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OUR INDUSTRY TODAY

Evaluation of Soil Deposition and Removal Processes: An Interpretive Review¹

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ABSTRACT

In each food processing plant, soil deposition and removal constitute unique processes influenced by the food, water supply, processing equipment, and cleaning regimen. Since there are so many complex as well as unique processes, the scientific literature represents many varied approaches to problems related to deposition and removal of soil. This paper considers the role of various constituents in soil residue. Likewise, the various factors contributing to the cleaning process are reviewed. Cleaning processes have changed considerably during recent years because of technological advances in food processing equipment as well as development of specialized cleaning equipment. Generally speaking, better sanitation has been attained. Yet, the exact attainment is commonly ill defined because there is a lack of precise definitions and methodology acceptable among professional sanitarians. Considerable scientific emphasis has been directed to methods of evaluating cleaning processes. The suggested methods involve microbiology, chemical analysis, and amplification of visual inspection. Yet, one of the greatest current needs is a simple, precise method for evaluating the cleaning process. This review of literature is intended to collate thought on soil deposition and removal processes. Together, these thoughts represent available knowledge and suggest possible directions for further research.

INTRODUCTION

Clean-up is one of the most critical stages in quality control of food processing operations.

A major challenge in cleaning is recognition and removal of residual soil that may resist removal by presently accepted methods of cleaning. Residual soil may nourish microorganisms that constitute potential health hazards.

Modern food processing industries have revolutionized their clean-up procedures through cleaning-in-place (CIP) and automation. Recently, advances have been significant in design and operation of CIP, understanding of various factors influencing cleaning and mechanisms of cleaning action, detergent formulations, and cleaning additives designed for specific cleaning jobs, etc. Unfortunately, progress in procedures and criteria for evaluating effectiveness of cleaning has not kept pace with growing complexities of food processing equipment and cleaning processes.

Effectiveness of cleaning is the central theme of this review. Other important topics include mechanisms of deposit formation and soil removal, factors affecting the rate of soil removal, and reuse of cleaning solutions.

CLEANING AND THE EVALUATION PROCESS

Nature of Food Plant Soil

The term "soil" may be defined in the simplest way to mean "matter out of place" (59). Soil on food processing equipment includes residues of product or one or more of its ingredients, possibly in combination with deposited water minerals (1). Such soil is subject to alterations in character depending upon food processing conditions, interaction of cleaner and soil, and subsequent contamination and microbial decomposition (84). Residues from food processing and imperfect cleaning generally are referred to as milkstone in the dairy industry (110), with similar nomenclature related to hard residues in other food industries (96). Compositions of different types of milk soils are in Table 1 (34).

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TABLE 1. Chemical composition of some milk soils (dry weight basis).^a

Composition	Cold milk soil	Hot milk soil		Milkstone	
		Swept surface heater	Tubular heater	Min.	Max.
		(%)			
Lactose	38.11	Trace	Trace	None	Trace
Fat	29.9	48.0	23.1	3.6	17.66
Protein	26.6	41.1	30.3	4.1	43.8
Ash	5.3	11.9	46.6	42.3	67.3

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Concept of Cleanliness

The concept of cleanliness as applied to food handling equipment encompasses physical, chemical, biological, and/or aesthetic considerations in developing criteria of acceptability (84). Visible residues on equipment surfaces following cleaning reflect uncleanness and a potential public health hazard. Freedom from such visible residues, however, does not indicate perfectly clean equipment as evidenced by the harborage of microorganisms even after apparently successful circulation cleaning (81). Soil that is not readily visible after inadequate cleaning generally increases with subsequent inadequate cleaning operations. The increase in soil residue may go unnoticed until it causes a serious soil problem. Several workers (65, 66, 114) have shown that even with repeated cleaning with relatively effective detergents, gradual build-up of residual soil can occur.

Food safety regulations, quality control guidelines, and most operating instructions prescribe that environment and equipment in which edibles are produced must be clean prior to each use. Hence, "clean" is probably the most frequently used word in the vocabulary of regulatory and industrial sanitarians. Experienced sanitarians commonly concede, however, that most food equipment in routine use approaches absolute cleanliness only to a degree. This is especially true if the concept of "clean" as applied to equipment surfaces is taken to mean "completely free" of soil residues from any preceding use or extraneous contamination. Hence, "relatively clean" is probably the most accurate term for description of much, if not most, food processing equipment (1).

Evaluation of Cleanliness and Measurement of Soil Deposits

In an effort to develop methods for evaluation of cleanliness of equipment, various techniques have been explored for estimating soil deposits and/or soil removal.

Visual methods. Visual appraisal of the state of cleanliness of equipment surfaces is regarded as unsatisfactory for quantitative work (59). Additionally, such visual inspections are subject to reduction in their reliability owing to the following factors (1): (a) Acuity of vision and perceptiveness in observation vary widely among individuals. (b) The intensity of lighting (natural or artificial) limits the reliability of appraisals of cleanliness. (c) Films of some product residues and even slight encrustations of milkstone are masked when equipment surfaces are wet. Few regulatory inspections involve viewing equipment when wet as well as when dry. (d) Films of components of some products (proteins, for instance) are not visually detectable readily even when product-contact surfaces are dry.

Sanitarians have developed a number of visual tests for cleanliness of equipment surfaces. Armbruster (7) listed nine such tests, primarily based on observable physical phenomena. These tests can be grouped as: (a) the water break test, in which the degree of cleanliness is indicated by the complete sheeting off of rinse water without separating into rivulets; (b) the droplet test, whereby droplets adhere to unclean surfaces; (c) the salt test utilizing salt sprinkled on wet surfaces to render more visible the adhering moisture; and (d) the carbonated water test, whereby gas bubbles adhere to soil films on unclean surfaces. Several

manufacturers of detergents determined the degree of cleanliness of equipment surfaces employing an extremely simple technique called squeegee-floodlight test. It consists of removal of clinging moisture from surfaces with a squeegee and subsequent drying of the surface by means of a 150 watt, exterior type, sealed beam floodlight. Protein film, otherwise generally undetectable visually, becomes visible when subjected to heat. Hairline cracks develop and the surface takes on the appearance of weathered aluminum (1).

In contrast to visually observable phenomena as indices of the presence of soil, a number of objective methods have been advanced for the positive determination of the presence of residual soil on washed surfaces by reproducible physical or chemical tests.

Fluorescent dye methods. Domingo (24) described a fluorochromatic technique for revealing residual soil, particularly on dishes and kitchen utensils. When the test surface was flooded with water soluble fluorescent dye and observed in darkness under ultraviolet light, residual soil which possessed fluorescent characteristics was revealed. Armbruster and Ridenour (8) devised a procedure for determining the effectiveness of washing and sanitizing of soda fountain and tavern glasses. Washed, sanitized, and drained dry glasses were dusted lightly with a mechanical mixture of talc (85%) and Safranin-O dye (15%). When wetted, the dye became red. Dusted glasses were subjected to a gentle rinse for 5 s, or until runoff was no longer red. Since the dye-impregnated talc clung tenaciously to residual organic matter on the glass, the appearance of red spots on areas of drained glasses was an index of ineffective washing. Beck (10), who employed this dye test in a study comparing findings with bacteriological swab counts on 1300 soda fountain glasses, reported the test was extremely sensitive and suitable for testing of glasses, dishes, etc. However, it was quite unsuitable for field application to stationary equipment, such as milk cooling, transportation, or storage tanks, processing vats, and pipelines. The talc-dye powder becomes air-borne easily and adheres to objects in the vicinity causing a red color problem under damp conditions.

