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S. M. Kulkarni
University of Nebraska-Lincoln

R. G. Arnold
University of Nebraska-Lincoln

R. B. Maxcy
University of Nebraska-Lincoln

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RESEARCH PAPERS

Reuse Limits and Regeneration of Solutions for Cleaning Dairy Equipment[1]

S. M. KULKARNI, R. G. ARNOLD, and R. B. MAXCY
Department of Food Science and Technology
University of Nebraska
Lincoln 68503

ABSTRACT

Usable life of cleaning solutions in a model heat processing unit was studied by evaluation of the nature and quantity of residue remaining on the surface of the equipment after cleaning. Lipoidal residue was recovered from equipment surfaces by ether rinsing. A surface displacement technique was used to quantify the lipoidal material, which was characterized by gas-liquid chromatographic analyses of triglycerides and fatty acid methyl esters. Effects of factors, such as water hardness, rinse water temperature, cleaning solution additives and acid versus alkaline cleaners also were investigated in relation to overall cleaning efficiency. After evaluating limits of reuse of cleaning solutions, used solutions were regenerated by sorption of lipoidal residue on Celite, resulting in restored cleaning effectiveness. When spent-cleaning solutions were circulated in previously cleaned equipment, there was redeposition of lipoidal material onto the equipment surfaces.

INTRODUCTION

Modern fluid food processing equipment is commonly cleaned-in-place (CIP) by circulation of detergent solutions. There is considerable literature on design and operation of CIP, various factors influencing cleaning and procedures to evaluate cleanliness. However, little information is available on the feasibility and means of extending usefulness of cleaners to enhance the economic efficiency of cleaning operations. Reuse of cleaning solutions also would reduce total amounts of cleaning materials in food processing operations, which in turn would conserve resources and reduce environmental pollutants.

Commercial experience has indicated that CIP solutions may be reused for cleaning soiled equipment, especially with surfaces that have no "burn on" milk deposits. Conditions for satisfactory reuse and specific limitations, however, are vague (12). Based on visual and bacteriological criteria to evaluate cleanliness of surfaces, extensive reuse of chlorinated alkali cleaning solution for milk plant equipment involving nonheated surfaces was satisfactory from a cleaning and sanitation viewpoint (12).

The tenacious residue resisting removal by circulation cleaning is lipoidal in nature (2, 10). Recent studies have shown that alkaline cleaners can be freed successfully of lipoidal material by sorption on certain inert substances (9).

The present investigation explored reuse potential of spent cleaners and regeneration of solutions for cleaning milk contact surfaces of heat processing equipment.

EXPERIMENTAL PROCEDURES

Equipment, Soiling and Cleaning Processes

A model heat processing system described previously (10) was used to simulate soiling and cleaning processes in a high-temperature short-time pasteurizer. Soiling of equipment surfaces was accomplished by heating Grade "A" raw milk to 75 C with a single pass through the stainless steel tubing (5 mm inside diameter (ID) x 200 cm) submerged in a hot water bath (90 to 95 C). The flow rate during 1 h soiling was approximately .30 m per s. The soiled equipment was prerinsed with tap water until the effluent was clear. Cleaning involved circulation of a 1% sodium hydroxide solution at 75 to 80 C at a flow rate of approximately .61 m per s which resulted in turbulent flow. A fresh solution (200 ml) was circulated for each 2 min

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interval giving a total cleaning time of 10 min.

In independent trials, the following variables were studied:

1) Soiling media; raw whole milk (3.3% fat), raw commercially separated skim milk (.1% fat), and commercial standardized raw cream (20% fat).

2) Rinse water temperature; cold (10 to 15 C), hot (75 to 80 C).

3) Acid cleaner, 1% (Dicoloid-FF, Diversey Co., Chicago, IL) vs. alkaline (1% wt/vol NaOH) cleaner.

4) Water hardness and cleaner additives; 1% NaOH solution in distilled water and tap water; .9% sodium hydroxide plus .1% sodium hexametaphosphate in tap water.

After each soiling and cleaning operation, the stainless steel tubing was rinsed thoroughly with water to remove traces of cleaning solution, air-dried, and then rinsed with ether to recover tenacious lipoidal material that might have remained. The ether rinsings of apparently clean equipment were retained for analysis.

