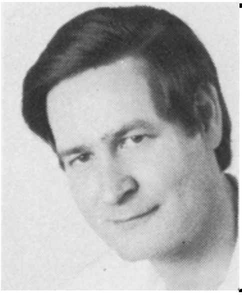

Evaluation of Detergents for Washing Fabrics

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ABSTRACT

Various methods are used today to check the washing effect of detergents. They differ mainly in whether the cleaning assessment is determined by the use of artificially soiled test fabrics, anonymous naturally soiled laundry in a single wash test, or new family bundles in multiple use and wash test series. Correlation of the information based on the actual field behavior of the different methods is discussed. Applicability of the different systems will depend on the special conditions which call for abbreviated procedures. Multiple use and wash tests are very close to practical use, give information about cumulative effects and thereby allow the detection of small differences. Their disadvantages are high costs and length of time for the test. For a realistic evaluation from a consumer viewpoint, they are of little significance without a ranking scale correlating to consumers' appraisal. Classification of tested products depends on the kinds of textiles in the family bundles which are used in the detergency evaluation. This factor is important in the detection of correlations between the two methods. For product evaluation and especially for overall quality ratings, the use of test fabrics is satisfactory if a carefully selected combination of these is chosen. Taking into consideration empirical data obtained with standard formulations, one can obtain satisfactory results corresponding to consumers' appraisals. Stain removal and soil redeposition have to be given the same weight as soil removal. An overall investigation of possible correlations is lacking so that some uncertainties still remain.

INTRODUCTION

The economic and technical value of a detergent can be determined only by an overall evaluation of its functions and properties in washing systems and in the environment. Toxicological, ecological, and performance tests are necessary prior to going from primary screening to actual field tests, test markets, and consumers' evaluations in order to introduce new products into the market. A multitude of test methods have been developed in order to check the washing effectiveness during the path from the initial idea to the marketable product.

In recent years the growing consumer awareness of knowing what he will get for his money plus intensifying competition have markedly increased the importance of testing of detergents by organizations outside of the detergent industry. Methods intended for use in the development of new products were often employed to assess the efficacy of commercial home laundry detergents without a critical examination of the scope and the limitations of these tests (1,2). To go to the other extreme, only actual field tests were considered valid to give sufficient information about quality and grading of detergents (3).

Any valid test method requires optimum correlation with results that the average consumer can observe in the home. Significant differences should be visible. On the other hand, differences that can be demonstrated only in idealized testing but cannot be noticed by the consumer should be discarded. A further test requirement is the necessity of obtaining results within a reasonable period a costs, e.g., it is of no practical value to have evaluation methods that take 3 months if the normal decision has to be made within a week or so. This is especially true for routine quality control and for routine buying decisions.

The Wäschereiforschung Krefeld is often confronted with a variety of short-term problems concerning the overall performance of washing powders. The known test methods are not always applicable to the specific problems. Abbreviated procedures, therefore, have to be used quite often, and the data have to be adjusted on the basis of results obtained with products already on the market.

TEST METHODS USED FOR THE EVALUATION OF PERFORMANCE

The important variables in testing the performance of detergents are discussed in the "Guidelines for Comparative Testing" (3). In this paper we will consider only the methods that are connected with the assessment of the washing effect. This does not mean that other properties are considered less important.

Various methods are used to check the washing effect (Table I). They range from pure laboratory tests with artificially soiled test fabrics in laboratory equipment, to mixed methods, to actual field evaluations. The method chosen in each case depends on the problem to be solved, availability of the detergent to be tested, money and time to be spent, and laboratory equipment and personnel available.

TEST METHODS EVALUATING THE NATURALLY SOILED ARTICLES

Table I shows a practical and realistic method using family bundles submitted to multiple use and wash cycles under controlled conditions (method 7). Small differences and cumulative effects can thus be detected. It has been published as an ASTM Tentative Method (4,5) in the U.S. and is being prepared for incorporation into the German standard for the assessment of washing machines (6). In order to compare more than three products, a complicated testing and evaluation system combined with statistical analysis is necessary. Special test schedules have been discussed extensively by Harder et al. (7,8). Visual evaluation combined with statistical analysis of the results will show significant differences but does not tell anything about their relevance to the consumer. Sommer and Milster (9,10), therefore, suggested a grey-scale standard which allows a more realistic assessment, especially when supplemented by instrumental measurements.