Light-transmittance technique. Several workers (29, 61, 73, 77, 92) evaluated cleaning of glass surfaces by comparing their light transmit-

tance before and after washing. An estimation of the amount of soil removed was made using the Beer-Lambert Law,

$$R = 100(\log I_w - \log I_s)/(\log I_c - \log I_s)$$

where R = percent soil removed, I_w = light transmitted by washed surface, I_s = light transmitted by soiled surface, and I_c = light transmitted by clean surface. The primary utility of these methods was for evaluating detergency with laboratory equipment rather than monitoring surfaces of food processing equipment.

Chemical methods. Maxcy and Shahani (88) used both turbidimetric and microbiological procedures to estimate the amount of milk solids picked up by detergent solutions circulated through pipeline systems. By Kjeldahl procedure, turbidity and nitrogen content of the solution were correlated. Maxcy (81) utilized a well established phenomenon of dissipation of free chlorine by soil residues and obtained an indirect indication of otherwise undetectable soil residues in the pipeline system, both with laboratory and commercial equipment. He attributed possible harborage of microbial growth to concentration of soil in "trouble spots," such as CIP joints rather than the soil distributed uniformly throughout the pipeline system.

Microbiological methods. In conjunction with visual inspection, a bacteriological count, with or without a swab test, has been employed for evaluating cleanliness and sanitization of food processing equipment surfaces. The bacteriological swab count was devised as a means for approximating the number of residual microorganisms on surfaces which had been washed and sanitized. Less than 100 microorganisms per 52 cm² of swabbed surface was indicative of an acceptable degree of cleanliness and safety (2). However, the literature is filled with data which indicate no precise relationship between degree of cleanliness and magnitude of bacteriological swab counts of a washed and sanitized surface (10, 47). The swab count, of course, reflected only the viable bacteria on the surface at the time of testing and gave no indication of microbial growth potential or physical cleanliness of the surface.

Few microorganisms survive modern circulation cleaning processes in the dairy and food

industry. Those surviving the cleaning operation constitute the microflora for objectionable growth (83). An examination of such microflora revealed its heterogeneous nature instead of a few resistant species that might be expected (80). Since the surviving microflora was heterogeneous, it was concluded that the origin of the surviving microflora was within protected areas, such as crevices of improperly sealed gaskets, pump seals, welded metal joints of equipment, or anywhere cleaning solution could not make contact with surfaces (82).

Total microbial count of a finished product has been used to indicate microbial cleanliness of equipment in which the product was processed. However, present sanitation makes total counts as an indication of a high degree of sanitation almost meaningless (106). In packaged food products, e.g., milk (83), the total count of a freshly processed product has little relation to potential spoilage or to pathogenic organisms.

Radiological methods. Several techniques have been suggested using radioisotopes to measure either the comparative effectiveness of detergent solutions or the cleanability of equipment. Cucci (22) used radioactive phosphorus, P^{32} , in studying the removal of milk deposits from rubber, Pyrex, and Tygon tubing. Seiberling and Harper (105) reported that radioactive tracer techniques offered greater sensitivity and reproducibility for measuring soil deposits than did other methods. However, they further stated that P^{32} was unsatisfactory because it reacted irreversibly with stainless steel. More reliable results were obtained with Ca^{45} . Jennings (52, 53, 54, 55) conducted detergency studies using P^{32} -labeled milks. In view of the results by others (35, 36, 45) and based on laboratory observations, Jennings (57) concluded that there was an adsorption phenomenon between P^{32} and stainless steel. Because the P^{32} tracer generally was added as an inorganic salt, it was considered possible that it was serving as an index of inorganic residues only and might be unsatisfactory for estimating total soil deposits. The author reported, however, that comparison of in vivo and in vitro P^{32} -labeled milk indicated no difference between the two methods of adding the tracer.

Anderson et al. (3, 4) used C^{14} -labeled tristearin to study the removal of fatty soils from glass surfaces. Stearic acid C^{14} -labeled,

tristearin, triolein, and algal protein were used as soil on various surfaces by Harris and Satanek (40, 41). Bourne and Jennings (13) employed C^{14} -labeled tristearin and C^{14} -labeled sucrose in their detergency studies. Others (42, 78, 79, 97) have used encapsulated sources of radioactivity by harvesting organisms grown in a radioactive broth, incorporating these into a soil, and following the decrease in radioactivity as an index of soil removal. Peters and Calbert (97) concluded that soil containing P^{32} -labeled bacteria best indicated the rate and extent of soil removal. Milk labeled in vivo or in vitro was removed more rapidly as indicated by weight of soil removed than by removal of radioactivity. Apparently, there was an interaction between labeled phosphate and metal surfaces when the phosphate was in an inorganic form. Jennings (56), in contrast, established a highly significant correlation coefficient of .91 between soil removal and radioactivity removal for in vitro labeled milk. He attributed the discrepancies in findings to differences in the milk films used by the different investigators. In vitro P^{32} -labeled milks also have been used successfully by Pflug et al. (98).

Jennings (55) made a justified distinction between homogeneous and heterogeneous soil systems with respect to distribution of radioactivity through the deposits. He stated that at least for some heterogeneous soil species, a nonuniform distribution of radioactivity was responsible for differential rates of decrease for soil and radioactivity loads.

The usefulness of radiological test procedures is limited to laboratory equipment and model systems since radioactive materials in food processing equipment would be unacceptable (1).

Calcium residue analysis method. Heinz et al. (43) investigated a technique for determining cleanliness of milk contact surfaces by analyzing for residual calcium. Stainless steel equipment surfaces were soiled with skim milk, allowed to dry, and then subjected to normal cleaning procedures involving rinsing with water, detergent washing, and again rinsing with water. Calcium from the test surfaces was recovered by applying 1 M HCl and scraping the surface with a plastic spatula, followed by atomic absorption spectrophotometric analysis. A direct relationship between the extent of soiling and the amount of calcium recovered

from test surfaces was observed. Cleaning was said to be acceptable when the calcium concentration in the recovery solution equaled that of the control acid solution. Anderson et al. (5) evaluated comparative effectiveness of four acids (hydrochloric, nitric, phosphoric, and lactic) at two concentrations (.1 N and 1.0 N) in removing calcium from stainless steel surfaces soiled with milk. They recommended that hydrochloric acid be used in the calcium residual determination test developed by Heinz et al. (43). In a recent study on measurement of calcium in milk in the presence of major ingredients of detergents, Anderson et al. (6) found that 97.3% of the variation in spectrophotometric response was accounted for by calcium of milk. Concentration of detergent ingredients (except sodium metasilicate) and temperature of solution had little influence on calcium measurements. The analytical procedure had a sensitivity of .2 mg Ca/liter. They concluded that the atomic absorption spectrophotometer holds considerable promise for monitoring removal of residues from milk contact surfaces.

Electrical conductivity method. Ruiz et al. (102) explored the use of electrical conductivity for monitoring removal of milk residue from pipes during rinsing. No difference was significant in amounts of milk residue removed from stainless steel pipe for rinse water temperatures of 35 C or 51.7 C or flow velocities ranging from 1 to 3 m/s. The sensitivity of the method was approximately .02 mg milk solids per ml. A limiting factor was the rate of dissolution of particles physically dislodged from surfaces during cleaning since solution and ionization were necessary for detection by electrical conductivity. The studies of Fischer et al. (28) on electrical conductance of various detergents in milk-water solutions led them to conclude conductance may not be a practical indicator for milk residues in detergent solutions since more than 75% of the variability in electrical conductance resulted from detergent concentration.

Other methods. Arnold and Maxcy (9) employed gas-liquid chromatography, thin layer chromatography, and infrared spectroscopy to determine the composition of milk residue and rate of its depletion in a small commercial high-temperature short-time pasteurizer. The sensitivity achieved indicated a potential for

application of instrumental methods in evaluating cleanliness of food processing equipment.

