A Wet Method for Pure Particulate Soiling of Test Cloths for Detergency Screening Tests

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ABSTRACT

A procedure is presented for soiling detergency test cloths with particulate soil. These cloths offer advantages over most commercial soiled cloths and laboratory soiling procedures in that there is no oily material necessary for dispersion and thereby the testor obtains results which are specific to particulate soil alone. The procedure involves a wet soiling technique, followed by drying and sorting for subsequent detergency evaluation. The soil composition includes Bandyblack clay, iron silicate, and iron oxide. Detergency results are discussed.

INTRODUCTION

Investigators in the field have needed soiled test cloths for detergency screening tests which will discriminate among the major formulation parameters such as type and amount of active, original water hardness, washing time, temperature, and others. Unfortunately, no single type of soiled test swatch gives response to all the differences which can be important.

A large variety of test cloths are commercially available (1-7), and many others are routinely prepared by test laboratories (8-10), both domestically and abroad. Test cloths differ as to type and amount of soil and method of soil application. For example, Kissa has described techniques for oily soil application (11) and oil/particulate soil application (12). Most of these procedures utilize a combination of particulate and oily matter.

There is a definite need for evaluation of particulate soiled material alone. Kissa (13) has reported a procedure for application of airborne particulate, and Inamorato et al. (14) have recently published another procedure for application of dry particulate soil by abrasion. Both procedures are tedious and require expensive equipment. Schwartz and Berch (15) discussed detergency screening tests using swatches soiled in wet slurry using vacuum cleaner dust. We have developed a procedure for the wet particulate soiling of detergency test cloths which allows multiple-swatch soiling in a single batch. Cloths soiled by this procedure are generally uniform in reflectance. Sorting soiled cloths by reflectance value before detergency testing results in excellent precision of testing. This procedure is the subject of this paper.

EXPERIMENTAL PROCEDURES

Materials

The cloth used in development of this procedure was obtained from Testfabrics, Inc. Cloth swatches are die-cut to give swatches with pinked edges of ca. 3-1/4 x 4-1/2 in. Bandyblack clay is available from H.C. Spinks Clay Co., Inc. (Paris, TN 38242); iron oxide is C.P. reagent grade; "frit" is iron silicate, 40% Fe, available from Frit Industries, Inc. (PO Box 1324, Ozark, AL 36360). Frit is prepared for use in this procedure by washing twice with ethanol, drying at ambient temperature in a vacuum oven, screened -140 mesh, pulverized in a Bleuler mill to screen -200 mesh.

Apparatus

An old, top loading manual washing machine, which would contain 30.0 liters of water heated to 120 (± 2) F, is used for a standard soiling (1,000 swatches). Small batches of wet soiled test cloths can be prepared in a standard Terg-O-Tometer, from U.S. Testing Co. Water is brought to temperature outside the washer. In the large (1,000 swatch) soiling batch, temperature is maintained by two 500-watt immersion heaters driven by an SCR temperature controller of in-house design.

Following the soiling procedure, soiled test swatches are dried with a large commercial print dryer adapted for this purpose. A very large SCR temperature controller maintains temperature of the print dryer drum at 170 (± 2) F. A household flour sifter is used to add soil to the agitated water (see Procedure). A constant temperature bath which will supply sufficient water for the soiling and rinse processes is necessary, as well as a suitable reflectometer for sorting soiled swatches. We presently use a Gardner XL-10A color difference meter with Adams-Smith interface for an RCA time-share computer terminal with paper tape attachment.

Procedure

To 30.0 liters of heated, agitated water add 3,000 g Bandyblack Research Clay, 3.00 g iron oxide, and 150 g prepared frit. This particulate soil is added through the household flour sifter to provide a well-dispersed suspension. Since the iron oxide and "frit" are relatively minor components of the particulate soil, these should be mixed into the Bandyblack clay before being sifted. The soil is agitated for 30 min to produce a uniform suspension, and ca. 1,000 swatches are dumped into the slurry. The swatches are agitated in the soil suspension for 20 min at constant 120 F; then they are removed quickly with gentle wringing action and allowed to drain on a rack covered with nylon netting.

The soil bath is drained, rinsed well, and an additional 30.0 liters of fresh water at 120 F is added. The soiled swatches are rinsed for 2 min, removed, and allowed to

TABLE I

Typical Reflectance Values for Particulate Soiled Cloths

Material	Unsoiled reflectance values	Reflectance values (R_d)				
		Number of soiled swatches	High	Low	Mean	SD
Cotton	88.2	957	54.5	49.7	52.1	0.76
Permanent press	86.5	1,011	56.1	51.2	53.7	0.88

drain again. The rinse procedure is repeated for a total of two rinses, and the cloths are print dried at 170 (± 10) F between paper toweling.

Cloths are sorted into groups differing by no more than 0.8 R_d units. Detergency screening tests utilize only swatches from a particular soiling batch and from a particular sorted group for comparative studies.

RESULTS

Results of two types of materials soiled by this method are given in Table I.

Soiling Results

Since in a normal distribution 95% of samples fall within ± 2 standard deviations, it can be seen from the above data that, after sorting, 95% of these cloths should fit into four groups differing by increments of 0.8 R_d units. Thus, this low standard deviation is an indicator of the uniformity of within-batch soiling, which determines the relative efficiency and reliability of the soiling. Obviously, the narrower the distribution range of reflectance values, the more swatches of a particular group are available for comparative testing from a given soiling batch.

Detergency Results

These particulate soiled cloths are used in our standard detergency tests (9). Separate tests include particulate soiled cloth by this method and oily soiled test swatches soiled by our adaptation of the Spangler office sebum soil method (8). This practice is necessary because of differences in detergency performance seen with different surfactants on different soil types.

Swatches soiled by this procedure can be used in multiple-cycle testing (10) if necessary. If multiple-cycle testing is desired, the test swatches are soiled on a large batch scale and sorted as described above for the first cycle. Cloths for subsequent cycles are soiled in a Terg-O-Tometer with proportionate water:Bandyblack clay:iron oxide:frit ratios.

Detergency test results with this wet-particulate-soiled method show generally excellent precision. We evaluate precision by the statistical method of H.O. Hartley (16). Low Hartley differences further substantiate the reliability of the soiling procedure.

Two examples of precision in detergency screening tests using particulate soiled swatches prepared by this method are given in Table II. Test 1 shows the least significant difference for a four-formulation test, and Test 2 shows the same indicator for an eight-formulation comparison. This precision is comparable to that found with our Spangler sebum soil procedure.

TABLE II

Statistical Evaluation of Detergency Tests Using Particulate Soiled[a] Cloths

Test	Formulations compared	Test material	Least significant differences
1	4	Cotton	0.89
		Permanent press	0.57
		Dacron	1.05
2	8	Cotton	0.70
		Permanent press	0.84

[a]CONOCO particulate soil.

DISCUSSION

The need for totally particulate soil test swatches is becoming more necessary in today's variety of complex formulating problems, We have shown a procedure for wet particulate soiling in multiple-swatch batches. This procedure is seen to be reproducible as indicated by standard deviation of soiled cloths and by precision of detergency test results. Such soiled swatches can be used for detergency screening tests in conjunction with other tests using oily soiled swatches for a thorough evaluation of detergency test parameters.

ACKNOWLEDGMENTS

Laboratory work was done by L.L. Cooper, P.R. Deichman, M.J. Fields, and D.L. Heppler.

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[Received December 22, 1975]