

Qualitative Foam Stability Evaluation of Hand Dishwashing Detergents

LESTER O. LEENERTS and HOWARD J. MYERS, Application Research Laboratory, Purex Corporation Ltd., South Gate, California

IN THE DAY-TO-DAY USAGE of hand dishwashing detergents both in home and restaurant applications, the determination of the effectiveness is most usually accomplished by observation of the foam. Although such an approach may not be realistic from the standpoint of theoretical detergency, it is nevertheless a practice which must be taken into consideration when the manufacturer or compounder begins his formulation work. Even though a certain combination of ingredients has been found to be effective in the removal of soil as measured by either semi-practical or instrumental tests, it is still necessary to determine the quantity and quality of foam generated in the dishpan both with and without soil.

Since the advent of synthetic detergents many tests have been developed for the study of the foaming properties of surfactants. All have certain degrees of merit when the factors leading to their development are taken into account. We shall not attempt to review all of the tests encountered in the literature since a large percentage of them do not deal with the problems encountered when soil is present in the detergent solution. We will however briefly discuss some of the types of tests most commonly used.

In the methods of evaluation which determine the amount of foam generated in a glass cylinder or tube, either by shaking or the free fall of the detergent solution (1), it has been observed that the diameter of the vessel plays a large part in the lasting properties of the foam. Since the housewife is not confronted with this situation, it would appear wise to devise a test wherein this variable would not be present. Also in these tests the foam is shielded from circulating air, thus reducing the effects of evaporation which act upon foam under household-use conditions.

In order to circumvent these shortcomings several tests (2, 4) have been devised which utilize a high-speed beater to generate the foam. In these cases it has been possible to add soil to the solution under test, a condition which makes the results more realistic from the point of view of composition of the aqueous phase. Such a test has been used in our laboratory for preliminary screening of surfactants and detergent formulations since it adapts itself very well to the evaluation of small quantities. This is especially important when working with research-size samples. Although the results have been helpful in preliminary work, correlation with consumer-panel testing has not been altogether satisfactory. Most of the other mechanical methods encountered in the literature either lacked a satisfactory soil or required the design or purchase of specialized equipment not readily available to the average detergent laboratory.

Manual tests (2, 5, 6, 7) are also frequently used in which detergency and foaming properties are observed simultaneously. On the surface this would appear to be the ultimate type of test since these are the conditions under which the user makes his decision. However in order to provide a uniform deposit

of soil it is not possible to incorporate many of the ingredients encountered in practical conditions. Further, in order to distinguish small differences in formulations, it is necessary to provide a soil which is neither too easy nor too difficult to remove so that a satisfactory range of results is possible over a wide selection of detergent compositions. In these "semi-practical" or plate-wash tests the human error cannot be overlooked even if the same operator were to perform all of the tests.

All of the advantages and shortcomings of the above-mentioned methods were considered in the problem of designing the current test. It was deemed necessary to provide a procedure which would evaluate the foaming characteristics independent of soil removal, be reproducible, and, above all, correlate with consumer-acceptance tests. It was also considered desirable to be able to make a permanent photographic record which could be used as future reference since word descriptions of foam are quite inadequate in conveying the complete picture.

Experimental

Machine. A test embodying the above elements was developed utilizing a Dexter Twin-A-Matic¹ washer, which, although designed for household-laundry usage, can conveniently be used for foam evaluation without any mechanical adaptation. This machine is of the manually operated (wringer) type and possesses the unique feature of having two tubs of equal size, each containing an agitator. Both of the agitators are driven by the same crank shaft and consequently provide identical mechanical action for their respective solutions. The degree and type of agitation is quite similar to that of actual dishwashing in that the foam is generated by water in rapid motion at the air-water interface. A camera is mounted on a stand at an angle of 45° to the horizontal. This position permits both an over-all view of the foam and provides a convenient means of judging the relative foam heights.

