

## BOTTLE-WASHING STUDIES UNDER PLANT CONDITIONS

C. N. STARK, R. F. HOLLAND, J. C. WHITE, AND M. J. GURDIAN

*Laboratory of Bacteriology and Department of Dairy Industry,  
- Cornell University, Ithaca, N. Y.*

IT little profits a milk dealer to enter an involved quality control program, pasteurize milk in the best of equipment, and handle it with extreme care if the milk is placed in containers which are not essentially sterile. On the other hand, any operator with an eye to economy will not want to use in his bottle washer caustic solutions which are stronger than necessary nor will he want to burden his washer with temperatures higher than are necessary to do an adequate job. For these reasons, it was believed that a thorough investigation should be made of the relationship between temperature, time of exposure, and caustic concentration in a soaker-type milk bottle washer.

The recommendations for caustic-temperature relationships which are in general use in the dairy industry throughout the United States are based on the work of Levine and co-workers<sup>1, 2, 3, 4, 5</sup> who investigated the washing of carbonated beverage bottles. Some laboratory tests were made on milk bottles and it is known that cleaning and "sterilizing" the milk bottle presents a radically different problem from the carbonated beverage bottle. The beverage bottle will contain residues of sugar, acids, and perhaps some extraneous matter; the milk bottle contains, in addition to these materials, a complex film of fat, protein, and minerals. This complex film provides a medium which is ideal for the growth of bacteria. The elimination of these bacteria from the bottle, together with the milk solids, presents the practical and important problem for the dairy plant. It is a problem comparable in importance with the pasteurization process itself. The plant operator

must produce a bottle which is clean and spotless in order to hold the consumer's confidence, and which has a bacterial count low enough to be safe and to meet public health standards.

The question, what constitutes optimum conditions for operating a bottle washing machine, is frequently answered in different ways. The manufacturer of the equipment will have his recommendations, but the representatives of the health department are the ultimate authorities and, in any event, must be satisfied. Recommendations of all equipment manufacturers and all health departments are not necessarily the same. Arnold and Levine<sup>5</sup> state "For any given temperature and concentration (of alkali) it would be necessary to expose the bottle 18.1 times as long to meet the New York City requirements as would be required to comply with the Chicago law." New York City requires a minimum soaking time of 7 minutes at the temperature of 150° F. with 2 percent of sodium hydroxide in the soaker tank; Chicago specifies a minimum of 5 minutes at 120° F. with 1.6 percent sodium hydroxide. There is little information available to show what standards are necessary to produce a clean, safe milk bottle.

The Association of Bottlers of Carbonated Beverages has recommendations; based on the work of Levine and associates,<sup>3</sup> stipulating that "Unclean bottles shall be exposed to a 3 percent alkali of which not less than 60 percent is sodium hydroxide for a period of not less than 5 minutes at a temperature of not less than 130° F. or an equivalent cleansing and sterilizing process." It has not been proved that

the standards for cleaning and "sterilizing" beverage bottles are necessarily desirable or correct for milk bottles.

In an attempt to determine the adaptability of these recommendations to milk bottle washing, a project for the study of these problems under practical commercial conditions was organized at Cornell University. The aims of the study were: (1) to determine the germicidal and detergent efficiency of different concentrations of certain caustic solutions at different temperatures; (2) to observe the importance of exceptionally high-temperature operation to cleaning and "sterilizing" efficiency; and (3) to observe the physical cleanliness of the bottles under these various conditions.

#### EXPERIMENTAL METHODS

All the experiments of this project were carried out at the Cornell University dairy plant at Ithaca, New York. This plant bottles quarts, pints, and half-pints. Most of the quarts are sold to household trade at the retail store in the plant, while most of the half-pints are consumed by the students in dormitories and cafeterias on the campus. The dirty bottles returned from the routes are stored indoors until they are washed and refilled. Quart bottles normally are rinsed by the consumer before being returned, but the pint and half-pint bottles rarely receive any rinse. They usually contain milk and very commonly cigarette ashes and other extraneous materials.

