Determination of Detergency of Soap Products

The Report Published in January Oil & Fat Industries Is Supplemented by the Comments of Collaborators

In the January number of Oil & Fat Industries, L. F. Hoyt, Chairman of the American Oil Chemists Society Sub-Committee on Determination of Detergency, published a paper which might be designated as a progress report. Equally of interest are the comments of collaborators in the work, which are given below.

THERE was difficulty in soiling the cotton sheeting uniformly and to the proper shade. The removal of sizing by boiling with acid as advised, rather than by washing with hot soap solution is of questionable advantage. Filtration of the soiling mixture through sheeting from which the sizing had been removed was found to be unsatisfactory, since the pores soon became clogged with lampblack and after the first few minutes only a clear filterate came through. The use of a double thickness of cheesecloth seemed to take out any clots and to avoid the above difficulty, and hence cheesecloth was adopted in place of the cotton. During the actual soiling process, evaporation of the CCl_4 was very rapid, resulting in such progressive concentration of the solution as to make a uniform, equal soiling difficult in two successive attempts.

"Attempts on a second day to match the strips soiled the day before rarely resulted in getting the same shade, although the same formula of soiling mixture and as nearly as possible the same technique were used, and even though the numbers recorded by comparison with the standard paper or paste scales might be the same. The steps between successive paper or paste standards were greater than gradations in cloth quite noticeable to the eye. The India Ink Cloth Scale was found to be unsatisfactory, due both to the magnitude of the gradations, and to the difference in chroma between the India Ink soiling and that with the lampblack mixture. The difference in chroma was found to be less between the soiled cotton strips, on the one hand, and the paper or paste scales on the other; however, the difference in texture, in surface smoothness and reflection of light militated against the last two scales, and necessitated holding the objects to be compared at a distance of fifteen or twenty feet from the eye. When viewed at close range the cotton cloth seemed to match a much lighter paper or paste standard than when viewed at a distance.

"Since soiling on two successive days did not produce towels that were alike, even though checking the same scale numbers, the A.O.C.S. recommendations were deviated from, in that the pieces of cloth for the complete series of washes were taken from a single strip of cotton, soiled at one time; the pieces were therefore soiled exactly alike, but varied in age from 1 hour to 32 hours before washing.

Also when ironing 'with a warm iron,' the warmth of the iron is a variable factor. We experienced occasional spotting of the cloth when ironed, and in place of ironing we therefore spread the cloths flat in an oven at 130°C., and dried to a smooth condition.

"In all washes, distilled water was used both for the soap solution and for the rinse. Washing experiments in this laboratory for a number of years have clearly shown that the use of rinse water containing hardness would give a darker cloth after washing than when distilled water was used."

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"The results with these few tests we consider decidedly tentative and not in any way final. We are unable to draw any conclusions from our results thus far. More work is required.

Soil

The A. O. C. S. materials were employed. The soil was made up:

2 g. Lampblack
3 g. Tallow
5 g. Mineral Oil
2000 cc. CCl_4

Method of Soiling

"Strips of sheeting about 15 ft. long were run through the solution and then between the rollers of an ordinary clothes wringer. We found that the tension on the rollers made a difference in the color of the soiled cloth and that if the rollers were screwed down tightly and the cloth run through the solution and the rollers a second time, the soil was deposited more evenly on the cloth than if the strips were run through the rollers loosely adjusted only once. Our cloth was all soiled by putting through the rollers twice.

"We seem able to get the soiled cloths more closely uniform in respect to depth of color using the Ives Tint Photometer than when we depend entirely upon the Munsell Paper Scale; that is, we appear to be able to detect smaller differences in the color with the Photometer.

"From the little work we have done it seems that we get a more uniform soil of the cloth if we mix up the soil and allow it to stand for a day or two before soiling the cloth.

"In making further detergent tests our experience indicates that time would be saved and equally reliable results obtained if one 15-minute washing were made rather than three 5-minute washings."