The main disadvantages of this method for routine testing are high costs and, more importantly, the long time required to obtain sufficient results. For comparison of more than three detergents, a very complicated test and evaluation system must be used, which is susceptible to many external influences. Furthermore, the evaluation obtained depends on, and therefore is relevant only for, the composition of the particular family bundles used in the test. Since only white textiles are normally used, and a very sophisticated comparison of identical but differently washed textiles is employed, this method can be regarded as over-sensitive compared to consumer judgments. The consumer normally is not in the same situation. Spot removal, for instance, will be of more importance to the consumer than established by this test.

Second, greying by dye transfer is excluded, but this negative side reaction plays a very important part in cumulative effects during actual washing (Fig. 1). In contrast to the opinion offered in the Guidelines (3), the human eye (11) can but seldom differentiate between this and soil redeposition, thus actual differences in the cleaning properties of detergents are evened out. Nevertheless, it is the most reproducible and realistic assessment.

Despite the overall advantages of the bundle test, methods 1 to 6 are quite important. Method 5 is as tedious and costly as 7 with the disadvantage that testers do not know the history of the textiles and furthermore lack information about additive effects; on the other hand, it is very close to consumer practice. Compositions of the load correspond better to normal use, and the procedure requires less time than method 7. In our experience this test can be simplified without losing too much information by making yes/no decisions instead of classification into a somewhat arbitrary cleaning scale. Influences due to the unknown previous history are larger than the inaccuracy caused by this coarse grading. However, the consumer normally judges in the same way.

Method 6, in my opinion, seems to be more useful in demonstrating large differences in cleaning efficiency rather than for practical evaluations, mainly because it is limited to only a few textiles (e.g., pillowcases) soiled very heavily and provides limited information about the washing effect. Similar arguments can be advanced concerning the towel test described by Vitale, Ross, and Schwartz (12) where a group of hand towels is soiled in a random manner in washroom usage, although cumulative effects may be detected.

LABORATORY METHOD IN THE LAUNDER-O-METER

The use of artificially soiled test pieces is the chief difference between methods 1 and 4 and the previously discussed methods. We interpret artificially soiled test pieces as textiles being fabricated under controlled conditions, soiled artificially with a known amount of a definite soil in a controlled process, irrespective of whether the soil itself might be a natural or artificial soil.

Use of artificially soiled test pieces to evaluate formulated detergents is highly questionable. Some workers have shown (13-16) that laboratory evaluation methods using artificially soiled test pieces show a considerable lack of agreement with relative performance in the laundry with natural soil. A realistic analysis of these publications concerning test conditions substantiates, however, that often planning and implementation of the experiments led to inaccurate correlation.

A complex mixture of variables influences visual difference in cleanliness obtained by two products as observed by the consumer. The total difference may be attributed to a summation of differences in soil removal, redeposition, bleaching effect, optical brightening, spot removal, and last but not least, as discussed, in greying due to dye transfer. Correlation between results obtained with artificially soiled test pieces and field tests, therefore, can be expected only when a combination of test swatches are included that take into consideration these major effects, and when the question of redeposition is not neglected. Experiments show that soil redeposition plays an important part and contributes substantially to the measured cumulative soiling. An investigation that includes these facets is still lacking so that

TABLE I

Methods for the Evaluation of Performance of Detergents

No.	Textiles	Apparatus	Multiwash	Correlation to practice	Information for further development	Cumulative effects	Cost	Time
1	Artificially soiled	Laboratory	No	*Fair* to low[a]	Fair to good[a]	No	Low	Low
2	Artificially soiled	Washing machine Clean load	No	*Fair* to low[a]	Fair to good[a]	No	Low	Medium
3	Artificially soiled	Washing machine Soiled load	No	Fair[a]	Fair to good[a]	Redeposition	Medium	Medium
4	Combinations of artificially soiled	Washing machine Soiled load	No	Fair to *good*[a]	Good[a]	Redeposition	Medium	Medium
5	Naturally soiled undefined	Washing machine	No	*Good*	Low	Redeposition	Very high	Long
6	Naturally soiled cut into parts (sector method)	Washing machine	No	*Fair* to good[a]	Limited	No	Medium	Medium
7	Defined family bundles	Washing	Yes	*Good* to very good[b]	Limited	Yes	Very high	Very long

[a]Depends on the selection of test pieces and problem to be solved, further on the procedure.
[b]Depends on the composition of family bundles.

