A Dynamic Foam Test for Evaluation of Hand Dishwashing Compositions[1]

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HAND-WASHING of dishes is generally accomplished by using a foaming type of detergent composition. The commonly desired requirements for hand-dishwashing compositions are high foam level during the course of the washing operation, stability of the foam to soils, proper emulsification and suspension of soil particles, and adequate cleansing of the dish or pan surfaces. Prevention of hard water film formation on dishes or glassware, prevention of redeposition of soil particles, and free and easy drainage are also required. Perhaps one of the more difficult aspects of this problem is maintenance of an adequate layer of foam, stable to soils such as greases, oils, and fats, and emulsification of these contaminants during the washing operation. A foaming type of composition must perform adequately in this respect, and a screening test which would reproducibly and rapidly evaluate this characteristic is desirable. Were it possible also to correlate such a foaming test with a reproducible dishwashing test, much uncertainty in laboratory evaluation would be removed and correlation with actual practice could then be determined.

No attempt will be made to review the numerous methods for foam measurement since most of them are mainly concerned with measurement in the absence of soiling elements. Mention should be made of the Ross-Miles (1) lather test, which is widely used throughout the industry for measuring the general lathering properties and lather stabilities of surfactants. This test does not show adequate correlation with plate wash tests. Furthermore it is difficult to run this test in the presence of soils and maintain uniform soil dispersion throughout the detergent solution. Dynamic foam tests, where foam is generated by blowing air through the detergent solution, were also inadequate for evaluation of hand-dishwashing compositions.

The housewife generally builds up a foam for dishwashing by running warm water with such force into her sink or dishpan that the detergent previously added is dissolved and foamed. Dishes are then manually washed until the suds either disappear or the soil is no longer easily removed. Loss of suds is primarily caused by the defoaming action of soils, such as greases, starch, and protein matter present on the soiled dishes. A test for foaming characteristics should measure rate of foam build-up, foaming capacity, and stability of the foam to soil. A reproducible method for developing the initial lather is a necessity and, in this case, is produced by a carefully speed-controlled mechanical stirrer.

Experimental

EQUIPMENT.

A. *Equipment for the Dynamic Foam Test.*

Cenco stirring motor, catalogue No. 18835
Propeller supplied with Eastern Industries Model 4 variable speed stirrer
Sola constant voltage transformer
2 variacs, 5-ampere capacity
1,000 ml. graduate cut down to 600 ml. volume
3,000 ml. beaker for water bath
Electric hot plate
300 p.p.m. hard water (60% calcium and 40% magnesium)

A diagram illustrating the stirring apparatus is presented in Figure 1. The speed of the stirring motor was maintained at 3,000 ± 50 r.p.m. by means of the voltage regulator and variac. A variac was also used to maintain the temperature of the water bath at 120° ± 2°F. on the hot plate.

DYNAMIC FOAM TEST APPARATUS

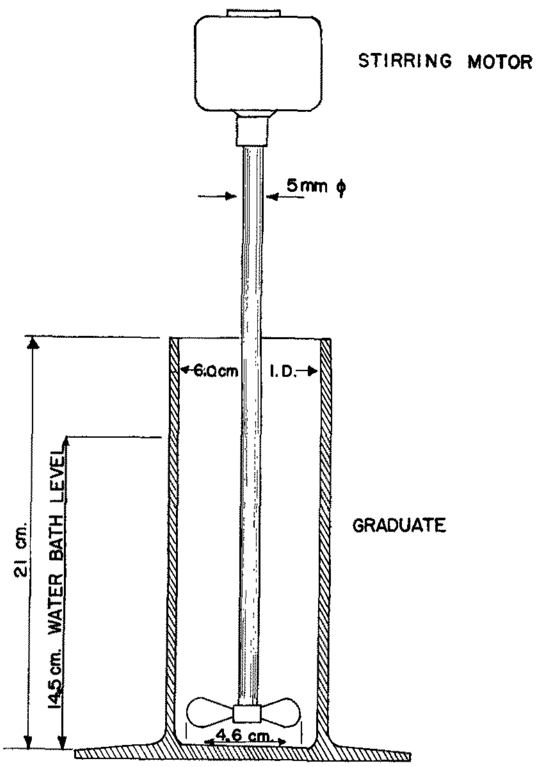

FIG. 1

B. *Equipment for the Semi-Practical Plate Wash Test.*

9-inch diameter plain white dinner plates
Oval-shaped dishpan (12 in. x 16 in. x 5 in. deep)
Dishcloth
Tub of 5-gal. capacity
¼-in.-mesh galvanized wire screen, 6 in. x 4 in.
Draining rack
Ultraviolet light

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TABLE I

Dynamic Foam Data

Detergent	Ml. Foam after X-Seconds of Stirring						Stability of Foam to Soil. Ml. Foam with X-g. Soil						
	30	60	90	120	150	300	0.1	0.2	0.3	0.4	0.5	0.6	0.7
Sodium Hexyl-4-Sulfophthalate	60	100	140	190[a]	215	270	270	40	30	20	15	10	
	65	115	185	210	230	265	260	50	35	25	20	15	10
	65	100	150	195	220	260	260	40	30	20	15	10	
	65	100	155	195	220	260	255	40	30	25	15	10+	10
	65	100	160	200	220	260	255	35	30	20	15	10+	10

[a] Foam build-up index $= \frac{190}{120} = 1.58$.

