhere, but the following is a comprehensive list summarising all the factors to be considered:

- product processed and soil type
- membrane installation
- membrane type
- level of membrane fouling
- pH and temperature sensitivity
- chlorine sensitivity
- equipment and membrane manufacturer's endorsements
- customer preferences
- local legislation
- environmental issues

Product processed and soil type

It is important to have knowledge of the product processed and understand its actual components in order to know which chemicals to use first to remove the easy soiling and also preventing components from depositing. The level of fouling is important. As an example, if a UF plant is processing whey to produce 80 g 100 g^{-1} WPC or 35 g 100 g^{-1} WPC in 20 h, the two production rates will generate different levels of fouling of the membrane, because the former product contains less salt: this is mainly because more salts are being washed out during DF (see section 10.4.3) of the whey for the higher production rate. This factor must be considered with other possible types of fouling that may occur (see Table 10.2).

Sometimes it may not have been possible to clean membranes properly, in which case it is important to carry out diagnostic testing of the membranes using electron microscopy to determine exactly what is blocking them, after which it should be possible to recommend a solution to remove the problem fouling. Another type of destructive test is scanning electron microscopy (SEM) analysis, which will even determine down to diatomaceous levels. This type of analysis should, however, be considered only as a last resort. Figure 10.12 shows a micrograph of a UF membrane installation used for filtering whey, which developed a growth of calcium phosphate crystals.

Membrane installation and type

It is important to know the type of membrane installation (MF, UF, NF or RO) to be cleaned, because each of them operates at a different pressure (see Section 10.3). Also, the design of the plant has to be considered: for example, is it a plate and frame, spiral-wound, hollow fibre, tubular flow or dead-end installation? Each of these plants uses different type(s) of membrane material (Table 10.3). Membrane filtration installations consist of many different components:

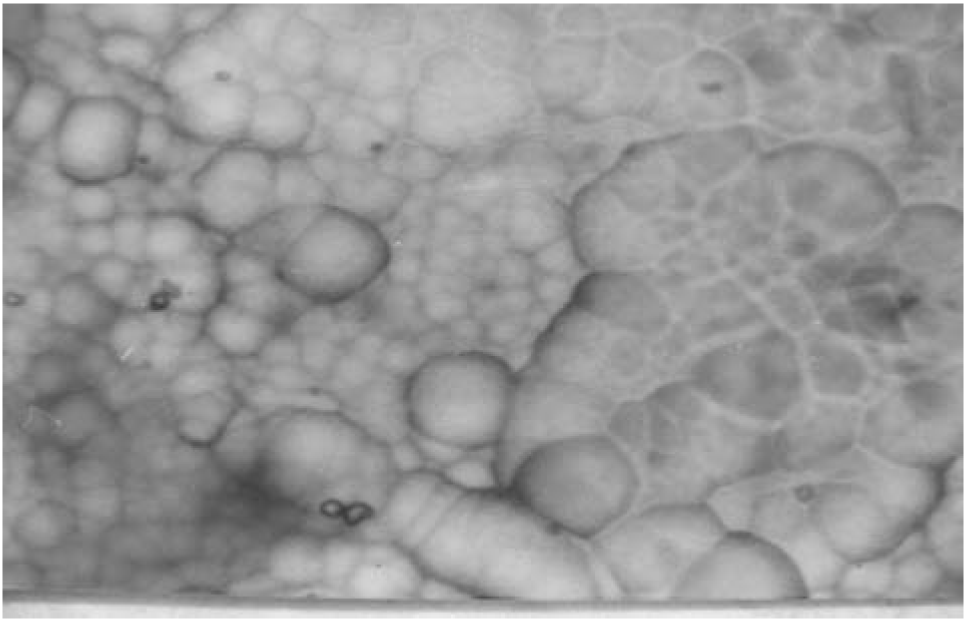


Fig. 10.12 Micrograph showing calcium phosphate deposits on a UF membrane. Reproduced by permission of JohnsonDiversey Ltd, Northampton, UK.

- the membrane material itself
- various types of membrane spacer material
- different materials for support plates
- different types of backing material
- different types of glue
- elastomer seals and O-rings

Table 10.2 Soil characteristics of membranes and recommended chemical for removal.

Soil component	Type of detergent
Fat	High causticity or medium causticity with surfactant
Protein	High causticity with surfactant or chlorinated alkalis, or medium causticity with enzymes and acid-based detergents
Lactose	Hot water, medium causticity or surfactant
Milkstone	High causticity with high EDTA content or nitric acid
Micro-organisms	High causticity, chlorinated alkalis or disinfectants
Casein defoamer	Surfactant additive compatible with the membrane

EDTA = Ethylenediaminetetra-acetic acid.

Table 10.3 Commonly used membrane materials, by plant design.

Membrane type[a]	Spiral wound	Plate and frame	Ceramic	Tubular flow	Hollow fibre	Dead end
CA		×		×		×
PS	×	×		×	×	×
PES	×	×		×	×	
TFC	×	×		×		
Al/Zr/TiO_2			×			
RCA		×		×		
AN				×	×	
PVDF	×	×				×
Nylon						×

[a] For abbreviations, refer to text.

It has already been established that all membrane materials have different pH, temperature and chlorine stabilities. In addition, these components have their own chemical, pH and temperature tolerances, which must also be considered when choosing products for cleaning, to prevent damage. Manufacturers do not generally disclose the materials they use in glues, for example: therefore always ensure that the pH, temperature and chlorine tolerances, as stated by the manufacturer in the relevant membrane specification data sheets, are not exceeded.

Level of membrane fouling

The level of fouling will depend on the product being processed and on the production time, compared with the original design specification for the membrane installation.

For example, 35 g 100 g^{-1} WPC compared with 90 g 100 g^{-1} WPC, or whole milk processed to 40 g total solids 100 g^{-1}, where the fat levels present vary considerably, will make a significant contribution to the amount of soiling that has to be removed during subsequent cleaning to restore membrane capacity.

pH and temperature sensitivity

As stated previously (see Section 10.3), most membranes are sensitive to both chemistry and temperature. Because membrane manufacturers do not all use the same materials to manufacture a polysulphone membrane element, for example, the pH and temperature tolerances will vary from one manufacturer to another. Examples of typical variations in these tolerances for the materials of construction commonly utilised for permeate spacers in spiral-wound PS/PES and TFC membranes are shown in Tables 10.4 and 10.5 respectively. Always check the specification of your actual membrane supplier.

Table 10.4 Typical permeate spacer tolerances found in spiral-wound PS[a] and PES membranes.

Material of construction	pH	Temperature (°C)
Polyester spacer	2–11.5	0–55
Polyester spacer with chlorine	2–10.5	0–55
Modified polyester spacer	2–12.0	0–60
Polypropylene spacer	1–13.0	0–75

[a]For abbreviations, refer to text.

Table 10.5 Typical permeate spacer tolerances found in spiral-wound TFC[a] membranes.

Material of construction	pH	Temperature (°C)
Polyester spacer	2–11.5	0–55
Polypropylene spacer	1–12.5	0–65

[a]For abbreviations, refer to text.

Care is also needed if more than one manufacturer's elements are used in the same installation. In these circumstances the lowest tolerances must be implemented, to ensure that pH and temperature tolerances are not exceeded, in order to avoid irreversible membrane damage and costly replacement.

Chlorine sensitivity

The other major chemical that must be considered is chlorine, which will cause irreversible damage to non-chlorine-stable elements. This is especially important in the case of RO and NF membranes because, if chlorine were to be used inadvertently, it would remove the thin polyamide layer from a polysulphone membrane TFC membrane. In effect this will change, for example, the TFC of an RO to a UF membrane, because the pore size will be increased, allowing larger molecules, e.g. proteins, to pass into the permeate stream instead of remaining in the retentate.

Equipment and membrane manufacturer's endorsements

Membrane and equipment manufacturers naturally require guarantees that any cleaning products/procedures used will not damage their membranes. Generally, the most important issue is that the original equipment manufacturer (OEM) knows the product range, and is confident with the chemical supplier and can endorse the use of the suggested chemicals.

Chemicals that are endorsed by one manufacturer on a specific type of membrane may not be assumed to have endorsements from other suppliers, because individual companies tend to use different test protocols. It is not possible to compare the way various OEMs test cleaning chemicals, because each one has its own individual and confidential test protocol. In addition, a surfactant in a detergent may be especially suitable for use with one type of membrane. However, it may produce an adverse reaction when used on a different type of membrane. As previously discussed in Section 10.3, this may cause irreversible damage and costly replacement charges.

Customer preferences

In addition to requiring guarantees that the cleaning products they use will not damage their installations, customers expect a higher than normal level of trust and confidence in the cleaning and sanitisation products supplier. This is because membrane cleaning is regarded as being at the highest level of CIP: therefore knowledge and understanding of this technology are of paramount importance.

The reputation of the supplier – not only in the local marketplace, but as a global supplier – is also considered, especially when a duplicate or mirror membrane system is to be installed in another country. Customers need to have absolute confidence in their supplier's ability to provide not only chemicals but also on-site commissioning and technical support.

Generally, customers prefer to stay with the supplier/account manager with whom they have built and maintained a strong inter-business partnership.