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"On making the color readings of the cloth, we find that the color varies at different sections and it is hard to pick out the spot on the cloth that is of the average color to compare with the scale. We find that the strips of cloth wash whiter nearer the ends. We believe for this reason that it would be better to take photometer readings on each quarter of the cloth, folded as directed to form a piece 3" x 4½" and take the average of these readings. We found 8 per cent difference in these readings on the same piece of cloth."

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"We found that sizing is not removed from the cloth upon boiling for 5 minutes in dilute hydrochloric acid. We found it necessary to boil with acid and then alkali and then to wash with soap.

"We found that 4 grams of lampblack were required in your formula using x grams in 2 liters of carbon tetrachloride in order to give soiled cloth which matched your sample (i.e. S 48). Samples were soiled in this solution and

withdrawn from the soiling bath between 2 glass rods held together at one end by a rubber band and by hand at the other end. The solvent was evaporated by holding the cloth before an electric fan. This soiled cloth was pressed at once with a hot flat iron and the washings begun within an hour."

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"After some considerable work for the purpose of developing the proper technique it was found possible to duplicate the standard soil (S 48) very closely. The matching of color with the various scales, however, involves considerable personal element. We believe that an effort should be made to lessen this variable by a closer approach in the standards to the bluish grey of the soiled cloths."

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"Have carried this work a little further than was originally outlined; for example, have done the work with both distilled water and tap water rinse, also have read our washings on all three scales submitted.

"If some of the other committee members have used the tap water rinse, it would be interesting to note just what effect the total hardness of the rinse water, which varies in different cities, has to do with the resultant color of the cloth. Chicago tap water runs about 9.0 grains total hardness.

"In regard to color standards: We found that the pastes were the most satisfactory. Considerable complaint has been presented against these pastes due probably to the sheen and glossiness of the glass containing the paste but we found this of no objection whatsoever.

"In many cases there were cloths, when completely washed, that would show a difference in color to the eye, yet the reading on the scales would be the same, on account of lack of intermediate standards.

"This work has been very interesting and as it develops I feel that some valuable information and data will be compiled."

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"The samples of cloth colored by India ink were not as satisfactory as the colored papers, for the reason that the differences between each were too great to permit close evaluation. It seemed rather unfortunate that the paper standards had a bluish tint which made it necessary to match by intensity, rather than by exact shade of color.

The Soiling Solution: "We met with the difficulty that in following the method as given, the lampblack was retained on the filter cloths to a variable extent, and that which did not pass through was not well suspended. By using Dr. Walker's method of grinding the lampblack with the mineral oil before adding any CCl_4, a satisfactory suspension was obtained.

"We wish to comment on the soiling solution from the point of view of its analogy to dirt actually encountered in practice. Carbon is certainly a considerable factor in the dirt which has to be removed in the vicinity of industrial centers, but there are a great many localities where this kind of dirt is negligible and detergents are called upon to deal with silicious materials almost exclusively. Lampblack is convenient on account of its color, but it is not typical of silicious soil which may be deflocculated by detergents in a very different way. There are some colored materials which behave more nearly like silicious dirt than lampblack. Some work has

recently been done under Bancroft, using manganese dioxide. Bone black is another possibility, but perhaps the most nearly typical material that could be worked out on a color scale might be a raw umber.

"This study will ultimately be extended to detergents other than pure soap which, in our opinion, makes it desirable to select a soiling mixture as nearly typical as possible, rather than one which works well with soap but may give misleading results with other useful washing materials.

"It is known that detergency cannot be rightly appraised by chemical analysis or yet by measurements of surface tension, and until it is possible to measure and evaluate the various factors which contribute to detergency, we believe that the test should be made to simulate as closely as practicable the conditions of actual washing. Perhaps, this could best be done by standardizing a mixture of lampblack with some other highly colored substance which reacts more nearly like silicious dirt."