The plant is equipped with a Cherry-Burrell Model C eight-wide soaker washer. This bottle washer had been in operation at the plant for only a few months when this project was undertaken. The ABCB standards for washing bottles were used as a guide for setting up the temperature-caustic concentration relationships up to 150° F. Above this temperature a caustic concentration of 0.9 percent was maintained as a practical minimum. These caustic-temperature combinations are indicated when the ABCB standards

are converted to the equivalent of a 3-minute soaking period. Table 1 gives these relationships.

TABLE 1  
TEMPERATURE-CAUSTICITY RELATIONSHIPS  
STUDIED USING A 3-MINUTE SOAKING  
PERIOD

Temperature ° F.	Causticity %
120	4.2
130	2.9
140	1.9
150	1.3
160	0.9
170	0.9
180	0.9

The washer feed water hardness varied between 5 and 7 grains per gallon during the experiment.

To assure accurate control of the soaking temperature at all times, air-operated, thermostatically controlled steam valves were installed on the washer. An abundance of high-pressure steam was available at all times. Two separate recording thermometers made continuous daily records of the temperature of the soak solution and of the tank providing the water for the so-called first rinse or pressure wash. The washer was operated at a speed corresponding to an immersion period of 3 minutes and 15 seconds and so maintained throughout the experiment. This relatively high speed was selected to produce the shortest soaking time that would be encountered normally in commercial operation, thus placing the greatest possible load on the temperature and alkalinity combinations used in the trials.

For each given temperature, the tests usually were divided into three 5-day periods, in a few cases the periods were 10 days. During the first period the causticity of the solution was adjusted to the desired level by the addition of 76 per cent flake caustic (sodium hydroxide). During the second period, an amount of trisodium phosphate equal to one-tenth of the required weight of the sodium hydroxide was added to the base solution. Finally,

during the third period tetrasodium pyrophosphate was added in the same proportion as the trisodium phosphate, while maintaining as a base the same solution that was used during the second period. A 32-pound sodium hydroxide charge was required to produce a solution having 1 percent caustic alkalinity in this machine. The indicated concentrations of these alkalis were maintained by several additions to the soak tank during the day's run. The required amount of sodium hydroxide was added and other materials, when in use, were added in correct proportion.

*Causticity.* When making causticity determinations, 5 ml. of the soak-tank solution were transferred with a pipette to a 100-ml. volumetric flask and made to volume; a 25-ml. portion of the diluted solution was transferred to a beaker and, if only sodium hydroxide was used in the soak solution, the aliquot was titrated directly with standard N/10 hydrochloric acid to the phenolphthalein and methyl orange end points to obtain causticity and carbonate alkalinity; if one or both of the phosphates were present in the basic sodium hydroxide solution, the carbonates and phosphates were precipitated by the addition of barium chloride and the solution was titrated to the phenolphthalein end point with N/10 hydrochloric acid.

*Bacteria.* To determine the bacterial count of the bottles, daily samples were selected. Three half-pint bottles were collected near the beginning, three near the middle, and three near the end of the run. As soon as the bottles were discharged by the washer, they were closed with sterile bottle caps which covered the pouring lip. These bottles were stored in a cooler at 40° F. until bacterial counts were made on them later in the day. The counts on these bottles were made according to standard procedures except that instead of using 100 ml. of sterile water, only 10 ml. were used for the rinsing operation because of the small numbers of bac-

teria present. One ml. samples of this wash water were plated in duplicate on standard skim-milk agar and the plates were incubated at 35° C. On occasions, 5-ml. amounts of rinse water were plated as a further check on the low bacterial counts being obtained.

*Visual cleanliness.* Twelve quart bottles, collected at random daily, were used for making observations on visual cleanliness.

*Specific gravity.* The specific gravity of the solutions was measured at the beginning and at the end of each of the three periods by means of two hydrometers, the scales of which covered the range of the solutions. All of these measurements were made at 25° C.

*Surface tension.* The surface tension of the solutions was determined with the same frequency as the specific gravities. Cenco-du Nouy precision tensiometer No. 10402, having a 4-cm. platinum ring, was used. The apparatus was properly calibrated and leveled; the platinum ring was washed with distilled water and dried with an alcohol flame before each determination. For surface tension measurements the temperature of the solution was adjusted to 20° C. in a water bath.