Local legislation

Local authorities can either restrict or prohibit the use of certain chemicals because of potential effluent problems. Care must be taken to understand these restrictions when selecting the appropriate detergent for the type and level of membrane fouling to be removed.

Examples of restricted materials include ethylenediaminetetra-acetic acid (EDTA), chlorine, nitrilotriacetic acid (NTA), certain surfactants, phosphorus as phosphate or phosphonate, nitrates, and/or enzymes. The degree to which these chemicals (and any others) may or may not be used has been found to vary between different countries. For example, EDTA restrictions apply in Germany, Sweden and Switzerland, and NTA restrictions apply in Denmark, France, Italy and Turkey

Environmental issues

Food and drink is the largest industrial sector in the UK in terms of turnover. It uses large amounts of water, and can produce significant quantities of waste. Although its emissions are largely organic in nature, and mostly biodegradable, they can have an environmental impact (Anonymous, 2005).

Integrated pollution prevention and control (IPPC) applies an integrated environmental approach to the regulation of certain industrial activities. This means that emissions to air, water (including discharges to sewer) and land, plus a range of other environmental effects (e.g. waste minimisation, water use, releases associated with energy, effluent management and accident risk) must be considered together. It also means that regulators must set permit conditions so as to achieve a high level of protection for the environment as a whole (see *Integrated Pollution Prevention and Control (IPPC) – A Practical Guide*, 3rd edn (2004), Department for Environment, Food and Rural Affairs – DEFRA).

One main class of product of concern is ethylenediaminetetra-acetic acid (EDTA), which was considered not to be environmentally friendly because of its ability to complex and remobilise heavy metals; along with NTA it is considered carcinogenic in some EU countries. Risk assessment shows that, although EDTA does not biodegrade in sewage treatment works, and is hence regarded as non-biodegradable, there is extensive evidence of its removal from the environment both by photo-degradation, particularly in the presence of iron, and by slow aerobic degradation in soil. Studies have shown that this degradation is to substances that are known to biodegrade rapidly. A predicted no effect concentration (PNEC) for EDTA in the aquatic environment was calculated to be 2.2 mg L^{-1}. In most normal circumstances, such as household use and low-volume industrial use, this concentration will not be reached (EU, 2004).

Feasibility studies carried out with wastewater from a dairy plant have shown that activated sludge systems can be operated to remove EDTA under alkaline conditions (pH 7.5–8.0) – a prerequisite for practical applications in wastewater treatment plants, provided the sludge retention time (SRT) is more than approximately 15 days (van Ginkel *et al.*, 1997)

These restrictions imposed by individual countries (e.g. local authorities) and possibly by customers will determine the types of chemical ingredient that can and cannot be used for membrane cleaning.

10.7.3 *Cleaning agents*

In general, six different types of cleaning agent can be distinguished by their nature and mechanism (te Poele, 2005):

- *Acid*: Removes crystallised hardness salts, metal oxides and metal hydroxides.
- *Alkali*: Removes general organic fouling.
- *Active chlorine*: Acts as an active component to minimise organic fouling and inhibit the growth of micro-organisms.
- *Oxide*: This is an active component to diminish mainly organic fouling and inhibit the growth of micro-organisms, but can also be used to minimise other types of fouling. Oxides are the strongest cleaning agents, but are often harmful to membranes and installations.
- *Organic solvent*: Removes organics by solubility.
- *Enzyme*: Specific enzymes adsorb specific organic foulants, and their removal is established by the addition of alkali soap or additive. Temperature and pH are important parameters in enzymatic cleaning performance.

The actual choice will, to a large extent, be determined by the nature of the soiling component to be removed, as shown in Table 10.2. Often a combination of agents is required, used either as a mixture or, more commonly, in separate stages of the cleaning programme (see Section 10.7.4).

Enzymes are major contributors to clean industrial products and processes. They show a variety of advantages over chemicals, including specificity, high efficiency and compatibility with the environment (Schäfer *et al.*, 2002). These materials are naturally occurring compounds, found in all living organisms, that facilitate the breakdown of specific materials into smaller pieces. Enzymes act as biological catalysts, and usually catalyse only one specific type of chemical reaction.

Enzymes are often used in food processing operations to aid in speeding fermentation, ripening, coagulation and other processes. Most detergent enzymes are derived from microbes that are engineered to produce large quantities of a single enzyme. Proteolytic enzymes or proteases have found many applications in cleaning. They are used in neutral to alkaline products to clean heavy protein-based soils in the dairy and meat industries. Since they are not corrosive, enzymes are commonly used on delicate equipment, such as RO and UF membranes.

The use of specialist enzymes to remove protein soiling from membranes is fairly well established, and reduces the pH requirements during cleaning. It is important that the mechanism by which they work is fully understood so that the correct cleaning procedure can be utilised. Enzymes, in common with all living organisms including humans, need moisture, warmth and nutrients to survive, and there is an optimum range in which they flourish. When any one or all of these requirements are gradually reduced, enzymes' activity initially begins to slow down before stopping and finally, in most cases, becoming deactivated altogether. On the other hand, gradually increasing these needs initially raises enzymes' activity, before they begin to slow down before stopping, and finally once again becoming completely deactivated. For enzymes, the essential items to control in order for them to work most effectively on the soiling are pH between 9.5 and 10.5, and temperature between 45°C and 50°C. A specific type of enzyme is used to degrade protein by breaking the peptide bonds, creating amino acids, which will reduce the pH of the cleaning solution. It is essential that constant checks are conducted during this stage to maintain the optimum pH range throughout the 30–40 min of this step of the cleaning programme.

At the end of the enzymatic stage, and without any rinsing to drain taking place, it is an advantageous procedure to add acid into the system to reduce the pH to < 2, and circulate for an additional 15 min. This will ensure that any residual enzymes are completely inactivated, eliminating the possibility of any contamination in the finished product.

Following rinsing, a normal alkaline step is generally carried out before final rinsing and completing a water flux test.

10.7.4 *Cleaning regime*

Having taken all the above considerations into account, a cleaning protocol for the category of installation being used, the type of membrane and the product being processed may then be created. A representative example would be the cleaning of an RO plant with high temperature (65°C) and high pH (12.5) tolerant membranes, which are used to process skimmed milk. The following key factors would need to betaken into account:

- type of membrane filtration process, i.e. RO;
- type of membrane (i.e. spiral-wound polyamide with medium tolerance to pH 11.5 and temperature at 50°C – see also the membrane manufacturing data sheet, which should always be present in the membrane plant operating manual); and
- product (e.g. skimmed milk).

These factors would result in creating a typical standard cleaning protocol, as shown in Table 10.6.

Table 10.6 Recommended procedure for cleaning and disinfecting an RO plant.

Operation	Concentration (g 100 g^{-1})	Water/detergent	pH	Temperature (°C)	Circulation time (min)
Product		Flush water		Ambient	Until runs clear
Alkaline wash	2.0 0.1	Divos 116 VM19[a] + Divos ADD1 VM29[b]	12.5	60	30
Rinse		Water or RO permeate		Ambient	Until pH neutral
Acid wash	1.0	Divos 2 VM13[c]	< 2	60	20
Rinse		Water or RO permeate		Ambient	Until pH neutral
Alkaline wash	2.0 0.1	Divos 116 VM19 + Divos ADD1 VM29	12.5	60	30
Rinse		Water or RO permeate		Ambient	Until pH neutral
Membrane preservative[e]	0.15–0.3 0.1	Divos LS VM15[d] + Divos 2 VM13	2–3	20	Maintain levels until production recommences
Rinse[f]		Water or RO permeate		Ambient	Until pH neutral

[a] Medium-alkaline detergent for high tolerant NF and RO membranes.

[b] Detergent adjunct to alkaline Divos products for removal of fat, protein and other organic soiling from all membrane types and casein defoamer from UF and NF membranes.

[c] Mixed acid descaler for UF, NF and RO membranes.

[d] Liquid preservative used in conjunction with Divos 2 for UF, NF and RO membranes.

[e] Optional membrane preservative step for shut-down periods less than 3–4 months.

[f] Only required following the optional membrane preservative step.

Note: if a plant throughput is 1000 L h^{-1}, the amounts of detergents and disinfectants required are: Divos 116 VM19, 20 kg; Divos ADD1 VM29, 1 kg; Divos 2 VM13, 11 kg; and Divos LS VM15, 2 kg.

10.8 Monitoring and recording

Following commissioning of the membrane installation by the equipment manufacturer, it is very important that accurate daily records are made and kept by the customer on both production performance and cleaning cycle. The following information should be recorded and stored for reference:

- feed pressure, and particularly any variations;
- booster pressure entering and exiting each loop,
- temperature readings entering and exiting each loop;
- specified product, if more than one being processed;
- production capacity after first hour;
- production capacity during last hour before production ceases;

- production time since membrane installation was last cleaned;
- source and specification of water used for cleaning;
- cleaning time and cleaning procedure carried out at end of production; and
- water flux/TMP reading at end of cleaning prior to commencing production; as flux/TMP varies with temperature, readings must be taken at a constant temperature, e.g. 10°C or 20°C, to avoid anomalies.

This is to ensure that the installation is performing according to the original design specification, to provide trends over a period of time and, more importantly, to provide an early warning system in order to prevent membrane damage.

