Test Method Utilizing Dry Natural Dirt for Evaluating Laundry Detergents

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ABSTRACT

A laboratory test has been developed for screening detergent compositions with respect to the removal of natural dry particulate soil (dirt). The fabrics are soiled uniformly with dirt obtained from a suitable area and then laundered under controlled conditions in a Tergotometer. The total soil removal is determined instrumentally. The test enables the researcher to check the effects of many variables, such as temperature, water hardness, builders, fabric, etc., with a minimum amount of preparation and time. The soil removal data is analyzed by a suitable statistical method, and the results are good indicators of practical performance.

INTRODUCTION

Researchers have been seeking a simple, single laboratory detergency test that will adequately reflect the performance of a laundry product under the varied conditions of use which are met in practice. Because of the numerous types of soils and fabrics that one encounters in a wash, no single laboratory test can be expected to predict performance results under all possible soiling conditions.

Tests utilizing natural and synthetic sebaceous soil have been developed. The Trowbridge test (1,2) uses natural soil found on the skin. Spangler's tests (3,4) deal with the removal of artificial sebum. Because these tests use soils that may be found on laundry, they can predict the outcome of a practical test when such soils are predominant. However, soils other than sebaceous type may be present on soiled laundry. Most prevalent and very apparent is the ordinary dirt found in the backyard, garden, athletic field, or on the streets. The mechanism by which such soils find their way onto fabrics in the everyday world is very simple. For example: (A) a child playing baseball slides into a base; (B) the gardener rubs his hands onto his trouser or shirt; or (C) a boy, after playing with dirt, "washes" and dries his hands on a clean towel. It is obvious that the list can be expanded easily.

The most disheartening aspect of ordinary dirt is that it usually is easily visible on the external surface of a garment or item. Unless one uses an efficient detergent system, natural dirt may not be removed easily. To evaluate the effect of detergent products in removing dirt from fabrics, we have developed a simple, rapid laboratory test which correlates well with results of practical laundering studies. The test involves the use of a Schiefer Abrasion machine (5,6) which is shown in Figure 1 and dirt from the local area. However, dirt from any part of the country can be substituted for the Piscataway, N.J. soil we use.

The soil is air-dried to constant wt and sieved through a 200 mesh screen without grinding the rock-type materials. A small volume of dirt is rubbed onto the fabric by means of a nylon bristle brush attachment on the Schiefer Abrader. The reflectance value of the soiled area is determined before and after washing. The results can be analyzed by an appropriate statistical method.

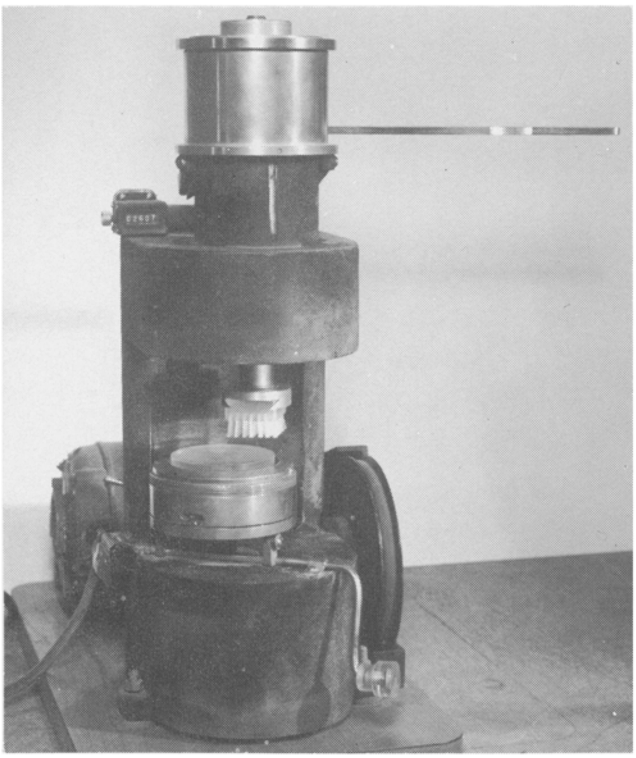

FIG. 1. Schiefer Abrashion machine.