## RESULTS

*Sodium hydroxide soak solution.* In table 2, recording the findings on tests made for 38 days using only sodium hydroxide in the soak tank, certain observations appear to be of interest. More than 55 percent of the bottles tested failed to show any colonies of bacteria. The average colony count per bottle never exceeded 15. The highest colony count of bacteria for any one bottle was 170; only once did the bacterial colony count exceed 100. Since the accepted standard requires less than 238 colonies per half-pint bottle,<sup>7</sup> all of the alkali concentrations and temperatures tested gave satisfactory bacterial counts.

Due to the known resistance of bacterial endospores, many Grampositive cocci, and acid-fast organisms to strong

TABLE 2

THE BACTERIAL CONTENT OF HALF-PINT MILK BOTTLES SOAKED FOR 3.25 MINUTES IN SODIUM HYDROXIDE SOLUTION

Caustic soda %	Soak temp. ° F.	No. of days run	No. bottles tested	Bottles showing no colonies %	Average no. of colonies, any bottle	Highest no. of colonies, any bottle
4.2	120	11	99	40	4	100
2.9	130	5	45	58	4	50
1.9	140	5	45	44	15	40
1.3	150	5	45	58	6	20
0.9	160	7	63	65	4	50
0.9	170	5	45	60	7	170

concentrations of alkalis, it would be expected that many such organisms would survive this treatment. It should be mentioned that the medium and incubation time used in these studies would not have detected the presence of surviving acid-fast organisms. Doubtless, all of the less heat-resistant and less alkali resistant organisms in these bottles were killed by any of these treatments. The observed bacterial colonies were formed by the more resistant types. Since the lowest concentrations of sodium hydroxide employed gave a pH of around 13.0, the alkali killing effect on many less resistant bacteria would be high in all the alkali concentrations tested. Because these bottles failed to rinse well, the use of only sodium hydroxide in the soaker tank is not recommended. Because of excessive bottle breakage at temperatures of 170° F. and above, these temperatures are not recommended for commercial operation. This is especially true under winter condi-

tions when bottles may be extremely cold and rinse water temperatures near freezing. A limited number of runs made at 180° F. and 190° F. gave results of the same order as at 160° F. and 170° F.

*Sodium hydroxide and trisodium phosphate soak solution.* If the addition of trisodium phosphate to the sodium hydroxide solution in the soak tank of a milk bottle washing machine is an advantage, these tests should indicate it, since the trisodium phosphate was added to the same concentrations of sodium hydroxide as used in the previously reported 42 tests. It is true that the amounts of trisodium phosphate added were small but they closely approximate the quantities commonly incorporated in bottle washing mixtures sold to the dairy trade.

In table 3, reporting the results of 29 days tests using sodium hydroxide and trisodium phosphate in the soak tank, 32 percent of the bottles tested showed no colonies of bacteria by the

TABLE 3

THE BACTERIAL CONTENT OF HALF-PINT MILK BOTTLES SOAKED FOR 3.25 MINUTES IN A SOLUTION OF SODIUM HYDROXIDE AND TRISODIUM PHOSPHATE

Caustic soda + phosphate %	Trisodium phosphate %	Soak temp. ° F.	No. of days run	Bottles tested No.	Bottles showing no colonies %	Average no. of colonies, any bottle	Highest no. of colonies, any bottle
4.2	0.42	120	6	54	36	23	350
2.9	0.29	130	5	45	49	7	65
1.9	0.19	140	5	45	44	9	185
1.3	0.13	150	5	45	42	9	100
0.9	0.09	160	4	36	11	23	160
0.9	0.09	170	4	36	3	41	210

tests employed. Only one bottle exceeded the permissible standard limit, having a count of 350. The bacterial counts were more variable and some were higher. The results indicate that the addition of these amounts of trisodium phosphate adds nothing to the bottle-cleaning properties of the soak solution and may be detrimental.