The water flux test relates to the permeation flux rate of new membranes when first tested after being cleaned to remove preservative and glycol from the membrane pores, and is carried out while running only the feed pump (booster pumps shut off). A water flux or TMP test procedure is also carried out after every cleaning cycle, and is performed (at a specific pressure and temperature); the data obtained are compared against the original new membrane value. This will indicate when and if the membranes have been restored to as 'good as new' condition, i.e. all fouling (soiling) has been removed.

As far as the production manager is concerned, the most important issue is that the overall output be kept as high as possible at all times: that is, filtration capacity does not become the rate-limiting step, because reduced capacity equals financial loss.

10.9 Recent developments

In general, much research is carried out by universities and research institutes, and sometimes in cooperation with sponsoring companies. Nowadays, the main focus of research in membrane filtration is in the field of membrane fouling and new membrane materials; little research is undertaken on membrane cleaning. However, ultrasonic cleaning seems to be an interesting topic that has been investigated by several research groups.

10.9.1 *Ultrasonic cleaning*

Recently, the use of ultrasound has attracted considerable interest as an alternative cleaning approach to the conventional methods. Muthukumarana *et al.* (2005) studied the ultrasonic cleaning of polysulphone ultrafiltration membranes fouled with dairy whey solutions. They found that ultrasonic cleaning improves the cleaning efficiency, suggesting that the ultrasonic energy contributes to the mechanical energy of the cleaning solution by increasing the turbulence. Furthermore, the ultrasonic effect was found more significant in the absence of surfactant, and showed less influence of temperature and trans-membrane pressure. Kyllönen *et al.* (2006) found similar results in their experimental studies on ultrasound treatment in cross-flow membrane filtration of industrial wastewater. Ultrasound treatment at atmospheric pressure during an intermission pause in filtration was found to be an efficient and gentle method in membrane cleaning. A relation was found between the membrane pore size and the effect of ultrasound treatment. The flux increased significantly when ultrasonic treatment was applied from the feed side of the membrane with tight pores, whereas the membrane became plugged by wastewater colloids when membranes with more open pores were used.

10.9.2 *Microsieves*

Microsieve membranes are very thin flat-sheet devices that contain pores with the same size and shape all along their surface (Gironès i Nogué, 2005). The major advantages of microsieves are their high porosity with very narrow pore size distribution, and their very thin separation layer, which allows very high fluxes and minimises internal pore fouling. According to Brans *et al.* (2006) the fluxes can be up to 100 to 1000 times higher than when using conventional polymeric or ceramic membranes, even at low trans-membrane pressures. Microsieves are made mostly of inorganic material, such as silicon nitride, and are manufactured with photolithographic techniques developed in the semiconductor industry. Recently, polymeric microsieves have been developed, which are fabricated by phase separation micro-moulding. Silicon nitride microsieves can be applied in dairies (fractionation of milk), in clarification of beer, in separation of blood cells, and for analytical purposes.

10.9.3 *High tolerant membranes*

Throughout time, mankind has always strived for improvements in all spheres, and the field of pH and temperature tolerance of membrane materials is no exception.

Over the past few years, spiral-wound elements with higher tolerances to both temperature and pH have been developed: for example, both Koch and Alfa Laval have introduced these kinds of spiral-wound UF element. Alfa Laval and Dow Filmtec have also developed spiral-wound RO elements with better tolerances.

These higher tolerances can lead to improved cleaning performance by enabling faster cleaning and at the same time providing better control over microbiological growth in the membrane plants. In addition, as some of the fatty acids have melting points close to 69°C, these can now be melted during plant cleaning, because the ‘short term’ cleaning tolerance, depending on membrane supplier, has been extended to pH 1–12. A further advantage is the potential elimination of traditional chemicals: for example, sodium metabisulphite solutions, which are used for membrane sanitisation, have been replaced by hot water up to temperatures of 85°C.

10.10 Conclusions

In common with other CIP applications, it is essential to know and understand the nature of the fouling (soiling) to be removed. Although the energy relationship (temperature, energy, chemical and time) holds true, there is another overriding factor that governs the cleaning regime – the membrane type. It is essential that the pH and temperature tolerance is not exceeded, to avoid irreversible membrane damage and costly replacement. Always check the detergent products(s) information sheet(s) to ensure compatibility with the specification of your membrane supplier.

Cleaning procedures are carefully planned to take into account site operational parameters provided by the customer in line with the OEM's design specifications of the installation: therefore it is essential to maintain daily records of both production and cleaning cycles, for trend analysis and for early warning of possible membrane damage. Should there be a change in operational conditions, e.g. feedstock, or production runs, it is quite feasible that the existing cleaning programme will be inadequate to remove all fouling (soiling) from the membrane. Advice should be sought from your chemical supplier as soon as is practical, and preferably before changes are made, to avoid unnecessary reduced production capacity or possible membrane damage.

Membrane filtration is a fast-developing separation technique that is applicable in many areas, such as food and beverages, (waste) water treatment, drinking water production, and the pharmaceutical, textile and electronic industries. Recent years have seen significant increases in the cost of water (both 'in' and 'out' – effluent) and energy charges, and the forecast is for continued increases. Accordingly, new applications are being sought, e.g. concentration of products to remove water for reuse and recycle purposes to reduce total site operational costs. Since the mid 1990s this has become more feasible through large-scale production plant, where the manufacturing costs have decreased, making membranes commercially economic. As a consequence, there is also a faster development of membrane materials. New membrane materials tend to have lower fouling potential, more uniform pore size distributions and a thinner separation layer, which provides higher fluxes through the membrane. In addition, these materials have higher mechanical, thermal and chemical resistances. However, biofouling is still difficult to prevent and to remove. Therefore, it is more likely that future developments will focus on new processing procedures and techniques to minimise deposition, and alternative cleaning products and techniques for more efficient biofouling removal.

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11 Laboratory Test Methods

W.J. Watkinson

11.1 Introduction

This chapter is a revision of the data presented as Chapter 9 in the 2nd edition of *CIP: Cleaning in Place* (Riley, 1990). Additional methods have been added for peracetic acid, and only minor modification has been made to certain of the other methods (see also Anonymous, 2006). Only wet or non-instrumental methods have been considered.

There are numerous possible reasons for testing detergents, disinfectant, detergent/disinfectant user solutions or neat products. These reasons could range from simple concentration checks of in-use solutions to full-scale analysis to check product quality and integrity. For ease of discussion, the test methods given below are segregated into two sections: (a) test methods for in-use solutions; and (b) test methods for neat products. Although in many cases the test methods applied are fundamentally the same in both sections, the end information obtained is invariably used for different purposes.

It is assumed, for all the methods, that the following will be in place:

- knowledge of basic analytical chemistry techniques;
- proper care and attention to the use of laboratory equipment;
- adequate precautions taken for health and safety reasons; and
- protective equipment, together with the necessary risk assessments.

11.2 Test methods for in-use solutions

11.2.1 *Alkalinity*

This is probably the most widely used test for detergent solutions, not only in the dairy industry, but also in the majority of industrial applications. In most cases the results express the active (or total) alkalinity as a percentage of NaOH (or Na_2O). This is irrespective of whether the alkalinity is derived primarily from caustic soda or from other non-caustic alkalis (e.g. sodium carbonate, sodium metasilicate, potassium silicate). Alternatively, if the active (or total) alkalinity of the neat product is known, then the percentage product in solution can be easily calculated. The information obtained from this test can be used as a check on standard procedures for cleaning-in-place (CIP) (or bottle washing), for calibration of dose and control equipment, or as part of an overall method of monitoring detergent usage.

Pipette 10 mL of the test solution into a conical flask. Add a few drops of phenolphthalein indicator, and the solution will turn pink. Titrate with 0.1 M HCl until the pink colour just

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disappears. Measure the number of mL of acid used; this is the titre. The active alkalinity can be expressed as follows:

$$\text{Active alkalinity (as NaOH, g 100 mL}^{-1}\text{)} = \frac{\text{titre} \times \text{molarity HCl} \times 4}{\text{volume of solution taken}}$$

Where percentage product is required, this can be calculated as follows:

$$\text{Product (g 100 g}^{-1}\text{)} = \frac{\text{\% active alkalinity found (as NaOH)} \times 100}{\text{\% active alkalinity (as NaOH) in neat product}}$$

For liquid products that are often sold by volume, the percentage product by volume (as opposed to weight) is often required.

$$\text{Product (mL 100 mL}^{-1}\text{)} = \frac{\text{\% product (g 100 g}^{-1}\text{)}}{\text{specific gravity of neat product}}$$

Where total alkalinity is required, the method is the same as that described above apart from the indicator. In this case, methyl orange indicator is used, an orange colour indicating the end point. The calculation for total alkalinity is

$$\text{Total alkalinity (as NaOH, g 100 mL}^{-1}\text{)} = \frac{\text{titre} \times \text{molarity HCl} \times 4}{\text{volume of solution taken}}$$

Note: The presence of available chlorine in the test solution will bleach the colour of most indicators. If the test solution contains available chlorine, then a few crystals of sodium thiosulphate should be added to neutralise this effect.

11.2.2 *Acidity*

The method for determining the acidity is very similar to that for alkalinity, but obviously uses an alkali as the titre. The results obtained are usually calculated as an *acid value* irrespective of the type or number of types of acid present in the solution. The acid value is the number of mg of KOH necessary to neutralise 1 g of the product solution.