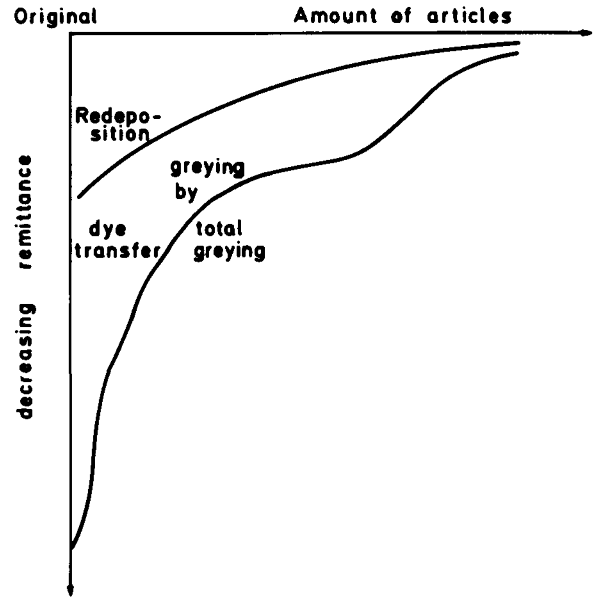

FIG. 1. Importance of dye transfer on greying during practical use according to (11).

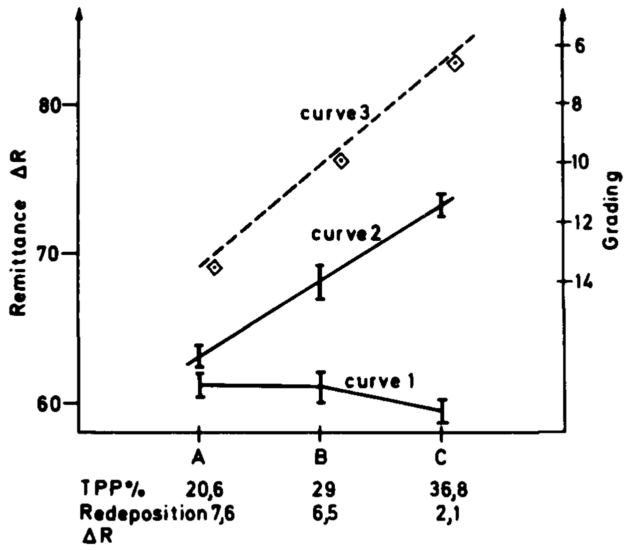

FIG. 2. Influence of phosphate content on washing effect (identical formulation). Curve 1: Launder-O-Meter, WFK test fabric, liquor-ratio 1:10, 5 g/1 detergent. Curve 2: Washing machine (drum-type), WFK test fabric, liquor-ratio 1:5, 5 g/1 detergent. Curve 3: Same washing machine, Bundle test, conditions as in Curve 2.

general statements about correlation with naturally soiled articles cannot be made.

Another problem area that is often neglected is the observance of identical experimental conditions, especially concerning mechanical action, liquor ratio, soil load, etc.

Figure 2 shows an example of a laboratory test with WFK soiled cotton test swatches (17,18), in a Launder-O-Meter (curve 1) compared to the same test in a commercial home washing machine (curve 2) and a bundle test in the same machine (curve 3). Results show a rather good correlation between curves 2 and 3 and a big deviation between 1 and either 2 or 3. This is due to the large effect of mechanical action on the cleaning efficiency. The Launder-O-Meter, as a closed system, is very sensitive to sudsing which will further reduce the already low mechanical action. In our opinion wrong interpretations due to situations like this occur more often than realized. Launder-O-Meter tests, therefore, need a very careful analysis of possible problems and should be used only when sufficient foam stabilizer is used. Furthermore, the normally prepared 4 x 4 in. swatches should be substituted by smaller ones (2 to 2.5 in.) in order to obtain a lower standard deviation.