EXPERIMENTAL PROCEDURES

Apparatus

Equipment used included the following: Schiefer Abrasion Test Machine (Frazier Precision Instrument Co., Gaithersburg, Md.), Gardner Reflectometer (Gardner Laboratory, Bethesda, Md.), and Tergotometer (United States Testing Co., Hoboken, N.J.).

Soil

Dirt from any appropriate region can be used, but we worked with soil from the Piscataway, N.J. area. The dirt is

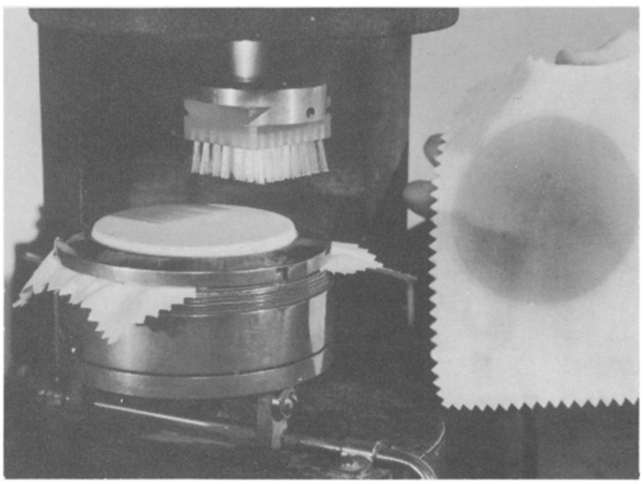

FIG. 2. A clean swatch mounted on Schiefer abrader and a soiled swatch.

air-dried to constant wt and sieved through a 200 mesh screen to remove large particles. A partical analysis of Piscataway soil is given in Table I.

Procedure

A 1 lb wt was added to the top of the Abrasion Machine. A 4 x 6 in. swatch (cotton, nylon, polyester, etc.) was mounted on the lower rotating disc of the machine (Fig. 2).

The swatch is secured in place with the appropriate top plate. A scoop (0.4 ml) of dirt is added onto the swatch directly below the nylon bristle brush. The brush then is lowered to the fabric surface, the machine turned on, and the brush allowed to revolve for 50 revolutions. The machine is stopped, the soiled swatch removed, and any loose dirt shaken off. The operation is repeated until 24 swatches have been soiled. The swatches are distributed randomly into four equal piles. The swatches in each pile are numbered so that they can be identifiied throughout the entire study. The initial reflectance value (Rdi) of the soiled circular area of each swatch is determined on a Gardner Reflectometer model AC-3 or a similar instrument. The swatches (6/treatment) are washed for 10 min at 100 rpm in a Tergotometer using the desired detergent product or system, concentration, and conditions. At the end of the wash cycle, the wash water is decanted, the swatches removed, and the excess liquid squeezed out by hand. A liter of clean water (100 F for hot water conditions and 70 F for cold water washing) is added to each bucket. The swatches are replaced in the same bucket in which they had been washed originally. The Tergotometer is allowed to agitate for 2 min, the swatches removed, excess water squeezed out, and the swatches dried in a conventional home dryer. After drying, the reflectance values of the soiled areas of each swatch are reread. The final Rdf values are recorded, and the change in reflectance (ΔRd) is calculated.

TABLE I

Partial Analyses of the Piscataway Soil

Elements	Percent
Fe	2.0
Ca	0.3
Al	2.9
Mg	0.6
Si as SiO_2	65.0
Mn	500 ppm
Cu	50 ppm
Na	600 ppm
K	50 ppm
Ether soluble	2.0

The data may be reported as percent soil removal or, by applying a suitable statistical method, it is possible to determine significant differences among wash treatments.

In our study, we have treated the data as a simple analysis of variance for two or more (preferably four) treatments (buckets) with six replications (swatches) within each bucket. A Newman-Keuls test is used to determine significant differences between means (7).