Pipette 10 mL of the test solution into a conical flask, and add a few drops of phenolphthalein indicator. Titrate with 0.1 M NaOH to the first permanent tinge of pink. The calculation is

$$\text{Acid value (mg KOH g}^{-1}\text{)} = \frac{\text{titre} \times \text{molarity of NaOH} \times 56.1}{\text{volume taken}}$$

The percentage product concentration can be calculated as follows:

$$\text{Product (g 100 g}^{-1}\text{)} = \frac{\text{acid value of test solution} \times 100}{\text{acid value of neat product}}$$

To calculate the mL 100 mL^{-1}, this needs to be divided by the specific gravity of the product.

The acidity can also be expressed as percentage acidity, usually as an equivalent of the predominant mineral acid present. These results are calculated as follows:

$$\text{Acid (g 100 g}^{-1}\text{)} = \frac{\text{titre} \times \text{factor} \times \text{molarity of NaOH}}{\text{volume taken}}$$

The factors for H_3PO_4, H_2SO_4, HNO_3 and HC1 are 4.9, 4.9, 6.3 and 3.65, respectively.

11.2.3 *Ethylenediaminetetra-acetic acid (EDTA)*

The need to know the concentration of free EDTA is not normally associated with calculations regarding product concentration. In this context, it generally means the amount of EDTA that is not complexed with alkaline earth metals, such as calcium and magnesium. The determination of free EDTA is usually used to calculate the residual activity at the end of a cleaning cycle where a predetermined minimum level has been stipulated. Alternatively, the calculation can be made at the beginning and end of a cleaning cycle to ascertain the amount exhausted during the cycle. This information may then be used to calculate the initial product concentration that should be used.

Pipette 25 mL of the test solution into a conical flask, and adjust the pH to approximately neutral (i.e. pH 5–8) using dilute hydrochloric acid. Add 50 mL ammonia buffer, and 3–4 drops of Solochrome black indicator. In the presence of free EDTA, the solution will go blue. Titrate with 0.01 M zinc chloride until the solution just changes to violet/red. The calculation is as follows:

$$\text{EDTA (g 100 mL}^{-1}\text{)} = \frac{\text{titre} \times \text{molarity of Zn} \times 29.2}{\text{volume taken}}$$

In many cases, the levels will be required as parts per million (ppm), which is equivalent to μg g^{-1}:

$$\text{EDTA (}\mu\text{g g}^{-1}\text{ or mg L}^{-1}\text{)} = \frac{\text{titre} \times \text{molarity of Zn} \times 29.2 \times 10^4}{\text{volume taken}}$$

11.2.4 *Total EDTA*

The total EDTA present in a solution (whether free or complexed) can be determined using the following method, and the result can be used to determine the initial concentration of EDTA or detergent used for a clean.

Pipette 25 mL of the test solution into a suitable conical flask. Neutralise the solution to pH 4–5 using dilute acetic acid (i.e. 2 M). Add several drops of PAN indicator, and titrate with 0.01 M copper sulphate solution. The end point has been reached when the colour has changed from yellow to a full wine red. Note the titre of copper sulphate.

$$\text{Total EDTA (g 100 mL}^{-1}\text{)} = \frac{\text{titre} \times \text{molarity CuSO}_4 \times 29.2}{\text{volume of test solution}}$$

Note: μg g^{-1} EDTA = % EDTA × 10^4, and the calculation for percentage product is as follows:

$$\text{Product (g 100 g}^{-1}\text{)} = \frac{\text{\% total EDTA in solution} \times 100}{\text{\% total EDTA in neat product}}$$

11.2.5 *Total water hardness*

This is a very important consideration when recommending the type and concentration of alkaline detergents for cleaning in the dairy industry. Ignoring water conditions can often lead to poor cleaning, the build-up of milkstone on plant, and subsequent product and production failures.

Pipette 50 mL of the water to be tested into a conical flask, add 10 mL ammonia buffer, and 5–6 drops of Solochrome black indicator. Titrate with 0.01 M EDTA until all traces of the red colour disappear. The end point is blue-grey; note the titre of EDTA.

$$\text{Total hardness (as µg g}^{-1}\text{ or mg L}^{-1}\text{ CaCO}_3) = \frac{\text{titre} \times \text{molarity of EDTA} \times 10^5}{\text{volume of water taken}}$$

11.2.6 *Disinfectants*

There is a wide range of chemical disinfectants and detergent/disinfectants used in the dairy industry. It is essential to be able to calculate the levels of these chemicals (i.e. active ingredients). In addition, it is often important that the recommended concentrations are not significantly exceeded, as this can lead to problems of corrosion or tainting of product, not to mention additional unnecessary costs.

The calculation can also be used to determine the concentration of product being used. However, this is not normally used for detergent/disinfectants, as determination of alkalinity/acidity (where possible) will give a more accurate result. This is equally true in the case of products that utilise oxidising agents (e.g. chlorine, iodine or peroxide) as the disinfectant. The reason for this is that the presence of soiling can have a significant negative effect on their concentrations.

In this section, the main types of chemical used as disinfectants will be reviewed. They are: chlorine, iodine, peroxides, and quaternary ammonium compounds (QACs).

Available chlorine

Pipette 100 mL of the test solution into a conical flask, add 10 mL (20 g 100 g^{-1}) potassium iodide (KI) solution and 10 mL (20 mL 100 mL^{-1}) sulphuric acid, and titrate with 0.1 M sodium thiosulphate until the solution is colourless. As the brown colour fades, a few drops of soluble starch may be added. The solution will go dark blue; titrate until the blue colour just disappears.

$$\text{Available chlorine (µg g}^{-1}\text{ or mg L}^{-1}) = \frac{\text{titre} \times \text{molarity Na}_2\text{S}_2\text{O}_3 \times 35\,500}{\text{volume of sample taken}}$$

Available iodine

Pipette 100 mL of the test solution into a conical flask, add 50–100 mL deionised water, and titrate with 0.01 M sodium thiosulphate until the brown-yellow colour is completely discharged. Starch indicator can be used.

$$\text{Available iodine (µg g}^{-1}\text{ or mg L}^{-1}) = \frac{\text{titre} \times \text{molarity Na}_2\text{S}_2\text{O}_3 \times 1.27 \times 10^5}{\text{volume of sample taken}}$$

Available oxygen

Pipette 100 mL of the test solution into a conical flask, and add 10 mL of 20 g 100 g^{-1} potassium iodide solution, 10 mL of 20 mL 100 mL^{-1} sulphuric acid and 2 g ammonium molybdate. Swirl to dissolve, and allow the flask to stand for 5 min. Titrate the liberated iodine with 0.1 M sodium thiosulphate until the solution is colourless (starch indicator may be used), and note the titre of sodium thiosulphate.

$$\text{Available oxygen (g 100 mL}^{-1}) = \frac{\text{titre} \times \text{molarity Na}_2\text{S}_2\text{O}_3 \times 8}{\text{volume of sample taken}}$$

Peracetic acid content

Pipette 50 mL test solution into a conical flask, and add 20 mL of 25 g 100 mL^{-1} sulphuric acid. Titrate with 0.1N potassium permanganate until a faint pink colour is obtained (be careful not to add excess permanganate). Add 5 mL of 10 g 100 g^{-1} potassium iodide solution and 2 mL starch indicator, and titrate with the 0.1N sodium thiosulphate until the brown colour disappears.

$$\text{Peracetic acid (PAA) } (\mu\text{g g}^{-1} \text{ or mg L}^{-1}) = \text{titre} \times \text{normality of sodium thiosulphate} \times 760$$

Quaternary ammonium compounds (QACs)

Caution: This method uses chloroform, and proper risk assessments must be performed prior to performing the method.

Pipette 10 mL of test solution into a stoppered 100 mL measuring cylinder, and add 15 mL chloroform and 10 mL mixed indicator. Add from a burette 0.0004 M sodium lauryl sulphate solution, and shake the contents vigorously after each addition. A blue colour should be visible in the lower chloroform layer as it separates out on standing. Continue the addition of sodium lauryl sulphate until the colour of the chloroform layer is just pink, and note the titre of sodium lauryl sulphate.

$$\text{QAC } (\mu\text{g g}^{-1} \text{ or mg L}^{-1}) = \frac{\text{titre} \times \text{molarity of anionic} \times \text{molecular weight of QACs} \times 100}{\text{volume of sample taken}}$$

Miscellaneous tests

There are several sample tests that are available in the testing of detergent solutions. In many cases they are not quantitative in the strict sense, but they may give a very good indication of the likely composition or concentration of the solution. Some examples of these are as follows.

- *pH* measurement is a good indication to check the presence of either acid or alkaline detergents.
- The *conductivity* test can be used in a similar manner to pH. It is not only for determining the presence of most detergents, but is also a good indication of concentration. This is especially true with highly alkaline or acid detergents.

- *Test strips* are available on the market that may help in identifying detergents in solutions. These include:
  - pH (an extensive selection of full and narrow range papers is available);
  - free chlorine and iodine (qualitative test papers only); and
  - total water hardness, chloride, nitrite, sulphite, peroxide (semi-quantitative test strips are available).
- A *specific gravity* check on the caustic-based liquid detergent may give a good indication as to the actual caustic soda level: see Table 11.1. The data shown are for caustic soda solutions without any other components; the presence of other components may

Table 11.1 Specific gravity (SG) of caustic soda solutions (with no additives).