Soil used in the tests was a starch-grease type composed of 50% flour, 48% Crisco, 2% oleic acid, and 0.05% Fluorescent Green H. S., 185% (Wilmot and Cassidy Inc., Brooklyn, N. Y.). The dye was added as a tracer to detect degree of cleanliness of plates under ultraviolet light. This soil is believed to represent a composition which might commonly be encountered in practice. The soil was prepared by blending together the ingredients at 120°F. and mixing until a homogenous mixture resulted on cooling. The soil was kept refrigerated at 45°F. and used over a period of a month before preparing a fresh batch.

TEST PROCEDURES.

A. *Dynamic Foam Test.* Detergents were evaluated at 0.10% active concentration in 300 p.p.m. hard water. A volume of 100 ml. of detergent was preheated to 120° F. and transferred to the cylinder suspended in the water bath. The stirrer was then lowered into the graduated cylinder to a position centered at 13 mm. above the inside bottom of the cylinder. One minute after the solution was in the graduate, the stirrer was started and operated for 30 seconds. The solution was then allowed to stand for 30 seconds, and the 30-second stirring and 30-second rest periods were carried out until 150 seconds of stirring time had been completed. Foam volumes were recorded at the end of the rest periods. Following this, the foam level was recorded after 150 seconds of continuous additional stirring. A maximum foam volume was usually reached after 300 seconds of stirring. After 120 seconds of stirring the foam volume increase tended to level off, and the volume of foam recorded here divided by 120 was arbitrarily called the foam build-up index. Figure 2 shows a photograph of the apparatus after 300 seconds of stirring.

Following this, the stability of the foam to soil was determined. Portions of 0.100 g. of soil, weighed on 1-in. diameter glassine paper discs for convenience, were added to the detergent solution. After 5 minutes of continuous stirring the foam height was recorded, another 0.100 g. of soil was added, and then followed a 5-minute stirring period. Soil addition was continued until the foam height dropped to 10 ml. This end-point was selected since compositions tested showed a gradual break of foam to 10 ml., but foam for most products could not easily be reduced to zero, even by addition of very large amounts of

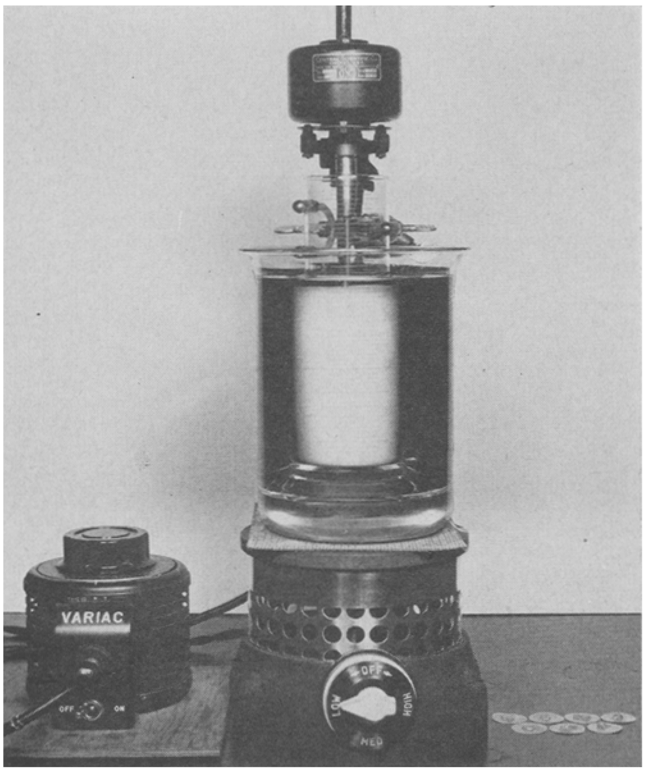

FIG. 2. Dynamic foam test at maximum foam volume. (Note soil on paper discs at lower right.)