The raw data and results of statistical analyses from two separate, four treatment studies is shown in Tables II and III. Significant differences is determined by a Newman-Keuls test. The mean ΔRd values for the four detergents are

TABLE II

80 x 80 Cotton[a]

Instrumental (reflectance) data

Unsoiled Rd value = 92.1

Product J				Product K			
Swatch	Rdi	Rdf	ΔRd	Swatch	Rdi	Rdf	ΔRd
1	32.9	77.0	44.1	1	31.1	77.5	46.4
2	32.3	77.3	45.0	2	32.2	76.4	44.2
3	31.2	77.7	46.5	3	30.2	76.4	46.2
4	31.8	79.2	47.4	4	32.2	76.0	43.8
5	33.1	78.2	45.1	5	31.9	75.5	43.6
6	32.7	78.4	45.7	6	30.9	75.0	44.1
Product L				Product M			
Swatch	Rdi	Rdf	ΔRd	Swatch	Rdi	Rdf	ΔRd
1	34.1	75.6	41.5	1	32.4	60.9	28.5
2	31.6	73.4	41.8	2	32.8	60.0	27.2
3	31.3	75.5	44.2	3	32.7	61.2	28.5
4	32.2	74.1	41.9	4	31.6	57.3	25.7
5	31.6	75.5	43.9	5	32.7	59.7	27.0
6	31.3	72.6	41.3	6	32.5	59.5	27.0

Newman-Keuls test of significance

Source	DF	Sum of squares	Mean square	F ratio
Treatments	3	1324.60160	441.53385	310.68
Residual	20	28.42326	1.42116	

Newman-Keuls test

Range	2	3	4
Significant difference at .05P	1.44	1.74	1.93
Significant difference at .01P	1.96	2.26	2.44

J	K	L	M
45.63	44.72	42.43	27.32

At .05P

At .01P

[a]Rdi = initial reflectance value, Rdf = final reflectance value, and DF = degrees of freedom.

TABLE III

Dacron-Cotton with Permanent Press Finish[a]

Instrumental (reflectance) data

Unsoiled Rd value = 90.4

Product J				Produck K			
Swatch	Rdi	Rdf	ΔRd	Swatch	Rdi	Rdf	ΔRd
1	32.8	78.1	45.3	1	31.5	76.9	45.4
2	31.0	80.6	49.6	2	31.4	76.2	44.8
3	32.0	80.5	48.5	3	30.8	75.8	45.0
4	33.1	80.4	47.3	4	33.6	76.5	42.9
5	32.9	78.8	45.9	5	32.7	75.6	42.9
6	33.1	80.7	47.6	6	32.5	76.3	43.8

Product L				Product M			
Swatch	Rdi	Rdf	ΔRd	Swatch	Rdi	Rdf	ΔRd
1	34.2	78.6	44.4	1	32.4	67.9	35.5
2	31.2	76.7	45.5	2	33.4	65.7	32.3
3	31.0	75.9	44.9	3	33.1	66.4	33.3
4	30.2	75.2	45.0	4	33.2	67.7	34.5
5	32.6	75.2	42.6	5	32.1	67.8	35.7
6	30.5	74.6	44.1	6	31.8	68.2	36.4

Newman-Keuls test of significance

Source	DF	Sum of squares	Mean square	F ratio
Treatments	3	552.60992	184.20331	102.04
Residual	20	36.10327	1.80516	

Newman-Keuls test

Range	2	3	4
Significant difference at .05P	1.62	1.97	2.17
Significant difference at .01P	2.20	2.55	2.75

J	L	K	M
47.37	44.42	44.13	34.62

At .05P __ __

At .01P __ __ .

[a]Rdi = initial reflectance value, Rdf = final reflectance value, and DF = degrees of freedom.

TABLE IV

Treatments in Both Tests[a]

Product code	Surfactant type	Builder type
J	Anionic	Phosphate
K	Anionic	Nonphosphate
L	Anionic	Nonphosphate
M	Nonionic	Nonphosphate

[a]Concentration, as recommended by the manufacturer. Water, 120 F, 150 ppm hardness.

TABLE V

Intercorrelation Matrix

Detergent products	A	B	C	D
A	---	0.9997	0.9978	0.9994
B		---	0.9973	0.9988
C			---	0.9970
D				---

listed in order of magnitude. Means which are not underlined by the same line are found to be significantly different at the specified (95% or 99%) confidence level. The treatments in both tests are given in Table IV.

The only difference between the two tests is the type of soiled fabric used. In Table II, the data were collected from 80 x 80 cotton (Test fabric style 400). The data in Table III were obtained when dacron-cotton with permanent press finish (Testfabric's, style 7406WRL) was used.