In the following experiments, a continuous cleaning process was performed by circulation of 200 ml alkaline solution for 10 min.

Reuse of Cleaning Solution

To study the reusability of spent cleaner, successive trials involved soiling from heating of raw whole milk and subsequent cleaning by circulation of solution used in the previous cleaning operation. Ether rinsings of the cleaned surfaces were collected after each trial as before.

Regeneration of Cleaning Solution

In similar soiling and cleaning experiments, 200 ml quantities of spent cleaning solutions were regenerated by filtration through 3 g of Celite in a 2.5 cm outside diameter × 10 cm chromatography column fitted with a sintered glass filter. Suction was applied to speed the filtration. The process was repeated using fresh Celite. The filtered solution was reused following regeneration to clean soiled surfaces in sucessive trials.

Analyses for Lipoidal Materials

All cleaning solutions were extracted with organic solvents (2). Quantification of lipoidal residue in the solvent extracts of cleaners and in the ether rinsings of cleaned equipment surfaces was accomplished by a surface displacement micromethod (3, 5, 7, 10).

The degree of effectiveness of cleaning was determined by the extent of tenacious material remaining on the apparently clean equipment surfaces and subsequently recovered in the ether rinsing treatment.

Gas-liquid chromatography (GLC) of triglycerides and fatty acid methyl esters was employed to characterize the tenacious residue remaining after circulation cleaning. GLC equipment and procedures have been reported (2, 10).

Analysis of Proteinaceous Material

Proteinaceous soil in the cleaning solutions was determined by the method of Lowry et al. (8).

RESULTS

Soil Deposition

Data in Table 1 represent amounts of soil deposited onto the equipment surface and subsequently removed by circulation cleaning

TABLE 1. Soil deposition on stainless steel surface (314 cm^2) from heating of different dairy products.

	Soil deposition (g)	
Soiling medium	Lipoidal	Proteinaceous
Skim milk[a] (.1% fat)	.055	1.206
Whole milk[b] (3.3% fat)	.329 (Std. deviation .093)	1.121 (Std. deviation .335)
Cream[a] (20% fat)	3.871	2.483

[a]Average of 2 replicates.

[b]Average of 10 replicates.

treatment when different dairy products were heat processed. The soil deposition increased with the total solids content of the product. The fat content of soiling medium appeared to exert a great influence on soil deposition. This observation is consistent with the findings of Burton (4), who noticed a strong positive correlation between the amount of deposit and fat content of milk

Rinse Water Temperature

In four replicates with cold water (10 to 15 C) rinse, an average of 23% of lipoidal and 43.9% of the total proteinaceous matter on the equipment surfaces was removed by rinsing. Hot water (75 to 80 C) rinsing was slightly more efficient in removing lipoidal material (33.5%) while removal of proteinaceous soil by cold or hot water was similar. Patel and Jordan (11) showed the amount of cold water required to rinse milk solids from stainless steel pipe increased significantly with the fat content of dairy product whereas with warm water rinsing, the increase with fat content was considerably less.

Acid vs. Alkali Cleaner

A comparison of the rate of soil removal by acid versus alkaline cleaner (four replicates each) indicated that alkaline cleaner (1% NaOH) was more efficient in removing both lipoidal (Fig. 1) and proteinaceous (Fig. 2) material from the equipment soiled by heating of raw whole milk. The soil removal by the alkaline cleaner was almost complete at the end of 6 min whereas with the acid cleaner the soil persisted throughout the 10 min cleaning operation.

Further evidence of relative ineffectiveness of acid cleaner was obtained by rinsing cleaned equipment with ether and determining the lipoidal material recovered. Similarly, cleaned equipment was subjected to additional washing with alkali for 5 min to recover proteinaceous matter not removed during normal cleaning operation. An average of 11.5 mg of lipoidal and 43 mg of proteinaceous soil was on the acid cleaned surfaces. After alkaline cleaning, however, only about 1 mg of lipoidal material remained and no protein residue was detectable.