Cumulative soiling effects cannot be detected. Some modifications, therefore, were proposed (19-21) with a series of successive soilings, and washings. To me a major problem in these experiments is the choice and the application of the soil, a problem which still has to be solved.

For special tests, in particular when developing new builders, it is advantageous to add Ca-containing soiled test fabrics which release hardness during the cleaning process and thus imitate normal water hardening by the laundry load.

In spite of all of these modifications and precautionary measures, it has to be kept in mind that the limitations of such laboratory test methods can hardly be overcome sufficiently to permit statements relating to actual laundry use.

PRACTICAL WASHING ASSESSMENT BY USING ARTIFICIALLY SOILED TEST FABRICS

For the evaluation of the performance of washing powders, artificially soiled test fabrics have been used for quite a long time. Commercial washing machines are loaded with carefully selected genuinely soiled laundry as the test pieces and with artificially soiled and unsoiled standard fabrics as check test pieces. Many products developed with the help of these simple methods are produced and kept on the market. On the other hand, quite a lot of money and time is spent by detergent producers and testing associations to assess products by tests close to actual practice or in field tests. These tests account for differences of opinion held at the present time, and this situation also demonstrates the necessity for a fundamental investigation of possible correlations and limitations. It is still lacking and, in my opinion, there are some difficulties that have to be solved before this task can be initiated.

One of the main problems is the selection of a representative field test which will give a reliable and quantitative determination. We did some preliminary tests comparing our standard test procedures with those conducted with actual home soiled laundry. Results in Figure 2 could be interpreted as if good correlation exists between the bundle

TABLE II

Evaluation of Heavy Duty Washing Powders at 60 C (Wash-and-Wear Program), Artificially Soiled Test Pieces Compared to Naturally Soiled Mens' Shirt (Polyester/Cotton [PES/CO] 65/35, Wash-and-Wear Finish)

Product	% TPP	Cleaning effect (ΔR)			Redeposition (ΔR)		Evaluation PES/CO shirts (25 wash-and-wear cycles)[a]	
		Cotton	PES/CO	PES/CO wash and wear	Cotton	PES/CO	Collar % clean	Front side ΔR
A	38	72.6 ± 0.1	59.6 ± 0.3	69.6 ± 0.3	2.9	0.0	70	83.6 ± 0.6
B	19	65.8 ± 0.7	54.1 ± 0.6	61.6 ± 0.7	5.5	2.5	40	81.8 ± 0.7
C	16	64.1 ± 0.8	50.0 ± 0.5	62.3 ± 1.2	6.8	3.8	35	81.3 ± 0.6

[a]Worn 2 days.

TABLE III

Evaluation of Heavy Duty Washing Powders at 95 C
Artificially Soiled Test Pieces Compared to a Single Wash Test with Anonymous Family Bundles
and the Sector Method (Soiled Pillow cases Cut in Parts)

Product	Cleaning effect ΔR		Redeposition ΔR	Tea ΔR	Blood	Evaluation of laundry % clean	Sector method[a]	Ash %
	Cotton (WFK)	EMPA						
A	69.7 ± 0.5	53.5 ± 0.6	3.4	83.8 ± 0.9	86.2 ± 1.0	98	26	0.4
B	67.7 ± 1.1	51.4 ± 0.7	10.0	80.9 ± 0.9	82.3 ± 0.8	94.5	58	10.8
C	66.6 ± 0.8	51.7 ± 0.7	5.7	80.7 ± 1.3	81.2 ± 0.6	96	56	11.8
LSD						0.3		

[a]Clean = 1; dirty = 2.

TABLE IV

Evaluation of Heavy Duty Washing Powders at 95 C
Artificially Soiled Test Pieces Compared to Single Wash Test with Anonymous Family Bundles (Yes/No Method)

Product	WFK Test fabric		Red wine	Visual grading					Family bundles	
	Cleaning performance	Redeposition		Coffee	Blood	Gravy	Cacao	Egg	Σ	Visual % clean
A	73.8 ± 0.3	4.5	2	1.5	1	1.5	1	2	9	92
B	69.7 ± 0.4	5.5	3	3	3	3	2.5	2	16.5	85
C	71.7 ± 0.5	3.0	1	1.5	2	1.5	2.5	2	10.5	90
LSD										1.0

test and results obtained with Krefeld test fabrics using an anonymous laundry bundle as a load. But it could be easily demonstrated that this result was partly due to the fact that the textiles employed in the family bundles were prone to contact soiling with pigments and sebum. Composition and application of the artificial soil used was very close to this soil. Second, soil redeposition and soil retention are influenced by the formulation chosen. On the other hand, this positive result may support some optimism in finding an acceptable correlation by selecting a combination of test fabrics according to problems to be solved.