Soil. The soil employed is similar to that used by Sanders and Knaggs (7) with minor variations and contains many of the common components found on soiled dishes. Although it is possible that this particular combination would not be found in any given type of food, it is sufficiently representative of the conditions that will be met in every-day use. The soil is readily dispersible by the action of the agitator and therefore acts continually on the lower surface of the foam layer throughout the test period.

Ingredients

- 150.0 g. Shortening (Swift's Jewel)
- 37.5 g. Flour (Pillsbury's Best Enriched)
- 7.5 g. Dried egg yolk
- 22.5 g. Corn starch
- 22.5 g. Margarine
- 22.5 g. Mayonnaise
- 22.5 g. Tomato juice
- 20.0 g. Higgin's India ink No. 4417

¹ Registered trade mark of the Dexter Company, Fairfield, Ia.

Mixing Steps. Weigh the shortening into a 2-liter metal beaker and heat on a hot plate to 180°F. Remove from hot plate, add flour, and stir until no lumps remain. Add the remaining ingredients in the order listed, thoroughly stirring each one into the mixture before adding the next. The mixture will still be liquid if the ingredients are added fairly rapidly. The beaker is then placed in a cold-water bath, and the contents are stirred until solidified sufficiently so that separation of the aqueous and oil components will not occur. The soil will remain solid at room temperature. (Note: The soil should be freshly prepared the day it is to be used.)

Testing Procedure. Water of desired hardness and temperature is placed in the washer, and the detergent is added. (The capacity of each tub is 58 liters when filled to the water-level line.) In most of our tests a temperature of 110°F. was used as representative of household conditions. The machine and timer are started simultaneously. After three minutes a picture is taken, and soil is added to each tub without stopping the agitation. Another photograph is taken two minutes later, then finally after 13 additional minutes, making a total agitation time of 18 minutes. Characteristically the foam builds up into a doughnut-shaped ring within the first minute. This ring usually breaks down gradually after the addition of the soil except in the case of exceptionally stable foams, in which case it will still be intact at the conclusion of the test. In the event that the breakdown occurs at a time not coincident with the pre-determined period of photographing, this time is recorded and also used as a criterion for rating the detergents. With a moderately good detergent a rich layer of foam will be present at the end of the test; with a relatively poor one almost no foam will be observed at this time.

Extreme care should be exercised in cleaning the washer, following each evaluation, in order to remove all traces of soil. A satisfactory method is to remove the agitators and wash the tubs with a good heavy-duty detergent. Rinsing should remove all of the foam.

Results and Discussion

The test was initially used for evaluating spray-dried detergents of the alkyl aryl sulfonate type. These detergents were used at a 0.1% level since a consumer research had indicated this to be a reasonable concentration. Early work revealed that 25 g. of the soil was a desirable quantity which would give the greatest degree of differentiation between relatively good and relatively poor products. More recent work with light-duty, liquid detergents has indicated a need for a lower concentration (0.05%) of this type of product. This may, in part, be caused by the presence of higher levels of foam modifiers in liquid detergents or by the unique property of the combination of ingredients used.

One of the variables which has a pronounced effect on foam volume and stability is the hardness of the water. For example, many dishwashing formulations which produce voluminous foam in hard or moderately hard water will exhibit foam characteristics in soft water that would be entirely unacceptable from the consumer's viewpoint. Typical areas in which this would be a major problem are parts of the Eastern seaboard and much of the Pacific Northwest. Such a condition would also exist in hard-water areas where water softeners of the ion exchange type are in widespread use. Figure 1 is a composite of the photographs at the various time-intervals of the test in which a typical alkyl aryl sulfonate detergent

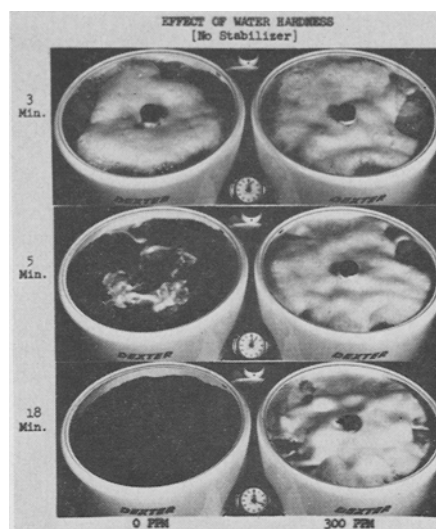


Fig. 1

without a foam modifier was evaluated in water of zero and 300 p.p.m. hardness. This clearly illustrates the difficulty that can be encountered because of water hardness alone.