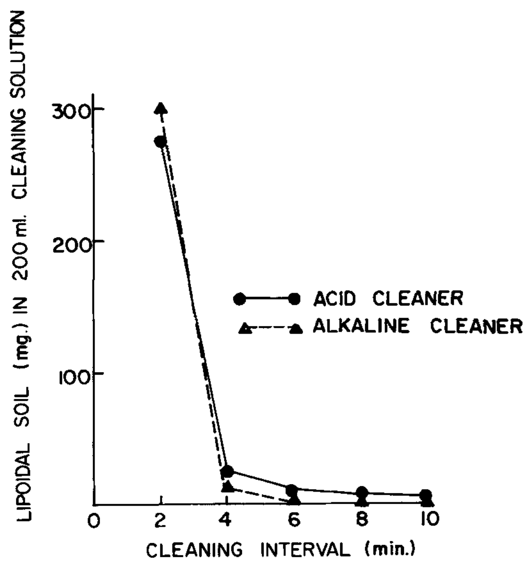

FIG. 1. Comparative rate of removal of lipoidal soil with progression of circulation cleaning by acid and alkali cleaners. The ordinate represents soil removed during each 2-min interval of cleaning with a fresh solution.

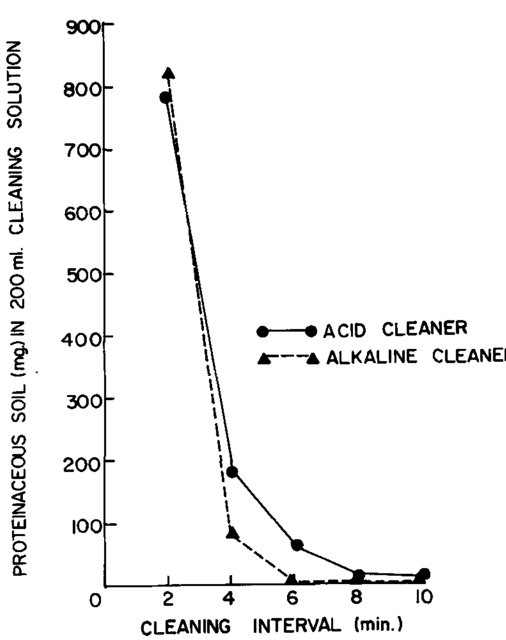

FIG. 2. Comparative rate of removal of proteinaceous soil with progression of circulation cleaning by acid and alkali cleaners. The ordinate represents soil removed during each 2-min interval of cleaning with a fresh solution.

Water Hardness and Cleaning Solution Additives

Water hardness (average 220 ppm or .84 g/3.78 liters) apparently did not impair the performance of cleaning solutions. Fig. 3 shows the rate of soil removal with progression of circulation cleaning with 1% NaOH solution in tap water. There was no difference in the cleaning rate when distilled water (soft) was used to prepare the cleaning solution. With both cleaning systems, the major portion of soil was removed within the first 4 min of cleaning, and only 1 to 2 mg of lipoidal soil was recovered in the ether rinse of cleaned tubing. Further, no proteinaceous residue was detected in the final alkaline wash following normal cleaning.

Inclusion of sodium hexametaphosphate [.9% NaOH plus .1% $(NaPO_3)_6$ in tap water] gave results similar to those for tap water and for soft water. Through the additive, sodium hexametaphosphate, did not increase removal of lipoidal material, the primary function was to prevent deposition of minerals from cleaning solutions.

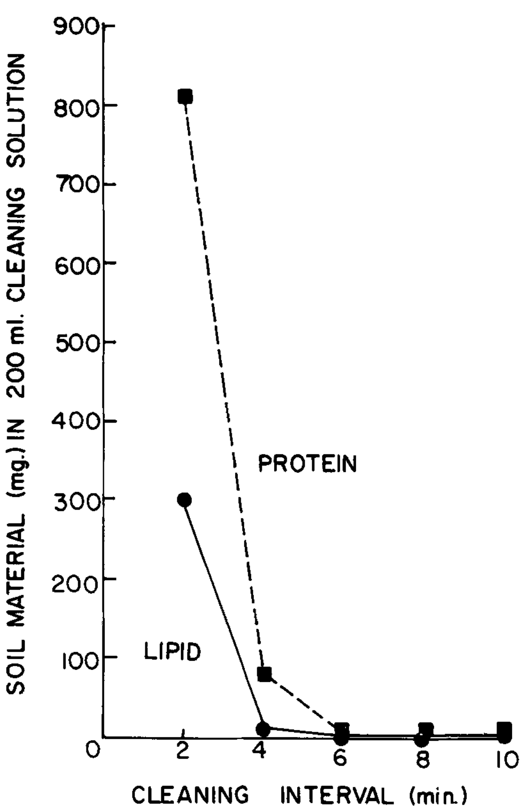

FIG. 3. Comparative rate of removal of lipoidal and proteinaceous material with progression of circulation cleaning by alkali cleaners. The ordinate represents soil removed during each 2-min interval of cleaning with a fresh solution.

