Comparison of Soil-Deposition and Redeposition Tests in Evaluating Drycleaning Detergents

WILLIAM H. SMITH, MANFRED WENTZ, and ALBERT R. MARTIN,
National Institute of Drycleaning, Silver Spring, Maryland

Abstract

A method for testing drycleaning detergents for their ability to inhibit soil redeposition is described. It involves the measurement of soil transfer from a soiled to a clean fabric. The customary practice of using suspensions of a model soil in a detergent solution is a soil-deposition test and does not give results comparable with the soil-redeposition test.

The major argument against redeposition tests in the past has been that they do not permit two detergents to be compared at the same soil concentration in the suspension. It is therefore argued that redeposition tests "stack the cards" against better detergents because soil must be removed before it can be redeposited. Present results refute this argument and show that detergents exhibiting low soil-removal generally show high graying and vice versa. These two qualities either correlate or they are two aspects of the same quality.

A possible explanation for the difference between the two test procedures is that the degree of dispersion of soil is much greater in the redeposition test.

Introduction

Soil redeposition during laundering or drycleaning occurs when soil that is removed from a soiled fabric becomes attached again to the surface of the textile fibers. This is particularly objectionable when the soil is redeposited on the white or pastel-colored areas of garments.

Since poor suspending power of detergents for insoluble soil has appeared to be one of the principal causes of this phenomenon, it was quite natural that most laboratory test methods for soil redeposition have been essentially soil-suspension tests. In performing these tests, workers first prepared dispersions of some model soil (most frequently carbon black) in the detergent solution and then added unsoiled fabrics to the suspension. After a period of agitation they removed the fabric samples and determined the decrease in reflectance.

In such tests there is difficulty in reproducing the kind of suspension one gets in actual laundering or drycleaning operations. There can be other problems involving unrealistic behavior of the model soil. Most soil redeposition, or "graying" as it is called, appears to be caused by colloidally dispersed particles, ranging from 2 μ down into the millimicron range. These particles are tiny enough to deposit firmly on the surface of textile fibers which are of the order of 10 μ in diameter (1).

Colloidal dispersions however do not form spontaneously, and their degree of dispersion depends on the method of preparation, particularly on the degree of liquid shear used in preparing the dispersion (2) and on the formation of a protective coating about the dispersed particles to prevent flocculation as soon as the mechanical agitation ceases.

It is not believed that the same kind of dispersion of a model soil results when the soil is removed from a soiled fabric as when bulk soil is added to a cleaning solution and stirred; hence "redeposition" tests based on the latter procedure are misleading, as Hensley has already shown (3).

The purpose of this paper is to offer additional evidence in support of Hensley as well as to provide a soil-redeposition test method that is more realistic than suspension tests. The test procedure involves soiling fabrics with a dry, insoluble model soil, then placing the soiled fabric and an unsoiled fabric together in the same cleaning bath. All soil that redeposits must come from the soiled fabric, as is the case in actual laundering or drycleaning.

This type of test is not new but has always been successfully attacked by the criticism that it "stacks the cards" against good detergents because they remove more soil, therefore there is more soil in suspension to be redeposited. The logic of this criticism has always seemed so obvious that it has never been examined experimentally as well as it should have been. However logical this objection may seem, colloid chemistry does not always follow the logical rules of Aristotle.

This laboratory for many years has been running cleaning performance tests in hundreds of drycleaning plants. These tests always include both soil-removal and soil-redeposition samples. This experience has led to the conclusion that high soil-removal is generally accompanied by low graying. This is not a new idea, as Wedell (4) of Böhme Fettchemie demonstrated several years ago. As a result of the work of Wedell, Hensley, and that described in this paper, objections to redeposition tests are not considered by the authors to be valid any longer.

Experimental Section

Model Soil Materials

Fabrics are soiled by applying a model soil to them in the form of a dry powder. In this work a rug soil was obtained from commercial rug-cleaning plants, generally referred to as "rug-beater soil." It is equivalent to vacuum-cleaner soil except that it is more representative because it is a mixed sample from many different rugs.

Before use it is screened through a 325-mesh screen to remove lint, trash, and coarse particles. It is a natural soil, similar to that normally found in outerwear garments. After screening it is a tan-colored, free-flowing powder. Fig. 1 is a photomicrograph of a typical specimen.