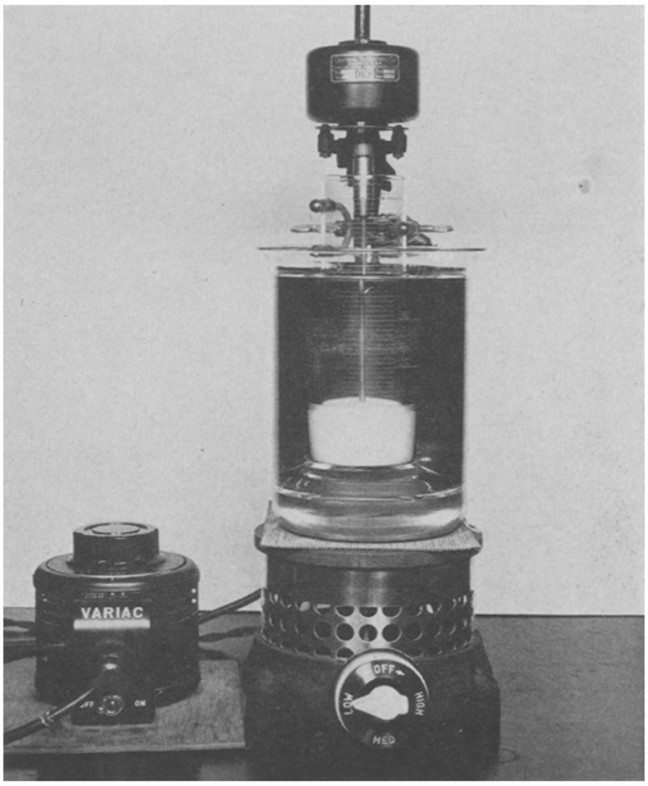

FIG. 3. Dynamic foam test at end-point. (Maximum soil added.)

soil, since soil itself will produce a slight froth. The appearance of the foam at this state is illustrated in Figure 3. Table I presents replicated dynamic foam data for a typical detergent composition. The method is found to have good reproducibility.

Some of the detergents exhibited good foam build-up indexes and had good stability to soil. Others were poor in both respects. A product which had a low foam build-up index and low foam volume after 300 seconds of stirring would be expected to show poor foam stability to soil. This was found to be true.

The dynamic foam test was used for screening detergents and detergent combinations, of which the better were put through a laboratory plate wash test.

B. *Semi-Practical Plate Wash Test.* Preparation of soiled plates consisted of applying 8.0 g. of the soil used in the dynamic test to each dinner plate with a spatula. The soil was spread over the plates evenly, and, after being stacked face up, the plates were ready for washing without further aging. Usually a series of about 50 plates were soiled at one time. The oval-shaped dishpan was filled with 6 liters of 300 p.p.m. hard water at an initial temperature of 115° ±1°F. The detergent was added in solid form for compositions readily soluble, and in solution form for others. In this test a standard detergent was run at sufficient concentration to wash 25 to 30 plates before the foam disappeared. To meet this, 0.10% active concentration of the best products was suitable.

After the detergent solution was in the dishpan, foam or suds were produced by manually agitating the solution with the ¼-in.-mesh galvanized wire screen for 30 seconds. Soiled plates were then washed with a dishcloth according to normal household procedure until all traces of soil were removed. No effort was made to apply pressure to the dishcloth for removing the soil. At the beginning of the test two soiled plates were placed in the dishpan, washed separately, rinsed in the tub containing 5 gallons of 300 p.p.m. hard water at 115°F., and then placed in the rack to drain dry. When the suds level had fallen appreciably, soiled plates were immersed and washed one at a time until the suds had just disappeared. At this point the test was then terminated, and the number of plates washed was recorded. A photograph of the plate wash test in progress is shown in Figure 4. Quality of soil removal from the plates washed was determined by examining the plates under ultraviolet light. Figure 5 shows a half-cleaned plate exposed to ultraviolet light.

FIG. 5. Appearance of half cleaned plate to ultraviolet light.

FIG. 4. Semipractical plate wash test.

Dishcloths and plates that were used in a test were thoroughly cleaned prior to the next test.

Test Data. Table II presents data for both dynamic and semi-practical tests.

Some correlation was evident in the maximum amount of soil added in the dynamic test and the number of plates washed in the semi-practical test. This might be expected since in both tests foam is generated, and then soil is added (on plates in the plate test) to the detergent solution until the foam disappears. Comparative tests were made on 19 detergent compositions. Figure 6 exhibits a graph of the number of plates washed plotted against maximum grams of soil added in the dynamic test.

Coefficient of correlation (2) for the test was 0.92, and the variation in the tests for 95% confidence limits was ± 3 plates and ± 0.15 g. of soil. A coefficient of 0.693 is required for a 99.9% level of confidence for the 19 samples tested.