NaOH (g 100 g^{-1})	SG at 15.5°C	NaOH (g 100 g^{-1})	SG at 15.5°C
1	1.012	26	1.289
2	1.023	27	1.300
3	1.034	28	1.310
4	1.045	29	1.321
5	1.056	30	1.332
6	1.067	31	1.343
7	1.079	32	1.353
8	1.090	33	1.363
9	1.101	34	1.374
10	1.112	35	1.384
11	1.123	36	1.394
12	1.134	37	1.404
13	1.145	38	1.415
14	1.156	39	1.425
15	1.167	40	1.434
16	1.178	41	1.444
17	1.190	42	1.454
18	1.201	43	1.463
19	1.212	44	1.473
20	1.223	45	1.483
21	1.234	46	1.492
22	1.245	47	1.502
23	1.256	48	1.511
24	1.267	49	1.522
25	1.278	50	1.530

have an effect on the specific gravity. However, in highly caustic products such as bottle washing and some CIP detergents, it does give a good correlation.

11.3 Qualitative test methods for neat detergents

This section concentrates on both the qualitative and quantitative methods of analysis of the more common components found in detergents used in the dairy industry. Unless otherwise indicated, the following tests are carried out on a detergent solution containing approximately 20 g detergent/detergent-disinfectant/disinfectant L^{-1}.

11.3.1 *Alkalinity*

A high pH (>13) and a high titratable active alkalinity would normally indicate the presence of sodium hydroxide in dairy detergents. However, there may be other inorganic compounds, such as silicates, carbonates and phosphates, that can make a significant contribution to the alkalinity.

Silicates

Add 2 mL of the detergent solution to a test tube followed by the addition of 3 mL concentrated hydrochloric acid. Boil for 5 min, cool, and add an excess of concentrated ammonium hydroxide (concentrated ammonia solution). A white precipitate that flocculates on boiling indicates the presence of silicates.

Phosphates

Add 2 mL of the detergent solution to a test tube, and then add 3 mL concentrated nitric acid and 5 mL ammonium molybdate solution. Boil the solution for 5 min; the appearance of a yellow precipitate indicates the presence of phosphates.

Carbonates

Add about 2 mL of detergent solution to a test tube and 1 mL of concentrated sulphuric acid. Effervescence indicates the presence of carbonates.
Note: If chlorine is present, effervescence may occur; if chlorine is suspected, add a few crystals of sodium thiosulphate before the addition of the sulphuric acid.

11.3.2 *Acids*

There are various acids used in the dairy industry. These are primarily phosphoric and nitric acids (or mixtures of both), although occasionally hydrochloric and sulphuric acids are used. The test methods for each are as follows.

Nitric acid

Add 3 mL of detergent solution to a test tube, 3 mL of filtered, saturated ferrous sulphate solution, and shake to mix. With care, and using a Pasteur pipette (or similar), add 3–5 mL concentrated sulphuric acid slowly down the side of the tube so that the acid forms a layer beneath the aqueous mixture. If positive, a brown ring will form where the two liquids meet.

Phosphoric acid

See 'Phosphates' in Section 11.3.1 above.

Hydrochloric acid

Add about 2 mL of detergent solution to a test tube, 2 mL concentrated nitric acid, and an excess of silver nitrate solution. A white flocculant precipitate indicates the presence of chlorides (or hydrochloric acid).

Sulphuric acid

Add about 2 mL of detergent solution to a test tube, 2 mL concentrated hydrochloric acid, and an excess of barium chloride solution. A white precipitate indicates the presence of sulphates (or sulphuric acid).

11.3.3 *Water-conditioning agents*

In addition to phosphates, there are several organic water-conditioning agents used in dairy industry detergents. Under certain conditions, many of these will act as reducing agents, such as sodium gluconate (and heptonate), EDTA and organic phosphorus compounds (phosphonates). However, other compounds, such as sulphites, nitrites and some non-ionic surfactants, will also give a positive test for reducing agents.

Reducing agents

Add about 2 mL of the detergent solution to a test tube, 2 mL concentrated sulphuric acid, and 2 mL potassium permanganate solution (3.16 g L^{-1}). Heat (if necessary) to over 60°C for a few minutes; decolorisation of the permanganate indicates the presence of a reducing agent.

Ethylenediaminetetra-acetic acid (EDTA)

Pipette 50 mL of detergent solution into a conical flask, adjust the pH to ~7. Add 50 mL ammonium buffer, 3–4 drops of Solochrome black indicator, and one drop of 0.01 M zinc chloride. If the solution remains blue, EDTA is probably present. Continue adding the zinc chloride until the colour changes from blue to violet. A clear end point indicates EDTA.
Note: Phosphonates and nitrilotriacetic acid (NTA) can interfere with this method.

Sodium gluconate/heptonate

Take a 5 mL sample of the detergent solution, and acidify with dilute nitric acid. Add 2–3 mL silver nitrate solution; if the solution is clear, proceed to the next step (if not, check the acidity; if acid is present, the solution should be filtered before proceeding). Add ~0.1 g (spatula-end) periodic acid (i.e. crystals) to a separate test tube and dissolve in 3 mL of deionised water. Add this to the first solution; a white precipitate, which will normally take a few seconds to appear, indicates the presence of sodium gluconate or heptonate.

Organic phosphorus compounds (i.e. phosphonates)

For this test, inorganic phosphate must be absent; this must be checked first by the acid/molybdate test. Having confirmed that inorganic phosphate is absent, take 1–2 g neat detergent in a clean platinum crucible, add ~3 g sodium hydroxide pellets, and heat the crucible. Apply the heat carefully and patiently until the sodium hydroxide has fused; a dull red glow appears, and the contents of the crucible are liquid. Allow the crucible to cool; the contents will 'set' when cool. Add some deionised water to the crucible, and heat it so as to dissolve the solid as much as possible. Filter the solution into a test tube. While cooling the tube under a cold water tap (hold the tube using a clamp), slowly add concentrated nitric acid until the solution is acidic. *Care*: Boil the solution, cool slightly, and add ammonium molybdate reagent; a yellow precipitate indicates a positive result.
Note: Anionic phosphate esters (hydrotropes) give a positive test.

Oxidising agents

Chlorine

To ~0.5 g sample of the detergent, add ~10 mL water and 2 mL concentrated sulphuric acid. Evolution of a pungent-smelling gas, which bleaches moist litmus paper, indicates the presence of chlorine. Alternatively, add a few drops of methyl orange indicator to the acidified solution; bleaching of the red colour indicates chlorine.

Peroxides

To ~0.5 g sample of detergent, add 10 mL water, 2 mL concentrated sulphuric acid and 2 mL of 20 g 100 g^{-1} potassium iodide. A brown colour indicates chlorine or peroxides. To a separate acidified solution, add a few drops of methyl orange. Unlike chlorine, peroxides will not bleach methyl orange indicator.

Surfactants

Anionics

Caution: This method uses chloroform; proper risk assessments must be performed prior to performing the method.

Add 25 mL of detergent solution to a 100 mL measuring cylinder, add 10 mL mixed indicator, and adjust to pH 2. Add 15 mL chloroform, stopper the cylinder, and shake vigorously. Observe the colour of the chloroform layer, adjust to pH 13, and shake vigorously. Observe the colour of the chloroform layer; a pink coloration in the chloroform layer at both pH 2 and pH 13 indicates the presence of anionic surfactants.

Quaternary ammonium compounds (QACs)

Caution: This method uses chloroform; proper risk assessments must be performed prior to performing the method.

As with the anionics test, a blue coloration at pH 2 and pH 13 indicates the presence of cationic surfactants. However, cationic bactericides that are not surfactants (e.g. polybiguanides) will not give a positive result.

Amphoterics

Caution: This method uses chloroform; proper risk assessments must be performed prior to performing the method (see anionics and cationics above).

A blue coloration at pH 2 and pink coloration at pH 13 indicate the presence of amphoteric(s).

Non-ionics

These compounds are more difficult to detect, especially when mixed with other surfactants. However, there is a relatively simple method that will indicate ethoxylated non-ionics, although some anionics (ethoxylated alcohol sulphates) will also give a positive result.

Caution: This method uses methylene chloride; proper risk assessments must be performed prior to performing the method.

In a beaker, carefully neutralise 50 mL of detergent solution to pH 7 using 1 M NaOH or 1 M HCl (or 0.1 M). Transfer the neutralised solution to a stoppered measuring cylinder, add 20 mL cobaltothiocyanate reagent and 20 mL methylene chloride (dichloromethane), and shake the cylinder. Allow the phases to separate; a blue colour in the lower solvent layer indicates a positive result.

Miscellaneous

In some formulations, predominantly powders, fillers (i.e. compounds that have no real positive or negative effect on the performance of the product) may be added. The most widely used filler is sodium sulphate, but in some cases sodium chloride is used.

Sulphates

Add ~2 mL of detergent solution to a test tube, 2 mL concentrated hydrochloric acid, and an excess of barium chloride solution. A white precipitate indicates the presence of sulphates (or sulphuric acid).