More recently, Kulkarni (69), Maxcy, and Arnold (87) and Kulkarni et al. (70) designed a simple, convenient laboratory model of a circulation unit to simulate soiling from heat processing of milk and the flow of cleaning solution in a commercial high-temperature short-time pasteurizer. The most tenacious residue resisting circulation cleaning was characterized by gas liquid chromatography and was lipoidal in nature, similar in composition to milk fat. The extent of lipoidal residue recovered in the ether rinse of circulation cleaned equipment surfaces gave a useful and direct index for evaluating thoroughness of cleaning (69, 70). A simple, yet extremely sensitive (<one microgram) and reproducible, surface displacement technique (11, 44, 63) was applied to determine microquantities of fatty material (69, 70).

Kulkarni (69) also applied the spectrofluorometric protein assay technique of Bohlen et al. (12) based on a new reagent, fluorescamine, which enabled detection of as little as 3 μ g of proteinaceous material by simply observing fluorescence in the dark under long wavelength (366 nm) ultraviolet light. Concentration of protein in alkaline wash solution by ultrafiltration further enhanced the sensitivity of detection to less than 1 μ g. An imperfect cleaning operation is likely to leave a proteinaceous residue. Therefore, an additional alkaline wash and subsequent analysis by the simplified fluorescence technique could contribute to a workable method to evaluate thoroughness of cleaning.

Mechanisms of Deposit Formation

Role of mineral salts. Burton (20) considered deposit formation from whole milk in heat treatments as two separate processes. First, high temperature produces a condition in the milk in which some of the milk solids are no longer in true solution but are in such a state that they will either adsorb to a surface or aggregate. Then, secondly, if a surface is present, the solids which have left true solution will adsorb to it and extend to form a deposit. If a surface is not present, the solids aggregate and become unavailable for deposition at a surface. He hypothesized that reduction in solubility of milk salts at high temperature forms the first stage, followed by slow formation of crystal

nuclei and the growth of the nuclei into crystals which constitute the deposit. Reitzer (101) showed that when rate of heating was constant, a linear increase in deposit thickness occurred, provided the minerals salts, e.g., $\text{Ca}_3(\text{PO}_4)_2$, were in a supersaturated state at the heating surface. He further suggested that spontaneous nucleation necessary for crystal growth was rare where there was only a slight degree of supersaturation of minerals at the heating surface. After slow formation of sufficient nuclei, deposit formation was governed by the rate of supply of new material to continue crystal growth.

Recently, Burdett (17) showed that addition of small amounts of phosphate salts reduced the amount of deposit formed from heated milk, both in laboratory experiments and during the operation of an ultra-high-temperature heat-exchanger. Sodium pyrophosphate was much more effective than the other phosphates tested. This phenomenon was attributed to reduction in destabilization of casein micelles on heating by providing an additional source of phosphate ions.

Burton (20) stated that the sequence of supersaturation of mineral salts, nucleation, and crystal growth leading to deposit formation is also applicable to directly heated (steam injection) systems where a large quantity of precipitable material becomes available immediately after mixing of the milk with steam. Some of this material is carried to the enclosing surfaces by turbulence, and deposition occurs.

Role of proteins and fat. Proteins seem always to be constituents of deposits, even when the major component is ash. Proteins normally appear to be denatured or even degraded into nonprotein compounds (48, 89). Denaturation of soluble proteins during preheating of milk prior to ultra-high temperature (UHT) processing results in marked changes in the structure and composition of the deposit formed on UHT equipment surfaces (20). Lyster (75) analyzed deposits collected from stainless steel plates of an UHT processing plant and found materials other than protein, fat, and minerals were present to the extent of 5 to 10%. Nothing was known of their nature or possible significance. He speculated that either denatured serum proteins become adsorbed on the surface of the mineral crystallite or denatured protein might act as nuclei for crystal

growth. In further studies (76), evidence was shown for an interaction between denatured β -lactoglobulin and precipitated calcium phosphate when a solution of milk salts and β -lactoglobulin was heated to 100 C. This seemed to explain, at least in part, the intimate relation between minerals and protein in deposits from heated milk.

Protein may not necessarily increase the amount of deposit. The addition of β -lactoglobulin appeared to depress the amount of deposit formed, both in milk (18) and in an artificial milk salt system (76). On the contrary, Ito and Nakanishi (50) found that when deposits from combinations of three separate constituents, tricalcium phosphate, casein, and whey powder, were compared, tricalcium phosphate plus whey powder gave the largest amount of deposit.

Burton (19) studied deposit formation from whole milk using a hot wire laboratory apparatus and observed that the amount of deposit was not positively correlated with the mineral or protein contents of milk. However, correlation was a strong positive with fat content. He later indicated that it is unlikely that fat is closely involved in deposit formation since the amount of deposit does not vary linearly with fat content, and since it is well known that the deposit problem exists with skim milk as well as with whole milk (20). He postulated that the deposition more likely is influenced by some minor milk constituent (5) which is closely associated and varies with the fat content but is also in skim milk. A phospholipid would be such a component, as phospholipids have varied seasonally in amount in a way similar to that for deposit formation (46). Ito and his co-workers (50, 51) have shown a correlation between amount of deposit and physical properties of milk, e.g., surface tension, which might be affected by the surface active nature of phospholipid, but Ito and Nakanishi (49) were unable to detect any preferential inclusion of phospholipids in the deposits. Recent findings, however, seem to uphold the postulate of Holden et al. (46) on the possible significance of phospholipids in deposit formation. Maxcy (85) showed that repeated cursory washing and presoiling of stainless steel test surfaces with phospholipids and subsequent growth of *Pseudomonas* sp. in milk film resulted in the development of a visible yellow film. This was

judged similar to the yellowish film (commonly termed "milkstone") on improperly cleaned dairy farm utensils. Phospholipids remaining after the cursory washing of raw-milk contact surfaces were considered to contribute to accumulation of additional soil, which nurtured subsequent growth of bacteria. The function of phospholipids perhaps was related to their dipolar nature, which influenced adsorption to the stainless steel and subsequent interaction with other soil constituents. The author (85) further recognized that such yellow film milkstone of dairy farm equipment where heat processing is not involved may not bear any relationship with the milkstone associated with heated surfaces and harborages of thermophilic contaminants (27).

Studies on cleanability of milk-filmed stainless steel by chlorinated detergent solutions (62) revealed that plates pretreated with 100 ppm chlorine prior to soiling with raw milk and subsequently washed with alkaline detergent solution accumulated high build-up of soil. Alkaline solutions supplemented with up to 50 ppm chlorine gave similar results. The author attributed the soil build-up to an "adhesive nonsoluble chloro-protein" which occurred at low concentration of chlorine ions but was solubilized and lost its adhesive property at higher chlorine concentrations (75 and 100 ppm).

Energy Relations in Detergency

Jennings (59) indicated that cleanliness is an unnatural state. The spontaneous soiling process apparently results in a decrease in the free energy of the system, represented as:

$$\text{Free soil} \rightarrow \text{Deposited soil}; \Delta F = -N \text{ calories.}$$

The negative value of ΔF (free energy) means that some energy is released during soil deposition. To reverse this process and clean the surface, it becomes necessary to supply energy to the system.

Several researchers have attempted to account for the forces required to remove a soil from a surface by summing the repulsive and attractive forces known to act on a discrete particle, based on the theory of the stability of lyophobic colloids developed by Derjagvim (23). Durham (25) showed that a soiled particle must pass an energy barrier in leaving the surface of a fiber. Lange (71, 72) demonstrated

that both the lowering of the interfacial potential by a detergent and increasing the ionic strength of the detergent solution decreased the energy barrier. Goette (31) showed that the energy necessary to remove a particle increases with decreasing particle size. He also emphasized the zeta potential as an important factor in soil removal. Harris (37) subsequently supported the concept of importance of the zeta potential.

Excellent reviews by Harris (39) and Jennings (57, 59) include detailed considerations of energy relationships in detergency as applied to textile cleaning and hard surface cleaning, respectively.