Reuse of Spent Cleaning Solution

Data in Table 2 indicated a progressive depletion in the cleaning ability of used cleaning solutions. Increasing quantities of soil were recovered in the ether wash of clean equipment surfaces after cleaning of soiled surfaces with spent solution from previous trials.

Analyses (GLC) of ether soluble residue as triglycerides (Fig. 4) and as fatty acid methyl esters (Fig. 5) revealed that the residue was lipoidal in nature and similar to the resistant fraction of alkaline circulation cleaning as reported (2). Results were similar for alkaline cleaner with or without added phosphate.

Redeposition Phenomenon

Cleaning solution previously used for three successive cleaning cycles was recirculated at 75 to 80 C for 10 min through the thoroughly cleaned and ether rinsed equipment. Approximately 8 to 10 mg of lipoidal material was recovered when the stainless steel tubing was again rinsed with ether after being rinsed with water and air-dried. The only apparent explanation was in terms of redeposition of lipoidal material onto the clean surface from the spent cleaner. In commercial CIP operations for food processing plants, such redeposition of soil would be undesirable. The inference was that the accumulated soil in the reused cleaning solutions must be removed to extend their usefulness and effectiveness for subsequent cleaning operations.

TABLE 2. Amounts of ether soluble material recovered from stainless steel surface cleaned with reused solutions.

Cleaning cycles with same solution	Ether soluble residue (mg) recovered from clean surface[a]
1	1 – 2
2	12 – 15
3	26 – 32

[a]Results of 3 replicates.

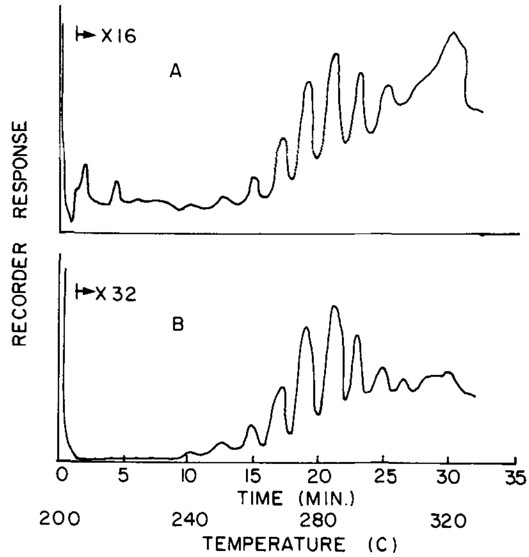

FIG. 4. Gas liquid chromatograms of triglyceride material in A, ether rinse of clean equipment surface; B, milkfat. Gas chromatograph: Varian Aerograph 1700 with flame ionization detector. Column: 61 cm X 3.2 mm OD stainless steel packed with 2.5% SE-30 on 60–80 mesh VarPort 30, programmed from 200 to 320 C at 4 degrees/min. Injection port: 270 C. Detector: 350 C. Carrier gas (N_2): 25 ml/min.