Figure 2 shows the same basic formulation, to which has been added an amide type of foam modifier. The improvement has been made largely in the soft-water condition, and yet no detriment is noted in the hard water. The improvement found in this type of formulation has also been observed in typical light-duty, liquid dishwashing detergents.

In any laboratory evaluation of performance of detergents it is necessary that the results correlate with those observed by the ultimate user. If a good correlation exists, then expensive consumer-testing can be kept at a minimum. A comparatively large number of housewives (more than 1,800) participated in a blind product-test in which several products were checked for foam stability and detergency. Figure 3 illustrates the performance of two formulations in which there was no significant preference in the practical test, 1.05 to 1.00 (95% probability level). Examination of the foam pictures also indicates very little difference between detergent Formulas A and

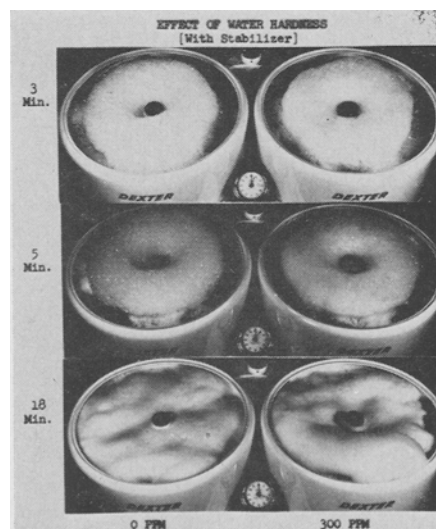


Fig. 2

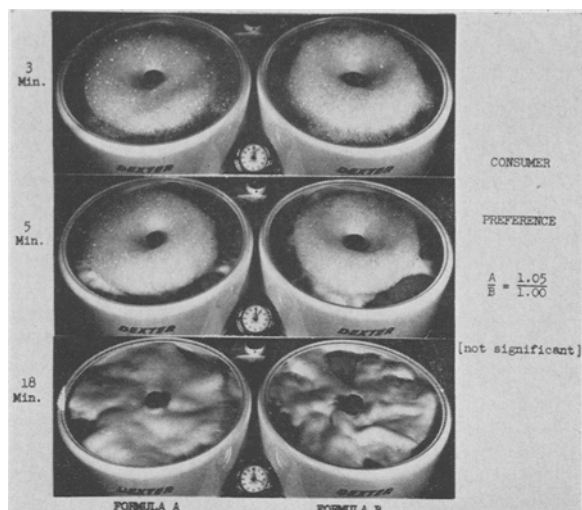


FIG. 3

B in either initial foam volume (3-min. photograph) or stability to soil (5- and 18-min. photographs). Figure 4 illustrates the performance of another variation, Formula C, when compared with the reference product, Formula A. In this case there was a sig-

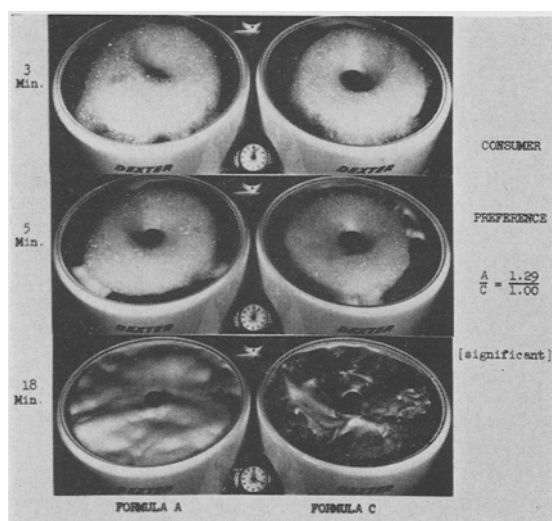


FIG. 4

nificant preference for the reference product, 1.29 to 1.00 (95% probability level). Definite performance differences are also apparent in the photograph. The initial foam build-up is somewhat lower, and the stability to soil is markedly poorer.