Soil and Substrate Characteristics

Niven (91) reviewed the topic of soil and surface properties in relation to the soiling process. Jennings (59) explained that the manner in which a soil is attached to a surface or substrate is related to the physical and chemical characteristics of both the soil and the substrate. Substrate characteristics of importance include smoothness, surface finish, porosity, hardness, wettability by a liquid soil component, and chemical reactivity with the soil. Important soil characteristics include particle size, viscosity, surface tension or wetting power, mutual solubility of soil constituents, and chemical reactivity with the substrate. Ginn et al. (30) stated that the extent of attraction between the deposited soil and the substrate is probably influenced by the polarity of both.

Masurovsky and Jordan (78, 79) demonstrated that the number of radiophosphorus-labeled bacteria that existed on surfaces after ultrasonic cleaning was influenced by the surface. Teflon showed the least retention and borosilicate glass, polyethylene plastic, and stainless steel showed progressively greater retentions. They also reported that retention after cleaning was lower for highly polished surfaces than for coarser finishes. Puelo et al. (100) showed ultrasonic energy was more reliable and efficient than mechanical agitation for recovering and assessing microbial contaminants from surfaces of various finishes. However, Kaufman et al. (65, 66) working with bacterial counts on stainless steel of various finishes found that surface finish exerted no effect on bacterial cleanability. Pflug et al. (98) studied the effect of stainless steel finish on the

deposition and removal of a radioactive milk soil. They concluded that, although surface finish seemed to exercise a slight but measurable influence on rate of soil deposition, rate and extent of soil removal were not affected.

Soil may be attached to the substrate mechanically by entrapment in surface irregularities or voids, held with cohesive or wetting forces, or bonded by chemical action or adsorptive forces. Many particulate soils are held to surfaces by oil or fat deposits (59). With respect to mechanical entrapment of soil, Jennings et al. (60) felt that it possibly could have an adverse effect on cleaning only where the surface is sufficiently irregular to create highly localized minute areas of low turbulence.

Soil Removal Mechanisms

Bourne and Jennings (16) stated, "The mechanism of detergent action in soil removal has been attributed by different research workers to many factors, including surface tension, interfacial tension, contact angle, surface viscosity, lather, electrostatic properties, solubilization, adsorption, cryoscopy, wetting, suspending action, emulsification, saponification and lubrication." However, the relevance of cryoscopy as a mechanism of soil removal is difficult to comprehend. Jennings (57, 59) has extensively reviewed the literature in this field with considerations to both homogeneous and heterogeneous soil systems.

Fundamental studies involving model systems of homogeneous soils on smooth homogeneous surfaces, as in removal of oily or particulate soil from textile fibers or glass surfaces, have helped gain considerable understanding of detergency processes and soil removal mechanisms. However, most food handling equipment involves heterogeneous soil deposits, frequently containing fats, proteins, carbohydrates, gummy residues, misplaced mineral oils and greases, particles of products such as milk, meat, egg, fruit or vegetable residues, occasionally complicated by mineral incrustations. Further, for the removal of such heterogeneous soil, use of a formulated detergent containing several ingredients commonly is recommended. Thus, the cleaning process is complex. Jennings (59) stated that the role of an individual fraction in displacing and stabilizing a heterogeneous soil is affected by and inter-

related with roles played by other detergent fractions.

In a study of the kinetics of detergent synergism, Jennings (58) pointed out that the relative contribution of a given detergent fraction probably varies with the nature of the deposit under consideration at a given point and a given time. He pointed out that detergency studies on heterogeneous soils represent complicated and interrelated phenomena and the true mechanism of action remains a sketchy picture. Harper (34) viewed the fundamental phenomena involved in the cleaning process in terms of a series of four essential steps: (a) bringing the detergent solution into intimate contact with the soil to be removed by means of good wetting and penetrating properties; (b) displacement of solid and liquid soils from the surface to be cleaned by saponifying of fat, peptizing the proteins, and dissolving the minerals; (c) dispersion of the soil in the solvent by dispersion, deflocculation, or emulsification; and (d) prevention of redeposition of the dispersed soil back onto the clean surface by providing good rinsing properties.

Using a single pass alkaline cleaning system with a small commercial high-temperature short-time pasteurizer, Arnold and Maxcy (9) found that saponification apparently was not involved in the initial removal of lipids from equipment surfaces since the lipids occurred in a cleaning solution as neutral fats rather than as fatty acids. They attributed the differences in conclusions to the use of a single pass cleaning system and further stated that in a common recirculation system involving prolonged exposure to alkaline solution, high temperature and agitation, some fat may saponify after removal from surfaces.

Maxcy and Arnold (87) studied saponification under conditions comparable to common chemical reactions in cleaning processes. Various concentrations of sodium hydroxide with a .5% suspension of milk fat were held at 77 C in a laboratory shaker providing constant, gentle agitation. The degree of saponification was determined by titration. The results showed that saponification was dependent on time and alkali concentration. Only approximately 19% of the fat was saponified in 5% sodium hydroxide at 77 C in 4 h; with 1% sodium hydroxide less than 1% of the fat was saponified in 1 h. From these data, the authors concluded that

saponification during CIP operations is of minor influence in cleaning, therefore, indicating an explanation of the phenomenon of removal of lipoidal material from surfaces must be sought in another mechanism(s).

Factors Affecting Rate of Soil Removal

A number of variables have been recognized which influence the rate of the cleaning process. Considerable work has been directed to establish their relative importance and to develop means for the evaluation of these variables (59). This work is discussed in further detail in relation to various factors.

Effect of age (time of contact between soil and surface). Cleaning recommendations frequently emphasize that surfaces should be cleaned as soon after soiling as possible. The aging phenomenon and increasing difficulty in soil removal generally are recognized. In cleaning of textile fibers with soap solutions, aging effect of oil-containing soil was attributed to the presence of moisture and polymerization of unsaturated oils (115). Durham (26) commented that "... it is well known that when fat is included in the dirt, the longer the fabric is stored before washing the more difficult it is to clean." Oxidation and/or polymerization of unsaturated oils was thought to be a causative factor. Anderson et al. (4), in studying removal of tristearin from frosted glass, observed aging effect and suggested that it was due to a layer of moisture on the aged discs. However, similar studies (13) on stainless steel ruled out the effects of moisture or polymerization and oxidation since stainless steel does not swell in water and tristearin does not polymerize or oxidize, being a fully saturated fat. Evidence by Bourne and Jennings (14) indicates that tristearin on stainless steel exists in two chemically identical forms that are removed at different rates and that the aging effect involves transition of the fast-removed soil species to the more slowly removed tenacious form.

Effect of cleaning time. Jennings (59) stated that it was perhaps misleading to consider "time" as if it were an independent variable of the cleaning process; time itself makes no contribution. Time, therefore, should be considered in relation to other variables such as detergent concentration, temperature, etc. Seldom can soil removal be considered a spontane-

ous process. Normally, most soil removal mechanisms involve a finite time, and the longer the solution is in contact with the deposited soil under a given set of conditions, the more soil is removed. This reaches a limit because the final traces of soil probably are never removed, and we may attain a state where soil redeposition occurs as rapidly as soil removal.

The two cleaning mechanisms described by Bourne and Jennings (13) in the removal of tristearin from stainless steel by sodium hydroxide involved the concept of two distinct soil species that differed in rate or ease of removal. The first mechanism was time-dependent and was termed the "flow effect." The second, and more powerful, was time-independent. This latter required sweeping of the soiled surface with an air-solution interface and was termed the "Dupre effect."