Similarly, analogous results were found in another test series where the cleaning effect of three differently formulated detergents was evaluated with naturally soiled mens' shirts in a 60 C wash-and-wear cycle by a multiwash test technique (Table II). The correlation obtained is similarly explained by pointing out that the main idea in development of Krefeld test fabrics was a design to imitate the dirt on the inner side of a collar, a very difficult cleaning problem. This limits the applicability of the test results. Table II also shows the necessity of choosing the proper fabric since corresponding data were obtained only with soiled finished polyester/cotton

Tables III and IV show comparisons of a single wash assessment with anonymous family bundles which corresponded to normal family loads with respect to the types and amounts of textiles. Here some deviations from the results obtained with WFK test fabrics can be observed. Table III shows no differences between products B and C either with the soiled test pieces or with the sector method. This is probably due to the exceptionally high redeposition of product B. The unexpected large difference between B and C in Table IV can be explained by large differences in spot removal. This was visually confirmed by the panel.

Some indications for a satisfactory correlation between bundle tests and assessment with artificially soiled test pieces were also found by Sommer and Milster (10) when a combination of different fabrics was used and redeposition effects were included. The main problem is to give proper weight to the differences in results and to grade the value of reflectance data obtained, or whatever other measurement method is used, on a realistically acceptable scale. Thus, all testing with soiled test pieces has to correlate with actual field tests.

On the other hand, since we have a practical and theoretically well developed realistic test method in the bundle test, we are often inclined to overlook that here the problem of correct weighting of cleaning effects on different textiles and realistic grading are as important as in the case discussed above.

Numerous results (10,19,20) support these findings and illustrate the problems of a reasonable correlation test. Detergents might be rated quite differently on soiled linen hand towels than on terry hand towels, although both types of textiles were used by the same family during the same test.

Long experience has shown that the evaluation of the cleaning properties of detergents with the aid of artificially soiled test fabrics can be satisfactorily utilized if the results are carefully watched and interpreted. At present it is the only way of making a fast decision for routine quality control and is a means of making purchasing decisions on the part of chain stores. The following preliminary conditions have to be fulfilled:

1. Domestic washing machines which can be exactly controlled must be used. At least a temperature-time graph should be plotted so that larger differences in voltage, water intake, and/or the temperature of the cold water can be detected. These will greatly influence results by extending the washing cycle of automatic machines and thereby increasing the mechanical energy input. Incrustations on the electric heaters are of minor importance (22). Well selected naturally soiled laundry should be used as a load.
2. A set of artificially soiled test fabrics should be employed. The textile substrates have to be selected in accordance with the washing program to be examined. As a minimum, one of each of the following types should be included: a test fabric based on pigment/natural fat and mineral oil soil; one based on the same soil but sensitive to bleach (e.g., cocoa); soiled test pieces sensitive to enzymatic activity; fabrics to determine redeposition tendencies (standard fabrics, terry cloth), and to determine also bleaching efficiency, incrustation, and fabric damage. A variety of stains has to be included. Practicable results can only be assured if this combination is regularly checked with detergents of known performance differences by washing in a standard washing machine (23,24).

When the above precautions were observed, a realistic assessment of detergents was possible provided redeposition and spot removal were given the same weighting as soil removal and fair to good correlations to the rankings of the latest "Siftung Warentest" evaluation (25) could thus be obtained.

Until comprehensive investigation has been conducted, some uncertainties will remain. Performance tests employing artificially soiled fabrics, therefore, should be used in the described manner only if required for valid reasons and bundle or field tests cannot be conducted. On the other hand, except for cumulative effects, the additional performance information given by the bundle test should not be overemphasized at the moment, in my opinion, since quite a lot of work, especially that of correct weighing of the results, still has to be done (10).

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