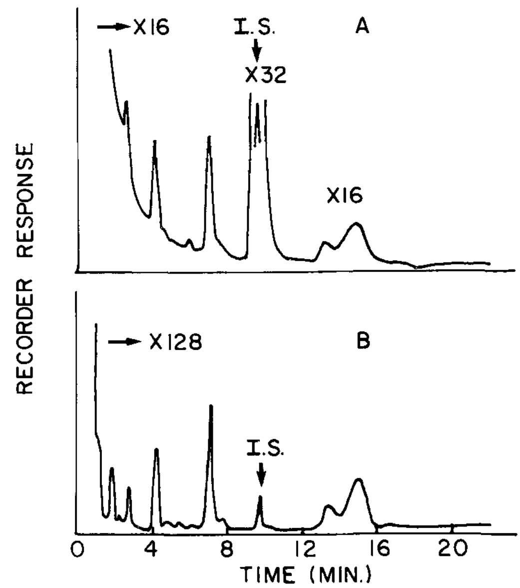

FIG. 5. Gas-liquid chromatograms of methyl esters of fatty acids of A, ether rinse of clean equipment surface, the internal standard (I.S.) was methyl heptadecanoate; B, milkfat. Gas chromatograph: Varian Aerograph 1700 with flame ionization detector. Column: 305 cm X 3.2 OD stainless steel packed with 7% DEGS on 60–80 mesh Chromosorb P; 180 C isothermal. Injection port and detector: 210 C. Carrier gas (N_2): 55 ml/min.

Regeneration of Cleaning Solutions for Reuse

Since increasing amounts of lipoidal residue in the used solutions resulted in reduced cleaning effectiveness, it was contended that removal of such material could lead to restored cleaning ability for further use. Maxcy and Arnold (9) investigated the capacities of various materials for sorption and elution of milkfat in alkaline solution and found Celite was the best. Our studies showed that when the cleaning solution was filtered twice through fresh Celite (3 g), over 90% of the lipoidal material in 200 ml of used cleaning solution was removed. Substantial amounts of proteinaceous soil also were removed leaving a relatively clear solution. Minor losses in volume were made up by adjusting the solution to 200 ml with fresh cleaner.

The spent cleaner regenerated in this manner was used for cleaning of soiled surfaces, and effectiveness of cleaning was evaluated by quantification of ether soluble lipoidal material remaining on clean equipment. Results in Table 3 demonstrated that used cleaning solutions can be freed effectively of lipoidal soil resulting in a significant restoration of cleaning ability. Active alkalinity of cleaning solution decreased by approximately 12% after one use, 22.2% after second use, and 32.7% when used thrice for cleaning soiled equipment. Therefore, making up the loss in alkalinity following each use may improve further cleanability of regenerated solutions.

In these studies the Celite for sorption of lipoidal residue easily can be reconditioned for further use by washing it with organic solvents. Studies on the cost of regenerating cleaning solutions and reconditioning of Celite were not feasible due to small laboratory operation.

DISCUSSION

The model heat processing unit provided a simple and convenient way to study various factors influencing the circulation cleaning process. The soil removal rate and nature of tenacious residue were similar to those of a small commercial high-temperature short-time

TABLE 3. Comparison of effectiveness of cleaning by reused solutions with and without regeneration.

Cleaning cycles with same cleaner	Lipoidal material (mg) recovered from clean surface	
	Non-regenerated cleaner[a]	Regenerated cleaner[a]
1	1 – 2	1 – 2
2	12 – 15	4
3	26 – 32	6 – 8

[a]Results of 3 replicates.

pasteurizer. Therefore, the data with our laboratory model on evaluation of cleaning effectiveness, reusability of spent cleaners, and their regeneration for extended use should be applicable to commercial CIP systems.

The surface displacement technique for the determination of microquantities of fatty material affords a simple, yet extremely sensitive and reproducible procedure for evaluating cleaning systems. The extent of tenacious lipoidal residue recovered from circulation cleaned equipment surfaces gives a useful and direct index for evaluating thoroughness of cleaning.

Progressive depletion in cleaning ability of reused solutions may in part be due to the redeposition of soil already removed in the cleaning process. The redeposition phenomenon has been postulated (6) and demonstrated in a system involving radiolabeled tristearin soil and glass surface (1). The loss in active alkalinity again may partly explain loss in effectiveness of reused cleaners.

Our results have shown cleaning ability of used solutions can be restored successfully by freeing them of lipoidal soil by sorption on Celite. Presently, no guidelines or criteria, however, are available (e.g., limiting residue per unit surface area) to determine the cleanliness acceptable from the public health standpoint. In commercial CIP systems, extending the usability of cleaners has two-fold significance. Economic benefits can be derived from increased usable life of cleaners and savings on cleaning cost. Secondly, discharge of highly alkaline spent cleaners is often a shock to the biological system of sewage disposal plants. Thus, our goal to enhance usability and effectiveness of commercial cleaning systems has an element of environmental protection.

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