Summary

A technique utilizing a common household washer for the evaluation of the foaming properties of hand dishwashing detergents has been developed, which has been shown to correlate well with blind-product consumer-testing. In contrast with many tests now in use this procedure incorporates the use of a variety of soil ingredients which may be present in actual home and institutional dishwashing practice, namely, fats, egg, vegetable juice, starches, etc. The design of the test equipment makes possible the simultaneous observation of two products or formula variations, thereby permitting the use of a standard of measurement for relative comparisons. Photographing the test at designated time-intervals makes it possible to study the character of the foams after the completion of the evaluation. The test measures the properties of initial foam build-up and stability to soil and has shown good reproducibility.

Acknowledgment

The authors wish to express their appreciation to Robert C. Ferris, director of research, Purex Corporation Ltd., for his suggestions in the development of this test and to Jack H. Elliott for his work during the initial phases of evaluation.

REFERENCES

- Ross, J., and Miles, G. D., *Oil and Soap*, 18 (5), 99-102 (1941).
- Weeks, L. E., Harris, J. C., and Brown, E. L., *J. Am. Oil Chemists' Soc.*, 31, 254-257 (1954).
- Harris, J. C., *Soap and Sanitary Chemicals*, 28 (12), 45-47, 101 (1952); 29 (1), 42-44, 77 (1953).
- Sisley, J. P., Loury, M., *Soap and Chemical Specialties*, 31 (4), 44-46, 99 (1955).
- Mayhew, R. L., Jelinek, C. F., Stefcik, A., *Soap and Chemical Specialties*, 31 (7), 37-40, 167, 169 (1955).
- Mizuno, W. G., Lanners, F. T., and Wilson, J. L., *J. Am. Oil Chemists' Soc.*, 32, 437-441 (1955).
- Sanders, H. L., and Knaggs, E. A., *Soap and Sanitary Chemicals*, 29 (6), 45-48, 93 (1953).
- Miles, G. D., Ross, J., and Shedlovsky, L., *J. Am. Oil Chemists' Soc.*, 27, 268-273 (1950).
- Brown, A. G., Thuman, W. C., and McBain, J. W., *J. Colloid Sci.*, 8 (5), 491-507 (1953).
- Kurz, P. F., *Soap and Chemical Specialties*, 31 (10), 77, 79 (1955).
- Fineman, M. N., Greenwald, H. L., and Gebelein, C. G., *Soap and Chemical Specialties*, 31 (8), 43-46, 183 (1955); 31 (9), 50-52 (1955).

[Received November 5, 1956]

The Evaluation of Crude Glycerine

J. B. SEGUR, E. L. WHITTAKER, L. LASKIN, T. T. KANNO, and C. S. MINER JR.,
The Miner Laboratories,¹ Chicago, Illinois

CRUDE GLYCERINE is customarily examined by the Official Methods of the American Oil Chemists' Society (1). These analyses give the content of glycerol, ash, organic residue, alkali, and sodium chloride. The results give a general indication of the quality of the crude but do not ordinarily foretell certain difficulties that are sometimes met in refining the glycerine. The principal difficulties that

may be encountered are a) foaming during distillation, b) low yield of glycerine, c) a dark-colored distillate, difficult to bleach, and d) development of color or odor in the refined glycerine as it ages.

It was our aim in the work described below to find tests that would detect the trouble-making batches. Since a method of quantitative distillation was already available (2), our study was directed to the detection of difficulties other than low yield.

¹Now a division of Arthur D. Little Inc.