Effect of type of detergent. Harris (35, 36, 38) pointed out that one of the primary functions of a detergent is to minimize the soil-substrate attractive forces by adsorption on soil and substrate. One school of thought defines "detergent" as "anything that removes soil" (59). Bourne and Jennings (15) proposed that a detergent be defined as "any substance that, either alone or in a mixture, reduces the work requirement of a cleaning process." They indicated this definition is consistent with the commonly accepted meaning of the word "detergency." It specifies no special chemical group of compounds, includes soap and other surface-active compounds that possess power of detergency, and excludes those that do not. It includes nonsurface-active detergents (e.g., sodium hydroxide), synergistic substances (e.g., polyphosphates), and materials that inhibit redeposition (e.g., carboxymethyl cellulose). It includes solvents and cleaners that degrade soil (e.g., acids) since these reduce the work requirement to zero. It, however, excludes abrasives since these affect the efficiency with which work is applied without actually reducing the work requirement in the cleaning operation.

Several workers have attempted to evaluate various types of detergents (29, 32, 61, 62, 74, 89, 94). Good general discussions pertaining to commonly used detergent chemicals and considerations on their usages are given by Schwartz and Perry (103), Schwartz et al. (104), and in a review by Swartling (112). Harris and Sataneek (40) found that surface

active agents were more effective in removing fatty soils than protein deposits. Jennings (58) pointed out that the role of an individual fraction of detergent mixture in displacing and stabilizing a heterogeneous soil is affected by and interrelated with roles played by other fractions and probably varies with the nature of the deposit under consideration at a given point and a given time.

White and Rabe (116) evaluated the use of nitric acid as a detergent in a model CIP system using an atomic absorption method for detergency measurement. Their results indicated that satisfactory cleaning of stainless steel surfaces soiled with milk can be achieved with low concentrations of nitric acid. Critical concentration and temperature combinations were observed.

Effect of detergent concentration. Tachibana et al. (113) studied beef tallow removal from cotton cloth in a launderometer using sodium dodecyl sulfate as a detergent. In plotting soil removed versus detergent concentration, they found a maximum beyond which higher detergent concentration removed less soil. This result agrees with that of Palmer and Rideal (93) but is in conflict with many other studies (40, 41, 64, 67) which shows that detergency increases to a limit and does not decrease at higher detergent concentrations. This is consistent with the theory that a detergent reduces the forces holding a thin film to a surface but has no further influence above a concentration representing some kind of saturation effect. Jennings (59) indicated that such saturation effects may not be involved with nonsurface active detergent materials such as sodium hydroxide or strong acid cleaners. Working at 46 C and a Reynolds number of 550,000, Jennings (53) studied the rate of removal of radio-labeled milk films from stainless steel by solutions of .01, .03, .05, .10, and .15 M hydroxide ion. The soil removal process was kinetically first-order with respect to hydroxide ion as well as soil concentrations. He used the equation:

$$-ds/dt = K(S)(\overline{OH})$$

where S = soil as percent of original deposit, \overline{OH} = hydroxide ion concentration, K = molar velocity constant, and t = time.

Effect of temperature. Opinions are conflicting regarding the effect of temperature on soil

removal. Some imply that high cleaning temperatures encourage the "burning-on" of residual soils, while others contend that higher temperatures yield better cleaning. Recommendations of detergent manufacturers and equipment manufacturers range from 46 to 82 C (57). Calbert (21), using a laboratory circulation unit, reported that pipelines soiled with milk solids could be cleaned by starting with washed solution temperatures of 54 to 60 C without concern for temperature drop during the washing cycle. Smith (107) recommended 49 C for cold-wall milk storage tanks and 77 C for plate heat exchangers.

Parker et al. (95) reported that higher temperatures yielded more effective cleaning, but Jones (64) stated that temperature has little effect on detergency between about 45 C and just below the boiling point of water. Below 45 C, a decrease in efficiency is expected as fatty material below its melting point is more difficult to remove. These and other contradictions have been ascribed to the use of ill-defined systems, unsatisfactory methods of experimental evaluations of detergent efficiency, or failure to differentiate between effect of temperature on soil removal and effect of temperature on the detergent form contributing to soil removal (59).

Jennings (53) demonstrated that under experimentation, removal of cooked-on milk films by solutions of sodium hydroxide exhibited a Q_{10} of 1.6 within the temperature range 46 to 82 C, i.e., the rate of soil removal increased by a factor of 1.6 for every 10 C rise in temperature. Increasing the temperature of circulating fluid presumably continues to increase cleaning efficiency until a point is reached where the detergent decomposes or the vapor pressure of the detergent fluid interferes with the operation. Ginn et al. (30) reported that solubilization of fatty soils may be one of the main, if not the controlling factor, in the mechanism of soil removal by nonionic surface active detergents. They also demonstrated that the critical micelle concentration and solubilizing power are affected by solution temperature.

Jennings (57) concluded that the use of kinetic theory, where applicable, is the only approach that will permit measurement and prediction of the effect of temperature (or any other controllable variable) on the cleaning operation with a high degree of precision and

reliability.

Effect of air, foam, and turbulence. Several milking-machine manufacturing companies advise cleaning teat cups and claw assemblies by sucking detergent solution through them and lifting them occasionally to permit an air gulp (57). Jennings (52, 55) found that air leaks contributed to cleaning, and cleaning was more effective at a reduced pressure. Merely repositioning the circulation pump to pull, rather than push, the cleaning medium through the area increased the cleaning action. These conclusions were valid only in the absence of foam formation since a buildup of foam created a cushion against the scrubbing action that is responsible for at least part of the increased efficiency. Sisley (107) noted that antifoams added to wash solutions effectively eliminated suds without affecting detergency. With soaps, lather serves only as an indicator that sufficient soap is present in the solution to overcome losses by precipitation of calcium soaps, reactions with soil or adsorption to soil and substrate, and that foam plays no discernible role in detergency (111).

In circulation cleaning and spray cleaning, the friction between deposited soil and fluid flowing past it provides the energy source essential for final displacement of the soil deposit (57). Shear force so generated is related to turbulence of the solution. Phillips (99) reported that increased rates of circulation resulted in more efficient cleaning of milking machines. Jennings et al. (60), working with P^{32} -labeled films of milk solids, concluded that the effect of turbulence is negligible until the rate of flow is high enough to achieve a Reynolds number of 25,000. The effect of turbulence becomes less pronounced as the temperature is increased or more effective detergents are used. The recommendation of Smith (108) that the cleaning medium be circulated at a velocity of 1.72 m/s is based on turbulence requirement. In a 3.8 cm diameter pipeline with a velocity of flow of 1.72 m/s, a Reynolds number of approximately 100,000 is achieved. Hankinson et al. (33) stated that Reynolds number is a better basis of requirement for fluid flow characteristics than the centimeters/second flow rate specifications. They also presented charts and the following formula in explaining the utility of this dimensionless number:

$$N_{Re} = LV/v$$

where N_{Re} = Reynolds number, L = diameter of pipe in centimeters, V = velocity in centimeters/second, and v = kinematic viscosity in centimeters²/second. They indicated turbulent flow occurs when N_{Re} is greater than 3000, and laminar flow occurs when N_{Re} is less than 2000. The transition zone is between these two flow rates. Other literature (68) indicates the transition zone may extend to an N_{Re} of approximately 10,000 when highly viscous products are involved.

The increased cleaning efficiency of air was long attributed to an increased scrubbing action or turbulence due to slugs of entrained air. However, Bourne and Jennings (13) observed that something more was involved. Studying the removal of thin films of C^{14} -labeled tristearin from stainless steel by sodium hydroxide, they found two soil removal mechanisms, one time-dependent and the other independent of time. They demonstrated that under their experimental conditions, most of soil removal was accomplished by the latter mechanism which they termed "Dupre effect" (13, 16). This mechanism, which required wiping of the deposited soil by the air-solution interface (advancing or receding), was most effective in the early stages of cleaning. After some minutes of cleaning, photomicrographs showed that the fatty soil had agglomerated, a state in which it was highly resistant to removal by the Dupre effect.