RESULTS AND DISCUSSION

Standard Deviation

From 8 independent replicate determinations (separate wash runs), the standard deviation of the individual values (as listed in Figures 3-6 and Table V) is estimated with 7° of freedom to be ±1.18 Rd units.

Correlation between Different Sources of Dirt

Our studies have been run mostly with dirt from the Piscataway, N.J. area. To determine if correlation exists between soils from other parts of the country, we evaluated dirt from nine different regions. Soiled 80 x 80 cotton swatches were washed in 120 F, 150 ppm water with detergent products A, B, C, and D which had been used at recommended concentrations. The relationship between soil removal (ΔRd) and the source of the dirt is shown in Figure 3.

Visual inspection indicates that the products do rank in the same order with the different soils. The matrix of intercorrelations (8) for the data (mean ΔRd) illustrated in Figure 3 is shown in Table V.

A correlation value of 1.0000 indicates a perfect relationship between two factors. A value of 0.00 would have shown no relationship between variables. Our coefficient values are very close to 1.00. A correlation coefficient based upon 9 soils with N-2 or 7° of freedom is significant at the 99% confidence level when the observed value is

TABLE VI

Correlation of Dirt and Bundle Tests

Test	Formulation comparisons[a]	Dirt test mean ΔRd	Bundle test % preference votes
I	Anionic STPP-built detergent (A)	42.8	46.2
	vs		
	Anionic STPP-built detergent (B)	43.5	43.1
II	Anionic STPP-built detergent (A)	42.0	50.0
	vs		
	Nonionic Na_2CO_3-built detergent (C)	30.0	31.0
III	Anionic STPP-built detergent (A)	42.0	67.0
	vs		
	Nonionic no builder liquid detergent (D)	32.0	24.0
IV	Anionic STPP-built detergent (A)	45	51.4
	vs		
	Anionic K pyrophosphate-built liquid detergent (E)	34	25.7
	vs		
	Nonionic K pyrophosphate-built liquid detergent (F)	32	22.9

[a]STPP = sodium tripolyphosphate.

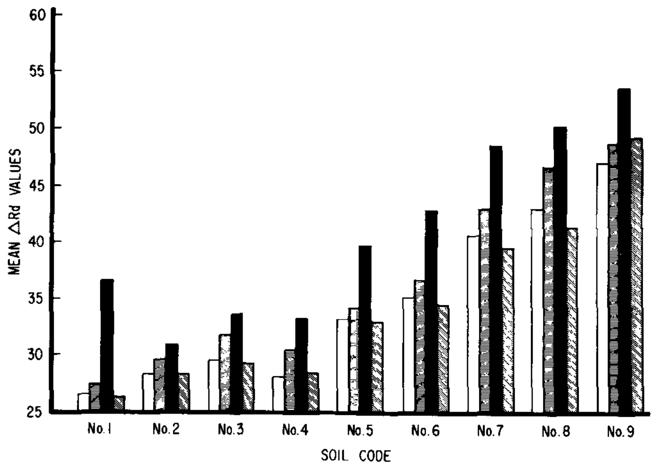

FIG. 3. Relationship between dirt source and soil removal (ΔRd); using concentrations as recommended by manufacturer, 120 F, 150 ppm. No. 1 = California, No. 2 = Missouri, No. 3 = Arizona, No. 4 = Georgia, No. 5 = Tennessee, No. 6 = Kentucky, No. 7 = Wisconsin, No. 8 = Pisataway, N.J. and No. 9 = East Keansburg, N.J. Products: □ = A, ▦ = B, ■ = C, and ▨ = D.

greater than 0.80. In other words, we could have used any one of the 9 soils and arrived at the same conclusions.

Correlation of Natural Dirt Test and Bundle Test

To check the correlation of the Natural Dirt test with the Bundle test (9), experimental runs were made in which the identical commercial detergent products were evaluated by both test methods. In these studies, the products were used at the concentrations recommended by the manufacturer. The water temperature was 120 F and hardness 150 ppm. Although various types of fabrics could have been used, we chose to work only with 80 x 80 cotton. The results of these studies are shown in Table VI.