Chlorides

Add ~2 mL of detergent solution to a test tube, 2 mL concentrated nitric acid, and an excess of silver nitrate solution. A white flocculant precipitate indicates the presence of chlorides (or hydrochloric acid).

Note: In many raw materials used for dairy detergents, sodium chloride is present as an impurity.

11.4 Quantitative tests for neat detergents

11.4.1 *Alkalinity*

The results of the standard tests for alkalinity will give the sum of all components present, acidic components contributing a negative value. The various non-caustic components (where present) can be calculated separately. The alkalinity derived from these can be subtracted from the initial alkalinity to give the caustic soda (or potash) concentration.

Free and total alkalinity

Weigh ~1 g of test product into a 250 mL conical flask, add 50 mL water, a few drops of phenolphthalein indicator, and shake to dissolve. Titrate with 1 M HCl to a colourless end point, and note the titre (i.e. T_1).

$$\text{Active alkalinity (NaOH, g 100 g}^{-1}) = \frac{T_1 \times \text{molarity of HCl} \times 4}{\text{weight of sample}}$$

For total alkalinity, add a few drops of methyl orange indicator to the colourless solution, titrate further to the methyl orange end point, and note the additional titre (i.e. T_2) of hydrochloric acid.

$$\text{Total alkalinity (Na}_2\text{O, g 100 g}^{-1}) = \frac{T_1 + T_2 \times \text{molarity of HCl} \times 3.1}{\text{weight of sample}}$$

Notes:

(1) To convert g 100 g^{-1} Na_2O to g 100 g^{-1} NaOH, multiply by a factor 4/3.1.

(2) Any foam formed during the titrations should be knocked down using isopropyl alcohol (IPA).

11.4.2 *Inorganic silicate*

Prepare a solution of the detergent by dissolving 20 g (i.e. accurately weighed) in 1 L of distilled water (the detergent solution). Pipette 50 mL of the detergent solution into a clean 250 mL Pyrex squat beaker, add 10 mL concentrated hydrochloric acid, and evaporate to dryness on a water bath in the fume cupboard. Add a further 10 mL concentrated hydrochloric acid, and again evaporate to dryness. Dry the precipitate in the beaker by placing it in the oven for at least 1 h at 120°C. Return the beaker to the water bath, and add 50 mL dilute hydrochloric acid (bench). Remove the solid silicate adhering to the sides of the beaker by means of a rubber ‘policeman’. Filter the hot solution through a Whatman No. 41 filter paper (ashless), and wash any solid remaining in the beaker with hot and very dilute hydrochloric acid (~2 mL dilute hydrochloric acid to 400 mL distilled water) into the filter paper. All solids must be removed from the beaker. Rinse the filter paper with hot and very dilute hydrochloric acid (~2 mL dilute HCl to 400 mL distilled water) at least twice to remove dissolved solids, and do not allow the washings to exceed 200 mL.

Carefully fold up the moist filter paper containing the silica, and transfer to a clean, weighed platinum crucible. Dry the paper slowly over a micro Bunsen, slowly char the paper (do not allow it to ignite), and then burn off the carbon with a normal Bunsen burner. If the contents are not completely white, a Meka burner may be employed, or a muffle furnace at 900–1000°C. Successive removal, cooling and reheating of the crucible normally whitens stubborn solids. Reweigh the cooled crucible and calculate the amount of silica obtained.

$$\mathrm{SiO_2}\ (\mathrm{g\ 100\ g^{-1}})\ \text{in the detergent} = \frac{\text{weight of silica} \times 20 \times 100}{\text{weight of detergent taken}}$$

Note: If the SiO_2 content is low, the detergent may be weighed directly into the beaker instead of using larger aliquots of a detergent solution (e.g. use 1–2 g detergent for SiO_2 less than 5 g 100 g^{-1}), and the calculation is as follows:

$$\mathrm{SiO_2}\ (\mathrm{g\ 100\ g^{-1}}) = \frac{\text{weight of silica} \times 100}{\text{weight of sample}}$$

$$\text{Sodium metasilicate (anhydrous)}\ (\mathrm{g\ 100\ g^{-1}}) = \frac{\%\ \mathrm{SiO_2} \times 100}{49.2}$$

$$\text{Sodium metasilicate pentahydrate}\ (\mathrm{g\ 100\ g^{-1}}) = \frac{\%\ \mathrm{SiO_2} \times 100}{28.3}$$

11.4.3 *Inorganic phosphate*

Prepare an accurately weighed (W_1) aqueous solution of ~20 g L^{-1} of detergent. Pipette V mL of this solution into a 600 mL beaker (see note 1 below). Add 13 mL concentrated nitric acid, and dilute to 100 mL. Boil the solution for 30 min (i.e. cover the beaker with a large watch glass and keep the volume up to 100 mL), and cool to < 70°C.

Afterwards, add 50 mL of the Quimociac reagent (see note 2), place the open beaker on a boiling water bath for 10 min, and then heat the contents to boiling for 30 s. Cool the beaker by standing it in a sink of cold water for 30 min.

Filter the yellow precipitate through a dried and weighed paper No. 4 and sintered glass crucible. Wash the precipitate with six-times 30 mL portions of cold water. Dry the precipitate in an oven at 250°C for 1–2 h, cool and weigh. Calculate the weight of precipitate (W_2) (see note 3).

$$\mathrm{P_2O_5}\ (\mathrm{g\ 100\ g^{-1}}) = \frac{W_2 \times 0.03207 \times 1000 \times 100}{W_1 \times V}$$

Trisodium orthophosphate (g 100 g^{-1}) = % P_2O_5 × 2.31

Sodium tripolyphosphate (g 100 g^{-1}) = % P_2O_5 × 1.73

Sodium hexametaphosphate (g 100 g^{-1}) = % P_2O_5 × 1.49

Tetrapotassium pyrophosphate (TKPP) (g 100 g^{-1}) = % P_2O_5 × 2.33
(this compound is normally found in liquid detergents only).

Notes:

(1) The aliquot should be chosen so as to contain 5–20 mg P_2O_5; for most products V = 10 mL.

(2) The Quimociac reagent is prepared as follows. Dissolve 70 g sodium molybdate AR in 150 mL deionised water. Dissolve 60 g citric acid in 160 mL deionised water and add 75 mL concentrated nitric acid. Gradually, add the molybdate solution to the citric acid solution with stirring. Dissolve 5 mL quinoline, purified for phosphate determination, in a mixture of 31 mL concentrated nitric acid and 100 mL water. Gradually, add this solution to the citric acid–molybdate solution with stirring, and leave overnight. Filter the solution through a paper No. 4 and sintered glass crucible without washing. Add 280 mL acetone, dilute to 1000 mL with water, and store in the dark in a polythene bottle.

(3) The weight of precipitate should not be more than 0.6 g. If this is so, the determination should be repeated using a smaller aliquot of detergent solution (V).

This method of analysis gives an acceptable result when silicate is present, but to determine the type(s) of phosphate present in the product a thin-layer chromatographic method can be employed.

11.4.4 *Carbonates*

Accurately weigh 1 g of neat detergent into a 250 mL conical flask, add ~50 mL deionised water and shake to dissolve. Add two drops of phenolphthalein indicator and titrate with 1 M hydrochloric acid to just colourless. Note the titre (T_1) of hydrochloric acid; then add a few drops of methyl orange indicator, continue the titration with the 1 M hydrochloric acid to the methyl orange end point, and note the total titre (T_2) of hydrochloric acid. Heat the contents of the flask to boiling and then cool the flask back to room temperature. Check that the contents are still exactly at the methyl orange end point (adjust if necessary), add several drops of phenolphthalein indicator, and titrate with 1 M sodium hydroxide to a permanent pink colour. Note the titre (T_3) of 1 M sodium hydroxide.

$$\text{Sodium carbonate (g 100 g}^{-1}\text{)} = \frac{(T_2 - T_1 - T_3)\times 10.6 \times \text{molarity of NaOH/HCl}}{\text{weight of detergent taken}}$$

Notes:

(1) To avoid complication, the molarity of the sodium hydroxide and hydrochloric acid should be the same.

(2) If available chlorine is present in the detergent, a few crystals of sodium thiosulphate should be added to the flask before titrating.

(3) For more accurate results, a pH meter may be used instead of the visual indicators; in this case, use pH 8.3 and pH 4.0 instead of the phenolphthalein and methyl orange end point, respectively.