Reuse of Cleaning Solutions

Smith and Hedrick (109) stated, "Commercial experience has indicated that CIP solutions may be reused for cleaning soiled equipment, especially with surfaces that have no 'burn-on' milk deposits but the conditions for satisfactory reuse and specific limitations are vague." Based on visual and bacteriological criteria for evaluating cleanliness of surfaces, extensive reuse of chlorinated alkali cleaning solution for milk plant equipment involving nonheated surfaces was satisfactory (109). When spent alkaline cleaning solutions were reused, a progressive depletion occurred in the cleaning ability of the reused solutions as evidenced by an incomplete removal of soil from the equipment surfaces (69, 70).

Recently, Maxcy and Arnold (86) demonstrated that alkaline solutions can be freed

effectively of milk fat by sorption on Celite and indicated a potential technique to extend the usefulness of cleaners. By this method, the spent cleaning solutions were regenerated successfully resulting in a significant restoration of lost cleaning ability (69, 70). Economic and environmental implications of regenerating used solutions were discussed.

Redeposition of Soil

Anderson et al. (3) defined redeposition as readsorption of soil which had been removed in the cleaning process. They encountered a redeposition phenomenon in a system involving radiolabeled tristearin soil and glass surfaces. Redeposition of lipoidal material onto stainless steel surfaces was observed when spent cleaning solutions were circulated through previously cleaned equipment (69, 70). Jennings (59) postulated that in most cleaning operations, the final traces of soil probably are never removed, and a state may be attained where soil deposition occurs as rapidly as soil removal. Anti-redeposition agents more commonly are used in built detergents intended for textiles than in hard-surface cleaners. They are usually hydrophilic colloids that form apparently clear solutions. Sodium carboxymethyl cellulose is an example of a common commercial anti-redeposition agent.

CONCLUSION

Cleaning is a complex, challenging process. Soil residue, water, equipment, and cleaning regimen are unique for each processing operation. The process is confounded by lack of precise methods for evaluating the effectiveness of cleaning. The goal of scientists in the dairy food industry is to apply appropriate methods both in cleaning processes and evaluation systems.

The personal view commonly is based on historical concepts that a visibly clean surface is satisfactory. Modern technology, however, involves equipment that is not routinely accessible for visible inspection. Even more importantly, modern processing and distribution systems require a bacteriologically clean surface. The degree of cleanliness which must be attained remains a question, and demands change with socio-economic development. Common practice is to provide a satisfactory

cleaning process and add a factor of safety. Determining and defining an adequate safety factor would be a major contribution to the field of sanitation.

The difficulty in developing an acceptable test for cleanliness that is applicable to modern CIP systems may exemplify the extreme degree of effectiveness of common cleaning operations. It is nearly impossible to quantitate such infinitesimally small amounts of residual soil.

This review of literature suggests many potentially favorable directions for future research. An example is the application of available laboratory data to commercial systems to determine rate of cleaning and the endpoint of cleaning processes. Studies of the extension of the utility of cleaning solutions might reduce waste of cleaners and reduce the problem of sewage disposal. Our laboratory data indicate some potentially useful techniques for determining microquantities of lipoidal material in alkaline cleaning solutions. These techniques should be useful in studying commercial systems. Usefulness of visual inspection might be increased by further development and application of fluorescence techniques. One of the greatest needs is a precise system of evaluation of the accomplishments in a cleaning process so that an acceptable endpoint can be determined during routine operation.

While awaiting the application of the above techniques and the development of others, industry should apply science when it can and art when it must.

REFERENCES

- 1 Abele, C. A. 1965. Needed: A reliable field determinant of cleanliness. *J. Milk Food Technol.* 28:257.
- 2 American Public Health Association. 1972. Standard methods for the examination of dairy products. 13th ed. Amer. Public Health Ass., NY.
- 3 Anderson, R. M., J. Satanek, and J. C. Harris. 1959. Removal of fatty soil from glass. Solvent system mechanism. *J. Amer. Oil Chem. Soc.* 36:286.
- 4 Anderson, R. M., J. Satanek, and J. C. Harris. 1960. Removal of fatty soil from glass. Electrolyte detergent builder effect. *J. Amer. Oil Chem. Soc.* 37:119.
- 5 Anderson, M. E., J. R. Fischer, R. T. Marshall, D. B. Brooker, and T. F. Webb. 1972. Comparative effectiveness of various acids in removing calcium from milk films on stainless steel. *J. Milk Food Technol.* 35:325.
- 6 Anderson, M. E., D. B. Brooker, J. R. Fischer, E.