The ability of the two test methods to rank the detergent products in the same order indicate that there is good correlation between them. However, results of a Bundle test may not always agree with the Natural Dirt test. Disagreement can occur when: (A) the laundry is not soiled or only very lightly soiled or (B) there is a big difference in brightener levels between the products being tested.

Effect of Water Hardness

To determine the sensitivity of the test, three different types of commercially available products were evaluated at four different hardnesses (Table VII). Water hardness, expressed as $CaCO_3$, was derived from a stock solution of

TABLE VII

Commercial Detergent Products

Product code	Type of surfactant	Builder type
F	Anionic	Phosphate
G	Nonionic	Phosphate
H	Nonionic	Nonphosphate

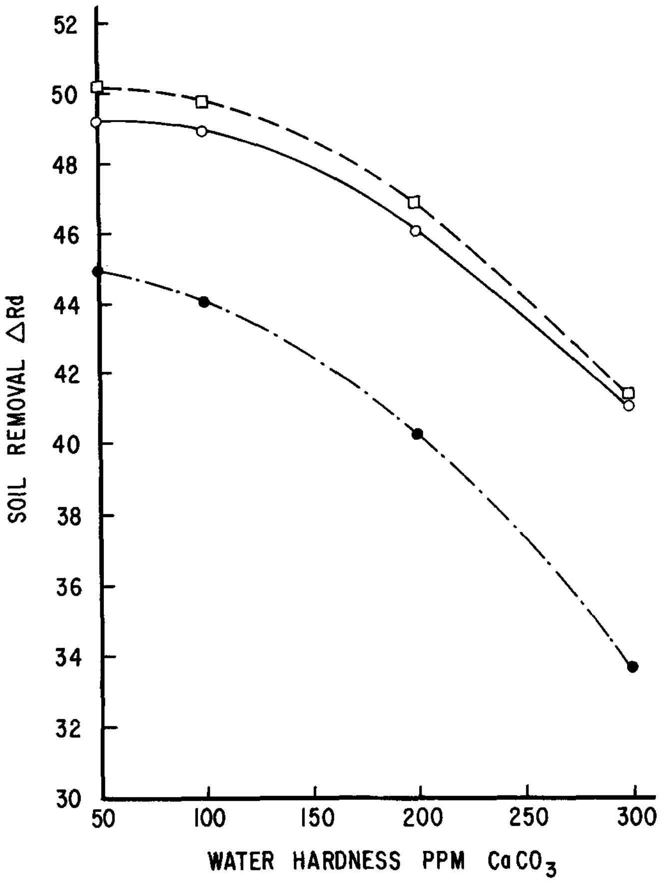

FIG. 4. Effect of water hardness upon soil removal; 120 F, using concentration as recommended by manufacturer. ○ = Product F, □ = product G, and ● = product H.

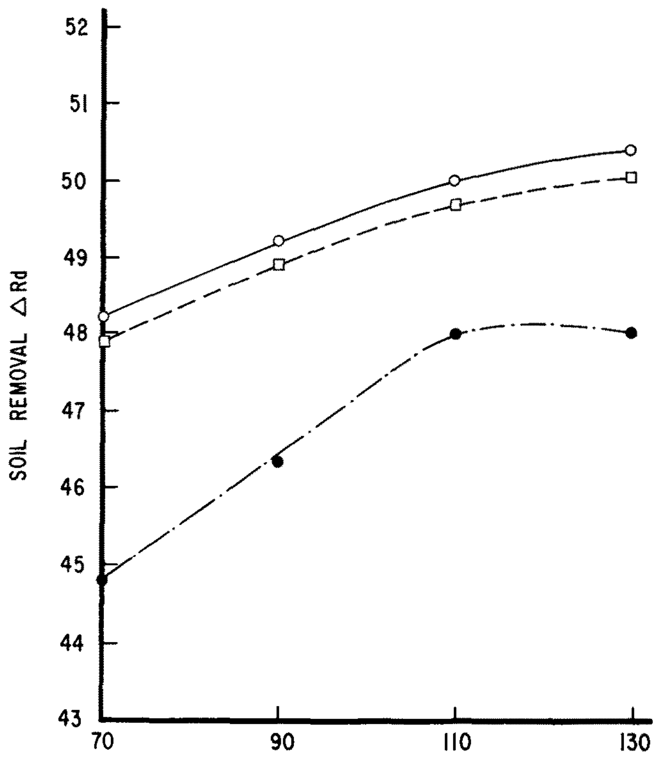

FIG. 5. Effect of water temperature upon soil removal; 150 ppm water, using concentration as recommended by detergent manufacturer. ○ = Product F, □ = product G, and ● = product H.

calcium and magnesium chloride having a 0.15/0.10 Ca^{++}/Mg^{++} molar ratio.