In our research projects there have been frequent experiments with model soils that are better characterized and more uniform. Among these is "Air Cleaner Test Dust," sold by A. C. Spark Plug Division of General Motors as a standard test material for air filters. Another is cerium oxide polishing powder, an extremely uniform powder sold by the Grace Division

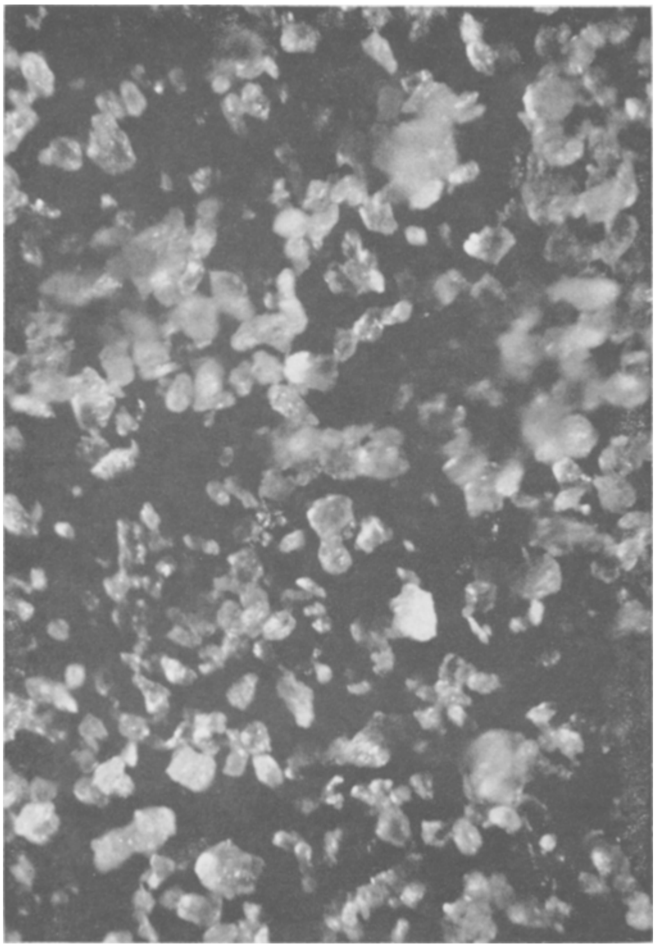

FIG. 1. Rug-beater soil, magnified 100 times.

of the Davison Chemical Company. More than 90% of this material is in the two- to three-micron range.

In general, it was found that rug-beater soil behaves in a manner similar to that of the other materials, and it has the advantage of higher tinctoral power for fabrics. Carbon black has too much tinctoral power, however, and mere traces of it can cause serious graying.

Soiling Procedure

An 80 × 80 cotton print cloth is used in this test. To insure uniform soiling the fabric is cut into 4 × 4 in. swatches with a dry weight of about 1 g each. Before soiling, the swatches are tumbled with cool air for 10 min and placed immediately in the soiling tumbler.

This is a 12 × 12-in. stainless steel cylinder with three 2-in. ribs, which operates at 48 rpm. It is loaded with 150 g of powdered soil and 1,000 g of #4372 fine "bird grit." The purpose of the sand is to keep the soil particles well dispersed and to bring them into close contact with the fiber surfaces. The sand acts somewhat like a ball mill toward the soil. Some 350 g of cotton are placed in the tumbler with the charge of soil and sand and tumbled together for 40 min. The ratio of soil to fabric and the tumble time are arbitrary. These conditions achieve a reflectance of 60 ± 2%.

After the swatches are removed, they are placed in a drying tumbler and tumbled for one hour in cold air to remove all loosely adhering soil and sand. This procedure results in a uniformly soiled fabric. The soil which remains shows no tendency to "crock off."

Next the reflectance of the samples is determined using six fabric thicknesses to screen out the background. The samples are then stored in a desiccator at room temperature and 75% RH until used. Unsoiled swatches are stored under the same conditions.

Redeposition Test Procedure

The redeposition tests are run in 500 ml steel Launder-O-Meter canisters. The normal procedure is to use 150 ml of a 1% v/v detergent solution in the appropriate solvent (perchlorethylene or Stoddard solvent). We use 30 stainless steel balls and six 1 g swatches (4 × 4-in.), three of which are soiled and three unsoiled. The canisters are tumbled for one hour in the Launder-O-Meter at room temperature.