- L. Ruiz, and R. T. Marshall. 1973. Measurement of calcium of milk by atomic absorption spectrophotometry in the presence of major ingredients of detergents. *J. Milk Food Technol.* 36:554.
- 7 Armbruster, E. H. 1962. Evaluation of surface contamination. *The Sanitarian*, July–August, 1962.
- 8 Armbruster, E. H., and G. M. Ridenour. 1960. Field test procedure for cleanliness measurement of multiple-use eating utensils. *The Sanitarian*, September–October, 1960.
- 9 Arnold, R. G., and R. B. Maxcy. 1970. Evaluating circulation cleaning by analysis of soil depletion from surfaces. *J. Dairy Sci.* 53:1540.
- 10 Beck, W. J. 1962. The antiquity of modern methodology. *Ass. of Food and Drug Officials of the U. S.*, April, 1962.
- 11 Bergquist, D. H., and F. Wells. 1956. The monomolecular surface film method for determining small quantities of yolk or fat in egg albumen. *Food Technol.* 10:48.
- 12 Bohlen, P., S. Stein, W. Dairman, and S. Udenfriend. 1973. Fluorometric assay of proteins in the nanogram range. *Arch. Biochem. Biophys.* 155:213.
- 13 Bourne, M. C., and W. G. Jennings. 1961. Some physicochemical relationships in cleaning hard surfaces. *Food Technol.* 15:495.
- 14 Bourne, M. C., and W. G. Jennings. 1963. Existence of two species in detergency investigations. *Nature* 197:1003.
- 15 Bourne, M. C., and W. G. Jennings. 1963. Definition of detergent. *J. Amer. Oil Chem. Soc.* 40:212.
- 16 Bourne, M. C., and W. G. Jennings. 1963. Kinetic studies of detergency. II. Effect of age, temperature and cleaning time on rates of soil removal. *J. Amer. Oil Chem. Soc.* 40:523.
- 17 Burdett, M. 1974. The effect of phosphates in lowering the amount of deposit formation during the heat treatment of milk. *J. Dairy Res.* 41:123.
- 18 Burton, H. 1964. Page 109 in *Rep. Nat. Inst. Res. Dairy*.
- 19 Burton, H. 1967. Seasonal variation in deposit formation from whole milk on a heated surface. *J. Dairy Res.* 34:137.
- 20 Burton, H. 1968. Deposits from whole milk in heat treatment plant—a review and discussion. *J. Dairy Res.* 35:317.
- 21 Calbert, H. E. 1958. The influence of water temperature and hardness on the cleaning of CIP farm milk pipelines. *J. Milk Food Technol.* 21:12.
- 22 Cucci, M. W. 1954. The use of radioactive phosphorus to measure the amounts of milkstone deposited on rubber, pyrex glass and tygon tubings. *J. Milk Food Technol.* 17:332.
- 23 Derjagvim, B. 1940. On the repulsive forces between charged colloid particles. *Trans. Faraday Soc.* 36:203, 730.
- 24 Domingo, E. 1950. Fluorochromatic method for organic matter on dishware. *Proc. 37th Ann. Meeting, Chem. Specialties Mfg. Ass., Inc.*, NY.
- 25 Durham, K. 1956. The role of interfacial electrical conditions in detergency. *J. Appl. Chem.* 6:153.
- 26 Durham, K. 1961. Surface activity and detergency. Macmillan Publishing Co., London.
- 27 Elliker, P. R. 1949. Practical dairy bacteriology. 1st ed. McGraw-Hill Book Co., Inc., NY.
- 28 Fischer, J. R., D. B. Brooker, M. E. Anderson, R. T. Marshall, and E. L. Ruiz. 1973. Electrical conductance of various detergent ingredients in milk-water solutions. *J. Dairy Sci.* 56:1405.
- 29 Gilcreas, F. W., and J. E. O'Brien. 1941. Laboratory studies of methods for cleansing of eating utensils and evaluating detergents. *Amer. J. Public Health* 31:143.
- 30 Ginn, M. E., E. L. Brown, and J. C. Harris. 1961. Solubilization of fatty soils by a radiotracer technique. *J. Amer. Oil Chem. Soc.* 38:361.
- 31 Goette, E. K. 1949. Theoretical considerations of detergency. *J. Colloid Sci.* 4:459.
- 32 Griffin, W. C. 1954. Calculation of HLB values for nonionic surfactants. *J. Soc. Cosmet. Chem.* 5(4):1.
- 33 Hankinson, D. J., C. E. Carver, K. P. Chong, and K. P. Gordon. 1965. Fluid flow relationships of importance in circulation cleaning. *J. Milk Food Technol.* 28:377.
- 34 Harper, W. J. 1972. Sanitation in dairy food plants. Page 130 in *Food sanitation*. R. K. Guthrie, ed. The AVI Publishing Co., Inc., Westport, CT.
- 35 Harris, J. C. 1948. Detergent adsorption on soil and substrate. *Soap Chem. Spec.* 24(11):40.
- 36 Harris, J. C. 1958. Detergent adsorption on soil and substrate. *Soap Chem. Spec.* 34(12):59.
- 37 Harris, J. C. 1958. Electrical forces affecting soil and substrate in the detergency process zeta potential. *Text. Res. J.* 28:912.
- 38 Harris, J. C. 1959. Detergent adsorption on soil and substrate. *Soap Chem. Spec.* 35(1):49.
- 39 Harris, J. C. 1961. Forces in detergency. *Soap Chem. Spec.* 37(5):68; (6):60; (7):53.
- 40 Harris, J. C., and J. Satanek. 1961. Removal of radiotagged protein and stearic acid soil from glass. *J. Amer. Oil Chem. Soc.* 38:169.
- 41 Harris, J. C., and J. Satanek. 1961. Radiotagged soils—removal from several substrates and adsorption site character. *J. Amer. Oil Chem. Soc.* 38:244.
- 42 Hays, G. L., J. D. Burroughs, and D. H. Johns. 1958. The cleanability of materials in contact with dairy products. *J. Milk Food Technol.* 21:68.
- 43 Heinz, J. V., R. T. Marshall, and M. E. Anderson. 1967. Determining cleanliness of milk contact surfaces by analyzing for calcium residual: Preliminary studies. *J. Milk Food Technol.* 30:337.
- 44 Heinemann, B., and M. R. Rohr. 1950. A micro-method for routine determinations of fat in skimmilk and nonfat dry milk solids. *J. Dairy Sci.* 33:703.
- 45 Hensley, J. W., A. O. Long, and J. E. Willard. 1949. Reactions of ions in aqueous solution with glass and metal surfaces. *Ind. Eng. Chem.* 41:1415.
- 46 Holden, T. F., N. C. Aceto, E. S. Dellamonica, and M. J. Calhoun. 1966. Seasonal variation of

- phospholipids and their influence on the foaming characteristics of concentrated whole milk. *J. Dairy Sci.* 49:346.
- 47 Holland, R. F., J. D. Shaul, D. A. Theokas, and H. M. Windlan. 1953. Cleaning stainless lines in place. *Food Eng.* 25(5):75.
 - 48 Ito, R., and T. Nakanishi. 1964. *Jap. J. Dairy Sci.* 13:A202 (cited in *J. Dairy Res.* 35:317).
 - 49 Ito, R., and T. Nakanishi. 1966. *Jap. J. Dairy Sci.* 15:A69 (cited in *J. Dairy Res.* 35:317).
 - 50 Ito, R., and T. Nakanishi. 1967. *Jap. J. Dairy Sci.* 16:A62 (cited in *J. Dairy Res.* 35:317).
 - 51 Ito, R., M. Sato, and M. Suzuki. 1963. *Jap. J. Dairy Sci.* 12:A165 (cited in *J. Dairy Res.* 35:317).
 - 52 Jennings, W. G. 1959. Circulation cleaning. II. Effects of entrained air. *J. Dairy Sci.* 42:476.
 - 53 Jennings, W. G. 1959. Circulation cleaning. III. The kinetics of a simple detergent system. *J. Dairy Sci.* 42:1763.
 - 54 Jennings, W. G. 1959. Effective in-place cleaning. *Food Eng.* 31(10):98.
 - 55 Jennings, W. G. 1960. Effect of pressure on circulation cleaning. *Food Technol.* 14:591.
 - 56 Jennings, W. G. 1961. A critical evaluation of in vitro radioactive phosphorus additions for estimating soil deposits. *J. Dairy Sci.* 44:258.
 - 57 Jennings, W. G. 1963. An interpretive review of detergency for the food technologist. *Food Technol.* 17(7):53.
 - 58 Jennings, W. G. 1963. A kinetic approach to detergent synergism. *J. Amer. Oil Chem. Soc.* 40:17.
 - 59 Jennings, W. G. 1965. Theory and practice of hard-surface cleaning. *Adv. Food Res.* 14:325.
 - 60 Jennings, W. G., A. A. McKillop, and J. R. Luick. 1957. Circulation cleaning. *J. Dairy Sci.* 40:1471.
 - 61 Jensen, J. M. 1946. Measuring detergency functions as affected by various detergents and procedures against milk films by application of a mechanical washing apparatus. *J. Dairy Sci.* 29:453.
 - 62 Jensen, J. M. 1970. Cleanability of milk-filmed stainless steel by chlorinated-detergent solution. *J. Dairy Sci.* 53:248.
 - 63 Jones, K. K. 1950. A micromethod for fat analysis based on formation of monolayer films. *Science* 111:9.
 - 64 Jones, T. G. 1961. *In Surface activity and detergency.* K. Durham, ed. Macmillan Publishing Co., London.
 - 65 Kaufmann, O. W., T. I. Hedrick, I. J. Pflug, and C. G. Pheil. 1960. Relative cleanability of various finishes of stainless steel in a farm bulk tank. *J. Milk Food Technol.* 23:377.
 - 66 Kaufmann, O. W., T. I. Hedrick, I. J. Pflug, C. G. Pheil, and R. A. Keppeler. 1960. Relative cleanability of various stainless steel finishes after soiling with inoculated milk solids. *J. Dairy Sci.* 43:28.
 - 67 Kling, W., and H. Lange. 1955. Zur Energetik des Waschvorganges bei oligen Anschmutzungen. *Kolloid Z.* 142:1.
 - 68 Knudsen, J. G., and D. L. Katz. 1958. Fluid dynamics and heat transfer. McGraw-Hill Book Co., Inc., NY.
 - 69 Kulkarni, S. M. 1974. Microcontaminants of food processing equipment. Ph.D. Thesis, University of Nebraska, Lincoln.
 - 70 Kulkarni, S. M., R. G. Arnold, and R. B. Maxcy. 1975. Reuse limits and regeneration of solutions for cleaning dairy equipment. *J. Dairy Sci.* 58:1095.
 - 71 Lange, H. 1957. Über die energetische Wechselwirkung zwischen Textilfasern und Pigmentschutz beim Waschprozess (I). *Kolloid Z.* 154:103 (cited in *Adv. Food Res.* 14:325).
 - 72 Lange, H. 1958. Über die energetische Wechselwirkung zwischen Textilfasern und Pigmentschutz beim Waschprozess (II). *Kolloid Z.* 156:108 (cited in *Adv. Food Res.* 14:325).
 - 73 Leenerts, L. O., J. F. Pietz, and J. Elliott. 1956. Soil removal by dishwashing detergents. *J. Amer. Oil Chem. Soc.* 33:119.
 - 74 Levi, S. M., and O. K. Smirnov. 1959. The relation between the structure of surface active substances and their adsorption properties (in Russian). *Colloid J. Acad. Sci. U. S. S. R.* 21:315 (cited in *Adv. Food Res.* 14:325).
 - 75 Lyster, R. J. L. 1965. The composition of milk deposits in an UHT plant. *J. Dairy Res.* 32:203.
 - 76 Lyster, R. J. L. 1966. Page 100 in *Rep. Nat. Inst. Res. Dairy* (cited in *J. Dairy Res.* 35:317).
 - 77 Mann, E. H., and C. C. Ruchhoft. 1946. A performance test for rating dishwashing detergents. *U. S. Public Health Rep.* 61:877.
 - 78 Masurovsky, E. B., and W. K. Jordan. 1958. Studies on the relative bacterial cleanability of milk-contact surfaces. *J. Dairy Sci.* 41:1342.
 - 79 Masurovsky, E. B., and W. K. Jordan. 1960. Studies on the removal of *Staphylococcus aureus* from milk-contact surfaces by ultrasonic cleaning methods. *J. Dairy Sci.* 43:1545.
 - 80 Maxcy, R. B. 1964. Potential microbial contaminants from dairy equipment with automated circulation cleaning. *J. Milk Food Technol.* 27:135.
 - 81 Maxcy, R. B. 1966. The relationship between residual soil and microbial growth after circulation cleaning. *Food Technol.* 20:123.
 - 82 Maxcy, R. B. 1966. Some relative effects of surface active agents and dissolved substances on microorganisms of importance in sanitation. *J. Environ. Health* 29:121.
 - 83 Maxcy, R. B. 1967. Nature and growth response of the microflora of pasteurized, packaged milk. *J. Milk Food Technol.* 30:213.
 - 84 Maxcy, R. B. 1967. Measure of concepts and challenges for cleanliness. *Food Technol.* 22:24.
 - 85 Maxcy, R. B. 1973. Nature and cause of yellow film occurring on dairy equipment. *J. Dairy Sci.* 56:164.
 - 86 Maxcy, R. B., and R. G. Arnold. 1973. Mechanism of removal of lipoidal residues from equipment by alkaline cleaning materials. *J. Dairy Sci.* 56:632. (Abstr.)
 - 87 Maxcy, R. B., and R. G. Arnold. 1974. Characteri-