The two products were used at the concentrations recommended by the manufacturer. Soiled 80 x 80 cotton swatches were washed in 120 F water, and the hardness was varied between 50-300 ppm. The effect of variation in water hardness upon removal of natural dirt from cotton fabric is illustrated in Figure 4.

The results show that hardness has a similar negative effect upon all three products. There are no significant differences in performance between the two phosphate-built products F and G, but both are significantly different from the nonphosphate detergent H.

Effect of Temperature

Commercial products F, G, and H, which were described above, were evaluated at 4 different temperatures in 150 ppm water. The effect of varying temperatures upon soil removal is illustrated in Figure 5.

Soil removal improves as temperatures increase. Maximum soil removal seems to take place at ca. 110 F. Analysis of the raw data show no significant difference in soil removal performance between products F and G over the entire range of temperatures studied. However, the two phosphate detergents are found to be significantly different from the nonphosphate product.

Effect of Concentration

Products F, G, and H were compared to each other at 5 different concentrations in 120 F, 150 ppm water. The products were deliberately under-used to strain the systems.

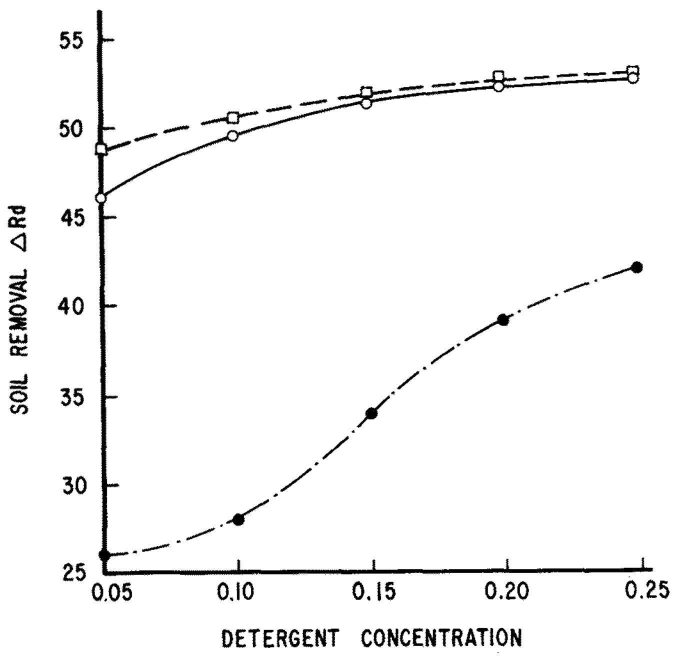

FIG. 6. Effect of concentration upon soil removal; 120 F, 150 ppm. ○ = Product F, □ = product G, and ● = product H.

The effect of varying the detergent concentration upon soil removal is illustrated in Figure 6.

Analyses show that, at 0.05% concentration, all 3 products are statistically different from each other. However, from a concentration of ca. 0.10% and higher, there are no performance differences between products F and G, but both are significantly different from H over the entire concentration range studied.

The results and conclusions can vary when different surfactant systems are used. Likewise, finishes and combination of fibers can alter the situation. Nevertheless, it is believed that the tool described can complement present day procedures in arriving at a true detergency comparison.

ACKNOWLEDGMENTS

J. Hauschild questioned the utility of available standard soiled fabrics in the assessment of performance of new detergent systems and initiation of detergency studies with natural clay soils. J.R. Trowbridge provided helpful suggestions and aid in statistically analyzing the raw data. K. Fitzsimmons and her Practical Laundry Laboratory staff conducted the Bundle test studies.

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[Received November 15, 1974]