11.4.5 *Acidity*

The calculation for acidity is normally expressed as an acid value, but it can equally well be expressed as percentage acid (g 100 g^{-1}). Weigh ~5 g of test product into a conical flask, add 50 mL of deionised water and a few drops of phenolphthalein, shake to dissolve, titrate with 1 M NaOH until the first permanent tinge of pink, and note the titre of NaOH.

$$\text{Acid value (mg KOH g}^{-1}\text{)} = \frac{\text{titre} \times 56.1 \times \text{molarity of NaOH}}{\text{weight of sample taken}}$$

To express as % acid the calculation is as follows:

$$\text{Acid (g 100 g}^{-1}\text{)} = \frac{\text{titre} \times \text{factor} \times \text{molarity of NaOH}}{\text{weight of sample taken}}$$

Factors:

- phosphoric acid (H_3PO_4) = 4.9
- sulphuric acid (H_2SO_4) = 4.9
- nitric acid (HNO_3) = 6.3
- hydrochloric acid (HC1) = 3.65

11.4.6 *Water-conditioning agents*

Ethylenediaminetetra-acetic acid (EDTA)

Accurately weigh between 1 and 10 g (depending on the anticipated result) of test product into a conical flask. Add 50 mL of deionised water, adjust the solution (omitting indicator) to pH ~4, add 50 mL of ammonia buffer and 3–4 drops of Solochrome black indicator, and titrate with 0.1 M zinc chloride solution from blue to just violet.

$$\text{EDTA (as acid} - \text{g 100 g}^{-1}\text{)} = \frac{\text{titre} \times \text{molarity of Zn} \times 29.2}{\text{weight taken}}$$

Sodium gluconate/heptonate by dimedone complex

This method is very sensitive, and an estimation of the sodium gluconate/heptonate has to be made before the test is undertaken. Should the precipitate obtained at the end of the method exceed 0.1 g, then the procedure should be repeated using a smaller sample of the detergent.

In a 100 mL beaker, accurately weigh out a sample of detergent estimated so as to contain no more than 0.07 g gluconate/heptonate in the sample. Dissolve/disperse the detergent in the minimum volume of water, add one drop of phenol red, and neutralise the sample using dilute hydrochloric acid or sodium carbonate. Add 5 mL 1 M sodium bicarbonate and then 5 mL 0.3 M periodic acid solution. Mix by swirling, cover, and allow to stand for 1 h.

Add 7.5 mL 1 M hydrochloric acid and 5 mL of a 1 M sodium arsenite solution (care!). Mix, and allow the brown colour to disappear (may need a few more drops of arsenite). Wash the sides of the beaker with as little water as possible, add 5 mL 1 M sodium acetate and 3 mL of 10 g 100 g^{-1} dimedone in industrial methylated spirit, and mix by swirling. Cover and leave overnight, and filter the precipitate on a weighed sintered No. 3 glass crucible. Wash the beaker with 150 mL cold water using a ‘policeman’, dry the precipitate at 85–90°C for 90 min, cool, and reweigh the crucible. Calculate the weight (W) of precipitate.

$$\text{Gluconate/heptonate (g 100 g}^{-1}) = \frac{W \times \text{factor} \times 100}{\text{weight of detergent taken}}$$

The factors for sodium gluconate and sodium heptanone2H_2O are 0.745 and 0.989, respectively.
Note: It is advisable to run a blank on the reagents.

11.4.7 *Oxidising agents*

Available chlorine

Weigh 24 g of test product into a conical flask, add 10 mL of 20 g 100 g^{-1} potassium iodide (KI) solution and 10 mL of 20 g 100 g^{-1} sulphuric acid, and titrate the solution with 0.1 M sodium thiosulphate until the solution is just colourless.

$$\text{Available chlorine (g 100 g}^{-1}) = \frac{\text{titre} \times \text{molarity of thiosulphate} \times 3.55}{\text{weight of detergent taken}}$$

Available iodine

Weigh 10 g of the test product into a conical flask, add 50 mL distilled water, and titrate with 0.1 M sodium thiosulphate.

$$\text{Available iodine (g 100 g}^{-1}) = \frac{\text{titre} \times \text{molarity of thiosulphate} \times 12.7}{\text{weight of detergent taken}}$$

Available oxygen

Weigh 1 g of the test product into a conical flask, add 50 mL deionised water, 10 mL of 20 g 100 g^{-1} potassium iodide (KI) solution, 10 mL of 20 g 100 g^{-1} sulphuric acid, and 2 g ammonium molybdate, and then warm the flask to 40–50°C. Allow to stand for 5 min, titrate with 0.1 M sodium thiosulphate to a colourless end point, and note the titre of sodium thiosulphate.

$$\text{Available oxygen (g 100 g}^{-1}) = \frac{\text{titre} \times \text{molarity of thiosulphate} \times 8}{\text{weight of detergent taken}}$$

In all three of the above tests, soluble starch indicator may be used to obtain a sharper end point.

Hydrogen peroxide and peracetic acid (PAA) in peracetic acid products

Disinfectant products based on peracetic acid contain hydrogen peroxide (H_2O_2) and peracetic acid (PAA – CH_3CO_3H). Both materials are determined in this method: initially the hydrogen peroxide content is determined using a standard permanganate determination, followed rapidly by the peracetic acid determination by an iodometric titration using sodium thiosulphate. The peracetic acid cannot be determined specifically in one iodometric step as the hydrogen peroxide would also contribute to the iodometric titration. It is important to perform the two steps of the determination together, and as quickly as possible, to minimise the variance in results caused by the equilibrium between the varying chemical species trying to re-establish.

Weigh (to the nearest 0.0001 g) 10 g sample of the PAA disinfectant product into a volumetric flask of 1 L. Pipette 25 mL of this solution into a conical flask (250 mL), and place the sample solution on crushed ice, in order to inhibit side reactions. Add 10 mL H_2SO_4 (6N) and ~100 mL demineralised water. While mixing (swirling or stirring) the solution, start titrating with 0.1N $KMnO_4$ solution. The first 5 mL can be added quickly, and the end point is reached when the solution attains a faint pink colour. Do not overshoot the end point; an excess of titre will react with the next phase of the titration. Record the volume of titrant (V_1 mL).

Afterwards, add to the conical flask 5 mL of 20 g 100 g^{-1} KI solution; the liberated iodine (I_2), colours the solution brown. While mixing (swirling or stirring), start titrating the released iodine with 0.1N $Na_2S_2O_3$ solution until a pale yellow colour is observed. Add 5 drops of the starch solution (indicator). A strong blue colour will appear. Continue the titration until the solution has changed from blue into colourless, and persists colourless for approximately 10 s. Record the second titre volume (V_2 mL).

Calculations:

$$\text{Hydrogen peroxide (g 100 g}^{-1}\text{)} = \frac{V_1 \times T_1 \times M_{\mathrm{H_2O_2}} \times 100}{2 \times W \times f}$$

$$\text{Peracetic acid (g 100 g}^{-1}\text{)} = \frac{V_2 \times T_2 \times M_{\mathrm{PAA}} \times 100}{2 \times W \times f}$$

where

- W = weight of disinfectant used to make the 1 L dilution (ca. 10 g, to the nearest weight 0.0001 g)
- f = titration sample (25 mL)
- V_1 = volume titrant $KMnO_4$ (mL)
- T_1 = actual normality of $KMnO_4$ titration solution
- $M_{\mathrm{H_2O_2}}$ = molecular weight of H_2O_2 (34.01 g mol^{-1})
- V_2 = volume titrant $Na_2S_2O_3$ (mL)
- T_2 = actual normality of $Na_2S_2O_3$ titration solution
- M_{PAA} = molecular weight of CH_3CO_3H (76.05 g mol^{-1})

In circumstances where value V_2 turns out to be much smaller than value V_1, a more convenient value for V_2 is obtained if a 0.02N sodium thiosulphate solution (dilution from 0.1N and standardised) is applied instead of the 0.1N solution.

11.4.8 *Surfactants*

Determination of anionic surfactants

Caution: This method uses chloroform; proper risk assessments must be performed prior to performing the method.

Dissolve in water ~10 g of neat detergent, accurately weighed (*W*), in a 1 L flask, and make up to the mark. Any foam generated may be knocked down using isopropyl alcohol (IPA) from a wash bottle. Pipette 25 mL of this detergent solution into a 100 mL stoppered measuring cylinder, and neutralise the aliquot to pH ~7. Add 10 mL mixed indicator solution and 15 mL chloroform. From a 50 mL burette, add 0.004 M Hyamine 1622 solution (or any other standardised cationic solution of the same molarity); shake the cylinder vigorously after each addition. Observe the colour of the chloroform layer as it settles out on standing; it should be pink. Continue adding the Hyamine, 1 mL at a time. As the end point approaches, the chloroform will separate out much more quickly. At this stage reduce the additions of Hyamine. The end point is reached when the colour of the chloroform changes from red to a pale blue. Record the titre of Hyamine.

$$\text{Anionic (100\% active} - \text{g 100 g}^{-1}) = \frac{\text{titre} \times \text{molarity Hyamine 1622} \times \text{mol wt of anionic} \times 4}{W}$$

Typical molecular weights of anionic surfactants are: sodium linear dodecylbenzene sulphonate = 348, sodium lauryl sulphate = 288, and sodium lauryl ether (3E0) sulphate = 420.

Determination of cationic surfactants

Caution: This method uses chloroform; proper risk assessments must be performed prior to performing the method.

Weigh out accurately 10 g of neat detergent (*W*), and dissolve in water in a 1 L flask. Make the volume up to the mark; any foam generated may be knocked down using IPA. Pipette 25 mL of the detergent solution into a stoppered measuring cylinder, and neutralise the sample (if required) to pH ~7. Add 10 mL mixed indicator and 15 mL chloroform. Add from a burette standard sodium lauryl sulphate solution (0.004 M), and shake the cylinder after each addition. In the early stages of the titration separation may be slow, but towards the end of the titration the two phases separate out, and a blue colour is observed in the lower chloroform layer. Continue the titration until a pink colour is observed in the chloroform layer, and note the titre of sodium lauryl sulphate.

$$\text{Cationic (100\% active} - \text{g 100 g}^{-1}) = \frac{\text{titre} \times \text{molarity of sodium lauryl sulphate} \times \text{mol wt of cationic} \times 4}{W}$$

Typical molecular weights of cationic surfactants are: benzalkonium chloride = 354, didecyldimethylammonium chloride =362, and dioctyldimethylammonium chloride = 306.