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"*Soil Solution*: After being advised that the original soil should be 25 per cent brightness (S 48 of the Munsell Scale) it was found practicable to make up the soil solution according to the directions given excepting that the solution could not be filtered through several thicknesses of washed sheeting. It was filtered through a No. 100 sieve.

"*Soiling*: The strips of sheeting, from which the sizing was removed, were immersed in the soil solution, allowed to dry by suspending in the air and finally placing in a drying chamber for a few minutes. No wringer or iron were used.

"*Washing*: The observation that soiled cloths several days old do not wash out as white as freshly soiled cloths, was confirmed. A difference seemed apparent between cloths soiled one day and washed the next, and cloths which were soiled and washed the same day.

"The speed of the washing machine was maintained at 250 r.p.m. —25 r.p.m. Temperatures were held to within 1° or 2° of those specified. Distilled water was used exclusively. After the first, third and fifth washes, four rinses were used. After thesecond and fourth wash only three rinses were used. The cloth was removed from the machine and allowed to dry partially by suspending in the air and then placed in the drying chamber for 10 or 15 minutes."

Discussion of Results

The incomplete and variable reports obtained from this year's work scarcely warrant drawing definite conclusions, although in the case of Tallow Soap the *average* results after the fifth washings indicate a definite improvement of detergency with increase in temperature, while with Olive Castile Soap the average results indicate very little change of detergent value with increase in the temperature of the washing test.

The results of this year's work, while admittedly incomplete and tentative, indicate that with the present form of detergency test (1) the *same* investigator finds very little difference between the soaps tested and (2) different investigators report quite variable results for the same soap.

It should be emphasized that although identical sets of materials and washing machines were used

by the various members of the committee, the technique of the involved and lengthy processes of soiling, repeated washings and rinsings and particularly the matching of color of the soiled and washed cloths unavoidably involves the personal equation to such an extent that it is not surprising that results reported by different investigators are not uniform.

Replies from a questionnaire addressed to members of the committee resulted in a practically unanimous choice (1) of the Munsell Paper Scale as against a standard scale of cloths soiled with India ink and (2) approval of 12 steps as in the present Munsell Scale as being a satisfactory number. Replies from some of the members who had opportunity to use sets of Dr. Walker's gray pastes showed a difference of opinion, about equally divided, as to whether these pastes were preferable to the Munsell Scale or not. It would seem advisable for members who have used the gray pastes to pass them on to other members of the committee for trial and comment.

The fact that the use of hard water for rinsing in washing tests would result in darker cloths than when distilled water is used, was confirmed by several other members of the committee. Hence, it is clear that to secure at all comparable results between collaborators, the use of distilled water in washing and rinsing should be adhered to unless it can be shown that the hardness of a particular water supply is such that no difference results when it is used in place of distilled water.

The cotton sheeting supplied by the chairman for this year's program unfortunately contained considerable starchy filler which was troublesome to remove before soiling as commented upon by some of the members of the committee. In future detergency work a fabric free from filler should be used to avoid difficulties of this character.

Judging by the comments of collaborators, the following factors may have influenced differences in results reported by investigators:

1. Difficulty in thoroughly removing starchy filler in sheeting supplied.
2. Variations in procedure of soiling, found necessary by collaborators to attain a soiled cloth of the prescribed shade.
3. Difference in chroma between the soiled fabric and the standard Munsell papers, coupled with the probability that various observers may not have the same degree of eye sensitivity in matching the shades of gray involved.
4. Possible effect of such factors as temperature of drying and ironing the soiled and washed cloths, and method of making up the soap solutions.

The chairman feels, and believes that other members of the committee likewise feel, that the work on determination of detergency should be continued. A start has been made but much intensive work remains to be done before it can be definitely concluded whether or not the type of detergency test on which we are working can be so perfected that in the hands of various investigators it will consistently differentiate the detergent value of soaps of widely different composition and properties.

The chairman wishes to express his sincere appreciation of the large amount of time and effort devoted to the work on detergency by the members of this year's committee and their assistants.