TABLE II

Comparison of Dynamic Foam and Plate Wash Tests

No.	Detergent	Dynamic Foam Test: Foam Build-up Index	Dynamic Foam Test: Ml. Foam Volume at 300 Sec.	Foam Stability to Soil: Foam Vol. with X-g. Soil, 0.1 g.	Foam Stability to Soil: Foam Vol. with X-g. Soil, 0.2 g.	Foam Stability to Soil: Foam Vol. with X-g. Soil, 0.3 g.	Foam Stability to Soil: Max. Soil Added, g.	Plate Wash Test No. of Plates Washed
1	Sodium Hexyl-4-Sulfophthalate[a]	1.75	270	270	40	30	0.6	23
2	Sodium Salt of Sulfated Lorol[a]	2.08	265	265	260	115	0.7	25
3	Competitive Product-A (50% Active Content)	2.12	270	275	275	255	0.7	26
4	Competitive Product-B (37% Active Content)	1.58	260	100	35	20	0.6	20
5	Competitive Product-C (30% Active Content)	1.00	200	25	15	10	0.4	19

[a] Commercial products which had inorganic salts removed. Compositions were tested at 0.10% active content.

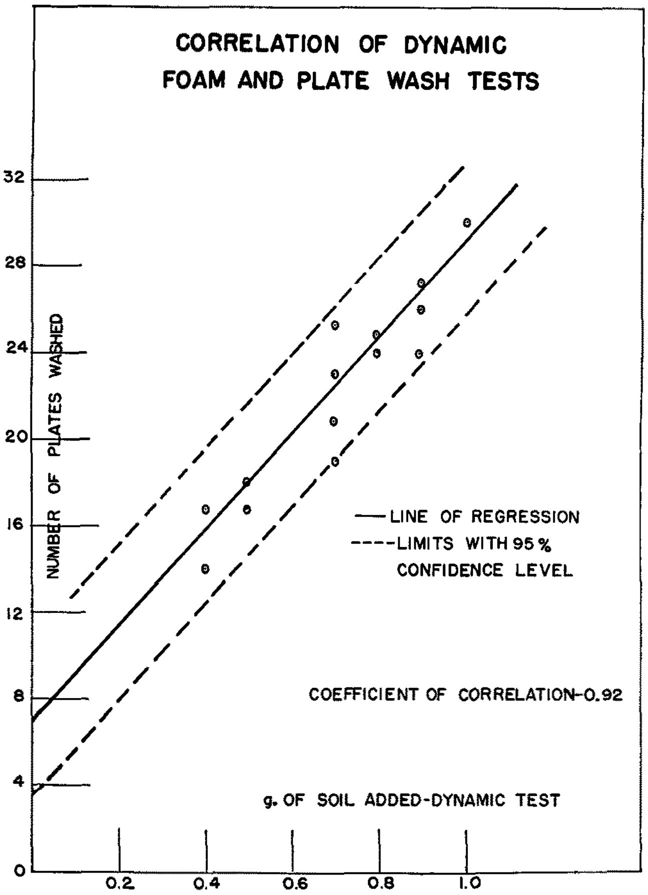

FIG. 6

The equation for the line of regression in Figure 6 is X_{1r} (No. of plates washed) $= 6.90 + 22.07\ X_2$ (g. of soil added). This represents the line of best fit for the set of points. By having determined the maximum amount of soil for the dynamic test, it would be possible for example to estimate how many plates the detergent will wash.

Discussion

Soil. The soil used caused sufficient defoaming to show differences in foam stabilities of detergent compositions and yet the defoaming action was not excessive. Other soils investigated, such as those high in free unsaturated fatty acids, caused too rapid defoaming action while, on the other hand, soils high in starch content had least effect on foam. A combination of 50% flour, 48% Crisco, and 2% oleic acid was found to provide a desirable degree of defoaming and also to represent a commonly encountered soil. Other soils should be evaluated, and some soil compositions to consider for future work are outlined in a review by Harris (3). Among these, combination soils and those of proteinaceous type would be of definite interest.

Correlation of Data. Analysis of data for the dynamic foam test and semipractical plate wash test show that there is excellent correlation between the two methods. The good reproducibility of the dynamic foam test makes it of interest for screening purposes. It is believed that further refinements in the plate wash test may help to reduce variations. Application of soil and washing plates entirely by mechanical means might minimize any variation on the part of the operator. Building up a standard height of foam prior to the test might also tend toward improvement.

The end-point of the semipractical plate wash test was characterized by the loss of suds on the solution surface. At this point all plates were free from fluorescence, a fact which indicates that they were clean and therefore that soil removal was complete. Since loss of suds is the practical end-point for hand washing of dishes, no attempt was made to ascertain the economical end-point.

In the development of a hand-dishwashing composition, screening tests such as those described here are usually followed by secondary semipractical or practical tests, product stability and shelf life tests, and eventually tests by a consumer panel.

Summary

A dynamic foam test for evaluation of hand-dishwashing compositions has been developed which gives good correlation with semipractical plate washing tests. Properties measured in the dynamic tests are foam build-up index, foaming capacity, and stability of foam to soil.

Acknowledgment

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