11.4.9 *Miscellaneous*

Determination of chloride

Accurately weigh 1 g of detergent (*W*) into a 250 mL conical flask, and add 50 mL deionised water to dissolve/disperse the detergent. When all the detergent has dissolved, neutralise the solution to exactly pH 7 using dilute sodium hydroxide or nitric acid. Add ~0.5 g precipitated calcium carbonate, and then 1 mL of a 5 g 100 g^{-1} solution of potassium chromate. Titrate against standardised 0.1 M silver nitrate solution until a faint red/brown coloration persists on shaking in the originally yellow solution, and note the titre of silver nitrate.

$$\text{Chloride (as chlorine} - \text{g } 100 \text{ g}^{-1}) = \frac{\text{titre} \times 3.55 \times \text{molarity of silver nitrate}}{W}$$

$$\text{Chloride (as sodium chloride} - \text{g } 100 \text{ g}^{-1}) = \frac{\text{titre} \times 5.85 \times \text{molarity of silver nitrate}}{W}$$

Note: This method of analysis is not suitable for detergents containing available chlorine or iodine.

Determination of sulphate

An estimation of the sulphate level in the detergent has to be made before carrying out this test. If the weight of precipitate obtained at the end of the method is greater than 0.5 g, the procedure should be repeated using a smaller sample of detergent.

In a 400 mL beaker accurately weigh out a sample of detergent estimated so as to contain no more than 0.25 g sodium sulphate. Add 50 mL water and dissolve the detergent if necessary. Neutralise to pH ~7 using dilute hydrochloric acid and, when neutral, add 0.5 mL of concentrated hydrochloric acid. Afterwards, dilute the solution to 200–250 mL using deionised water. Boil the solution and, while stirring, add dropwise 10–12 mL of warm 5 g 100 g^{-1} barium chloride solution. Allow the precipitate to settle, and test the supernatant liquid for complete precipitation by adding a few drops of barium chloride solution. When precipitation is complete, keep the solution hot, but not boiling, for 1 h (steam bath). Filter off the precipitated barium sulphate through a weighed filter crucible (Gooch, sintered glass or porcelain), and wash it with hot water until the chloride reaction of the washings is negative. Dry to constant weight in an oven at 120°C, and obtain the weight of barium sulphate by subtraction.

$$\text{Chloride (as sodium chloride} - \text{g } 100 \text{ g}^{-1}) = \frac{\text{titre} \times 5.85 \times \text{molarity of silver nitrate}}{W}$$

Preparation of reagents

- *Ammonia buffer*. To prepare 5 L buffer solution, dissolve 44.5 g ammonium chloride AR in 4720 mL deionised water, and add 280 mL concentrated (specific gravity 0.88–0.90) ammonia solution. Mix thoroughly; the solution is very stable, and does not require special storage conditions.
- *Ammonium molybdate*. Dissolve 125 g ammonium nitrate in 125 mL deionised water, and add 175 mL nitric acid (concentrated, i.e. specific gravity 1.42). Also dissolve 12.5 g ammonium molybdate AR in 75 mL deionised water, and add this slowly with constant shaking to the nitrate solution. Dilute to 500 mL with deionised water, heat the solution to 60°C for several hours, and then allow the solution to stand overnight. If necessary, filter the solution through a No. 42 Whatman filter paper. This reagent has good keeping qualities, and is stable for several months.

- *Cobaltothiocyanate*. Dissolve 30 g cobalt (II) nitrate hexahydrate, 143 g ammonium chloride and 256 g potassium thiocyanate in deionised water, and make the volume up to 1 L.
- *Copper sulphate (0.01 M)*. Dissolve 2.497 g copper (II) sulphate pentahydrate AR in deionised water in a 1 L flask, and make the solution up to the mark.
- *EDTA (disodium salt) solution (0.01 M)*. This reagent is readily prepared from vials of concentrated volumetric solution or purchased as a ready-to-use solution from laboratory suppliers. Various molarities and pack sizes are available.
- *Hyamine 1622 (0.004 M)*. This reagent can be purchased ready to use from laboratory chemical suppliers.
- *Hydrochloric acid (0.1M and 1.0 M)*. These reagents are readily prepared from vials of concentrated volumetric solutions or purchased as ready-to-use solutions from laboratory chemical suppliers. Various molarities and pack sizes are available.
- *Sodium lauryl sulphate (0.004M)*. The sodium lauryl sulphate should be standardised (e.g. 1.152 g L^{-1}) before it is used.

  Caution: This method uses chloroform; proper risk assessments must be performed prior to performing the method.

  Take 25 mL sodium lauryl sulphate solution in a 100 mL stoppered measuring cylinder, and add 10 mL mixed indicator and 15 mL chloroform. Titrate with standardised 0.004 M Hyamine 1622 solution, and note the titre of Hyamine 1622.

$$\text{Molarity of sodium lauryl sulphate} = \frac{\text{titre} \times \text{molarity of Hyamine 1622}}{\text{volume of sodium lauryl sulphate solution taken}}$$

  Note: To obtain 0.0004 M sodium lauryl sulphate, pipette 50 mL 0.004 M sodium lauryl sulphate into a 500 mL volumetric flask, and make up to the mark with deionised water.

- *Silver nitrate (0.1M)*. This reagent may be purchased in concentrated or ready-to-use forms from laboratory suppliers.
- *Sodium acetate (1.0 M)*. Dissolve 6.8 g sodium acetate trihydrate in 50 mL deionised water.
- *Sodium arsenite (1.0 M)*. Dissolve 7.8 g sodium arsenite in 50 mL deionised water. *Caution*: This compound is highly toxic.
- *Sodium bicarbonate (1.0 M)*. Dissolve 4.2 g sodium hydrogen carbonate in 50 mL deionised water.
- *Sodium hydroxide (0.1 M and 1.0 M)*. These reagents are readily prepared from vials of concentrated volumetric solutions, or purchased as ready-to-use solutions from laboratory chemical suppliers. Various molarities and pack sizes are available.
- *Sodium thiosulphate (0.1M and 0.1N)*. This reagent is readily prepared from vials of concentrated volumetric solution, or purchased as a ready-to-use solution from laboratory chemical suppliers. These titrants have limited shelf-life, and should be replaced regularly.
- *Zinc chloride (0.1 M)*. Dissolve 13.63 g zinc chloride AR in deionised water in a 1 L flask. Add 3 mL concentrated bench hydrochloric acid, and make up to the mark with deionised water. For zinc chloride (0.01 M), it is prepared by diluting 0.1 M ten times with deionised water.

- *Standardisation of the zinc chloride*. This is most conveniently performed on the 0.01 M solution, multiplying the result by 10 to obtain the exact molarity of the approximately 0.1 M solution. Pipette 25 mL zinc chloride solution (~0.01 M) into a 250 mL conical flask, and add 50 mL ammonia buffer and a few drops of Solochrome black indicator. Titrate with standard 0.01 M EDTA from the original violet colour to a full blue, and note the titre of 0.01 M EDTA.

$$\text{Molarity of zinc chloride} = \frac{\text{titre} \times \text{molarity of EDTA}}{\text{volume of zinc solution}}$$

Preparation of indicators

Most of these indicators are available as ready-to-use solutions, but the preparation methods are as follows.

- *Methyl orange*. Dissolve 0.1 g methyl orange in 100 mL industrial methylated spirits (20 g 100 g^{-1}), and filter if necessary.
- *Mixed indicator*. This reagent, which is actually a dimidium bromide-disulphine blue mixed indicator, is available as a concentrated stock solution from reputable suppliers. Alternatively, it can be made up by dissolving 0.5 g of dimidium bromide in 20–30 mL of warm industrial methylated spirits (IMS) (10 g 100 g^{-1}) in deionised water, and 0.25 g of disulphine blue VN in 20–30 mL of warm IMS in water. Mix and dilute to 250 mL with IMS; this solution is to be the stock indicator. However, for dilution of the indicator, half-fill a 500 mL volumetric flask with deionised water, add 20 mL of 5 N sulphuric acid and 20 mL of the stock indicator, and dilute to 500 mL.
- *Phenolphthalein.* Dissolve 0.5 g of the solid reagent in 50 mL IMS, and add 50 mL deionised water with constant stirring. Filter the solution if necessary.
- *Pyridylazo naphthol (PAN) indicator*. Dissolve 0.03 g PAN indicator in 100 mL methanol, and filter if necessary; PAN = 1-(2-pyridylazo)-2-naphthol).
- *Phenol red*. Dissolve 0.1 g water-soluble phenol red in 100 mL deionised water.
- *Solochrome black*. Dissolve 0.5 g Solochrome black in 100 mL IMS or methanol, and add 4.5 g hydroxylammonium chloride. Filter after allowing the solution to stand overnight. The indicator is stable for up to two or three months.

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