- zation of tenacious residue of alkaline circulation cleaning. *J. Milk Food Technol.* 37:469.
- 88 Maxcy, R. B., and K. M. Shahani. 1960. Evaluation of circulation cleaning of welded pipelines. *J. Dairy Sci.* 43:856. (Abstr.)
 - 89 Merrill, E. P., J. M. Jensen, and S. T. Bass. 1962. Detergency effects of trisodium phosphate with and without sodium hypochlorite on milk-protein soils. *J. Dairy Sci.* 45:613.
 - 90 Nakanishi, T., and R. Ito. 1966. A study on milk deposit formation on heat exchange surfaces in the UHT system. 17th Int. Dairy Congr., Munich B:613.
 - 91 Niven, W. W. 1955. Industrial detergency. Reinhold Publ., NY.
 - 92 Norris, F. I., and C. C. Ruchhoft. 1948. Some improvements in the performance test for rating dishwashing detergents. U. S. Public Health Rep. 63:97.
 - 93 Palmer, R. C., and E. K. Rideal. 1939. Adhesion in detergence. *J. Chem. Soc.* (No volume number):573.
 - 94 Parker, M. E. 1943. Acid detergents in food sanitation. *Ind. Eng. Chem.* 35:100.
 - 95 Parker, R. B., P. R. Elliker, G. T. Nelson, G. A. Richardson, and G. H. Wilster. 1953. Cleaning pipelines in place. *Food Eng.* 25(1):82.
 - 96 Parker, M. E., and J. H. Litchfield. 1962. Food plant sanitation. Reinhold Publ. Corp., NY.
 - 97 Peters, J. J., and H. E. Calbert. 1960. Use of P^{32} in the study of the cleanability of milk handling surfaces. *J. Dairy Sci.* 43:857. (Abstr.)
 - 98 Pflug, I. J., T. I. Hedrick, O. W. Kaufmann, R. A. Keppeler, and C. G. Pheil. 1961. Studies on the deposition and removal of radio-active soil. *J. Milk Food Technol.* 24:390.
 - 99 Phillips, D. S. M. 1958. Recent developments in automatic cleaning of storage tanks. *N. Z. J. Agr.* 97:537.
 - 100 Puelo, J. R., M. S. Favero, and N. J. Peterson. 1967. Use of ultrasonic energy in assessing microbial contamination of surfaces. *Appl. Microbiol.* 15:1345.
 - 101 Reitzer, B. J. 1964. Rate of scale formation in tubular heat exchangers. *Ind. Eng. Chem. Process Des. Dev.* 3:345.
 - 102 Ruiz, E. L., D. B. Brooker, M. E. Anderson, and R. T. Marshall. 1972. Use of electrical conductivity for monitoring milk residue removal from pipes during rinsing. *J. Milk Food Technol.* 35:257.
 - 103 Schwartz, A., and J. Perry. 1949. Surface active agents. Interscience Publ., NY.
 - 104 Schwartz, A., J. Perry, and J. Berch. 1958. Surface active agents and detergents. Vol. II. Interscience Publ., NY.
 - 105 Seiberling, D. A., and W. J. Harper. 1956. Evaluation of the cleanability of CIP Automatic valves. *J. Dairy Sci.* 39:919. (Abstr.)
 - 106 Sing, E. L., P. R. Elliker, L. J. Christensen, and W. E. Sandine. 1967. Effective testing procedures for evaluating plant sanitation. *J. Milk Food Technol.* 30:103.
 - 107 Sisley, J. P. 1944. Determination of the industrial value of detergency. I. Methods to determine wetting power. A review. *Ind. Corps Gras* 2(1):13.
 - 108 Smith, G. A. 1957. Practical points to consider in automation cleaning. *Amer. Milk Rev.* 19(10):48.
 - 109 Smith, G. A., and T. I. Hedrick. 1967. Reuse of cleaning solutions in CIP. *J. Milk Food Technol.* 30:256.
 - 110 Sommer, H. 1952. Market milk and related products. 3rd ed. Published by the author. Madison, WI.
 - 111 Stevenson, D. G. 1961. Chapter 6 in *Ancillary effects in detergent action. Surface activity and detergency*. K. Durham, ed. Macmillan Publishing Co., London.
 - 112 Swartling, P. A. 1959. The influence of the use of detergents and sanitizers on the farm with regard to the quality of milk and milk products. *Dairy Sci. Abstr.* 21:1.
 - 113 Tachibana, T., A. Yabe, and M. Tsubomura. 1960. The existence of a maximum in the curve of washing power vs. concentration of detergent. *J. Colloid Sci.* 15:278.
 - 114 Thomas, S. B. 1963. Bacteriological aspects of circulation cleaning of pipeline milking plants. *J. Soc. Dairy Technol.* 16:126.
 - 115 Utermohlen, W. P., Jr., and E. L. Wallace. 1947. Detergency studies. *Text. Res. J.* 17:670.
 - 116 White, J. C., and G. O. Rabe. 1970. Evaluating the use of nitric acid as a detergent in dairy CIP systems. *J. Milk Food Technol.* 33:25.