*Sodium hydroxide, trisodium phosphate, and tetrasodium pyrophosphate soak solution.* Tetrasodium pyrophosphate was added to the same solutions of sodium hydroxide and trisodium phosphate used in previously reported tests. The observations made during running of the 25 tests using these materials are reported in Table 4.

having tetrasodium pyrophosphate or similar material in the soak solution of the bottle washer. Some other equally good polyphosphate could possibly be used; only tetrasodium was used in these investigations.

Several runs were made using only sodium hydroxide and tetrasodium pyrophosphate in the soak tank. The results obtained were as good or better than those in which all three alkalis were present, indicating that trisodium phosphate adds little or nothing to the cleaning qualities of the soak solution.

*Physical cleanliness of bottles.* In Table 5 it can be seen that tetrasodium pyrophosphate in the soaker tank was necessary to obtain properly rinsed bot-

TABLE 4

THE BACTERIAL CONTENT OF HALF-PINT MILK BOTTLES SOAKED FOR 3.25 MINUTES IN A SOLUTION OF SODIUM HYDROXIDE, TRISODIUM PHOSPHATE, AND TETRASODIUM PYROPHOSPHATE

Caustic soda %	Tri-sodium phosphate %	Tetra-sodium pyrophosphate %	Soak temp. ° F.	No. of days run	Bottles tested	Bottles showing no colonies %	Average no. of colonies, any bottle	Highest no. of colonies, any bottle
4.2	0.42	0.42	120	4	36	31	19	125
2.9	0.29	0.29	130	4	36	56	4	60
1.9	0.19	0.19	140	3	27	56	4	5
1.3	0.13	0.13	150	3	27	52	7	25
0.9	0.09	0.09	160	6	54	33	7	30
0.9	0.09	0.09	170	5	45	47	7	75

In all of the 25 days tests made, 44 percent of the bottles tested failed to show any bacterial colonies. The largest number of bacteria indicated for any one bottle was 125. The majority of the counts per bottle was nearer 25. Less variation in the bacterial counts from day to day was observed. The bottles rinsed satisfactorily at all temperatures and all concentrations of chemicals used. This better rinsing is believed to be a factor in obtaining more uniform and lower bacterial counts. Since an improperly rinsed milk bottle is unsatisfactory, even though it shows a low bacterial count, the findings reported in these studies emphasize the major importance of

tles. This condition is the ideal desired, since the bacteriological findings show these glistening, clean bottles to contain only small numbers of bacteria. Rinsing was never satisfactory without tetrasodium pyrophosphate, although in other solutions the bottles did rinse somewhat better at the higher temperatures.

*The surface tension readings* were all low and varied only slightly due to different alkalinities, and the chemical composition of the soaker tank solutions. It was obvious that surface tension readings on the soaker tank solution do not measure, to even a slight degree of accuracy, the efficiency of the bottle washing process. A low

TABLE 5  
OBSERVATIONS ON BOTTLE CLEANLINESS-AS RELATED TO TEMPERATURE AND PERCENT OF CAUSTICITY

Added chemicals	120° F.—4.2%	130° F.—2.9%	140° F.—1.9%	150° F.—1.3%	160° F. or higher—0.9%
NaOH	Poor rinsing—water spots Blue-white film on bottom		Water spots		Slight cloudiness
10 parts NaOH 1 part Na <sub>2</sub> PO <sub>4</sub>	Cloudy, white streaks Water spots, slippery when wet		Gradually become blue white		Few white spots Slight blue-white film
10 parts NaOH 1 part Na <sub>2</sub> PO <sub>4</sub> 1 part Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	Satisfactory Clean		Satisfactory Clean		Satisfactory Clean

surface tension is generally held to improve the penetration of the washing solution and to give better rinsing. These readings did not, in any degree, correlate with the rinsing results observed.

The specific gravity would be expected to increase as the soaker tank solution ages. This does not always occur. Specific gravity measurements in these tests did not measure or correlate with efficiency in washing milk bottles.

Carbonate alkalinity would be assumed to increase as the soaker tank solution ages. At the higher temperatures and lower causticities the carbonate alkalinities were lower, but no noticeable correlation between efficiency of bottle washing and carbonate alkalinity could be observed.

#### CONCLUSIONS

1. The concentrations of alkali and the corresponding holding time and temperatures suggested by Levine for washing bottles under commercial plant conditions, when followed, will meet the U. S. Public Health Service standard for a bacteriologically satisfactory milk bottle.

2. To obtain satisfactorily rinsed bottles tetrasodium pyrophosphate was essential. The addition of trisodium phosphate, in the amounts used, was not an advantage.

3. The measurements made on surface tension, specific gravity, and carbonate alkalinity did not correlate with the satisfactory rinsing of the bottles or the number of surviving bacteria found. Apparently there are other more important factors which these tests do not measure.

4. Soaker solution temperatures of 170° F. or higher are not needed to obtain a low bacterial count bottle of excellent appearance. Breakage may be high at this temperature and above.

5. The alkalinity, temperature, and time of exposure standards for milk

(Continued on page 205)

TABLE 4  
COPPER SERUM READINGS OF SAMPLES GROUPED ACCORDING TO  
PERCENTAGE SOLIDS-NOT-FAT

Solids-Not-Fat Groups	Number of Samples	Mean	Range	Standard Deviation
-8.00	25	36.72	38.8-35.4	0.960
8.00-8.49	42	37.57	38.8-35.6	0.737
8.50-8.99	46	37.75	40.7-36.4	0.897
9.00-	17	37.95	38.8-37.0	0.560
All	130	37.52	40.7-35.4	0.910

In Table 4 the samples were grouped according to percent solids-not-fat and the mean, range, and standard deviation are shown for the copper serum readings. While the copper serum readings increased slightly with the increases in solids-not-fat, this was not so noticeable as was the case of increases in the butterfat content, especially with samples containing more than 8 percent solids-not-fat. A statistical study comparing the probable error of the groups showed the results to be highly significant. This was especially true when comparing the low butterfat and solids-not-fat group with the next higher grouping, indicating that low copper and acetic serum results of these samples were normal for their composition.

#### SUMMARY

An investigation was made of the refractive indices of the acetic and copper sera and the ash of the acetic serum of 130 samples of milk. Some of the samples were chosen because of known low composition, especially abnormally low solids-not-fat content. All samples showed a normal freezing point depression, indicating that none of the samples were adulterated with water. Of all the samples analyzed, 38 had a low solids-not-fat content, and these either had refractometer readings that indicated added water or the readings were in the low range.

Data have been presented indicating that it is possible to obtain low refractometer readings on unadulterated milk samples having a low composition solids-not-fat.

#### Bottle-Washing Studies

(Continued from page 201)

bottle washing maintained by some of the larger cities provide a very large margin of safety.

#### REFERENCES

1. Levine, Max, Buchanan, J. H., and Toulouse, J. H. Influence of Sodium Chloride, Sodium Carbonate, and Trisodium Phosphate on Germicidal Efficiency of Sodium Hydroxide. *Iowa State College J. Sci.*, 2, 19-29 (1927).
2. Levine, Max, Buchanan, J. H., and Lease, Grace. Effect of Concentration and Temperature on the Germicidal Efficiency of Sodium Hydroxide. *Ibid.*, 1, 379-394 (1927).
3. Buchanan, J. H., and Levine, Max. The Testing of "Washing" Solution. *ACCB Educational Bulletin No. 1*, American Bottlers of Carbonated Beverages, 1929 (Reprint 1936).
4. Arnold, C. R., and Levine, Max. Here Are Ways to Wash Bottles Clean at Low Cost. *Food Industries*, pp. 205, 235, 236, April, 1939.
5. ——— and ———. Evaluation of Germicidal Properties of Sodium Hydroxide and Alkaline Washing Compounds. *Iowa State College J. Sci.*, 16 (4), 519-538 (1942).
6. Myers, R. P. The Effect of Hydroxyl Ion Concentration on the Thermal Death Rate of *Bacterium coli*. *J. Bact.*, 15, 341-356 (1928).
7. *Public Health Bulletin No. 220* (1939).