The canisters are all removed at the same time, and the entire contents are poured into a Buchner funnel. As the swatches drain, they are picked up with tongs and rinsed manually in two changes of clean perchlorethylene. This rinse is essential because there is a rather high concentration of soil. During draining the swatches will filter some of this soil out of suspension so they need to be rinsed.

After rinsing, the swatches are air-dried at room temperature. Finally, the reflectance of both the white and soiled fabrics is measured. The decrease in percentage reflectance (green filter) by the unsoiled fabric is defined as the "graying." The blue filter reflectance is also measured to determine whether any serious yellowing has occurred or not.

With each set of detergents a comparison test is also run with a standard or reference detergent. We use "Aerosol OT" for the purpose, not because it is an outstandingly good detergent but because it is a pure compound as well as an anionic drycleaning detergent of the sulfonate type. It has been widely used in drycleaning research for these reasons. We have selected a soiled fabric load high enough to cause Aerosol OT to give poor results. This gives superior detergents sufficient leeway to demonstrate their effectiveness without question.

The final reflectance of the soiled fabric is also recorded as an index of soil removal; the higher the reflectance, the better the soil removal, of course. This is a good index of the soil-removal power of the detergent although it is not linear. Kubelka-Munk calculations of soil removal are not valid with this kind of soil. The tinctoral value of the soil is enhanced by exposure to the detergent solution because of better dispersion. Therefore the soil remaining on the fabric, or redepositing on it, causes a greater effect on reflectance than the same quantity of soil when it is applied dry.

The drop in reflectance of the unsoiled fabric and increase in reflectance of the soiled fabric during cleaning are used to compute two quality index numbers that are called the "Graying Index" and the "Cleaning Index." These are computed by using the corresponding values for the reference detergent. The Graying Index is the ratio of the percentage of graying of the test detergent to the percentage of graying with the reference detergent. The Cleaning Index is the ratio of the increase in reflectance of the soiled cloth in the test detergent solution to that observed with the reference detergent. Thus the lower the Graying Index, the better is the detergent, and the higher the Cleaning Index the better.

Soil-Deposition Tests

These are run in the same manner as the redeposition

TABLE I

Ten Best and Ten Poorest of 44 Detergents Tested by Laboratory Procedure

Rank	Type	Graying index	Cleaning index
	Best		
1	"Ampholytic"	0.62	2.33
2	Amine sulfonate	0.40	2.14
3	Sorbitan fatty acid ester	0.41	2.02
4	Amine sulfonate	0.32	1.82
5	Sulfonate blend	0.46	1.78
6	Sulfonate blend	0.54	1.76
7	Anionic-Nonionic blend	0.70	1.67
8	Petroleum sulfonate	0.53	1.62
9	Anionic-Nonionic blend	0.72	1.62
10	Phosphate ester	0.78	1.54
	Poorest		
35	Anionic-Nonionic blend	1.21	0.88
36	Amine sulfonate	1.34	0.88
37	Anionic-Nonionic blend	1.37	0.85
38	Petroleum sulfonate	1.24	0.82
39	Amine sulfonate	1.24	0.82
40	Amine sulfonate	1.15	0.81
41	Anionic-Nonionic blend	1.07	0.79
42	Amine sulfonate	1.24	0.71
43	Ethoxylated alkyl phenol	1.40	0.65
44	Ethoxylated alkyl phenol	1.43	0.62
Reference	Sulfosuccinate	1.00	1.00

tests except that no soiled fabrics are used. Instead a weighed quantity of soil is dispersed into the detergent solution, then six unsoiled fabrics are added. The deposition of soil on the fabrics is measured by reflectance in the same manner as described above.

Comparison of Removal and Redeposition

Launder-O-Meter Experiments. The redeposition test procedure was used on a total of 44 commercial drycleaning detergents. The quality indices of each were computed, and they were ranked according to the Cleaning Index. For the purposes of this paper the 10 detergents showing the highest cleaning indices and the 10 having the lowest were selected (Table I). It will be noted that all detergents showing high soil-removal also show a low Graying Index. Conversely each detergent showing a low Cleaning Index exhibited a high Graying Index.

Tests in Drycleaning Plants. The rug-soiled cotton fabric has been used together with unsoiled samples in Cleaning Performance Test Service for about 2 years. Data have now been obtained for thousands of tests in commercial drycleaning plants all over the United States. There are data on removal of the rug soil and data on graying for the same cleaning bath. The

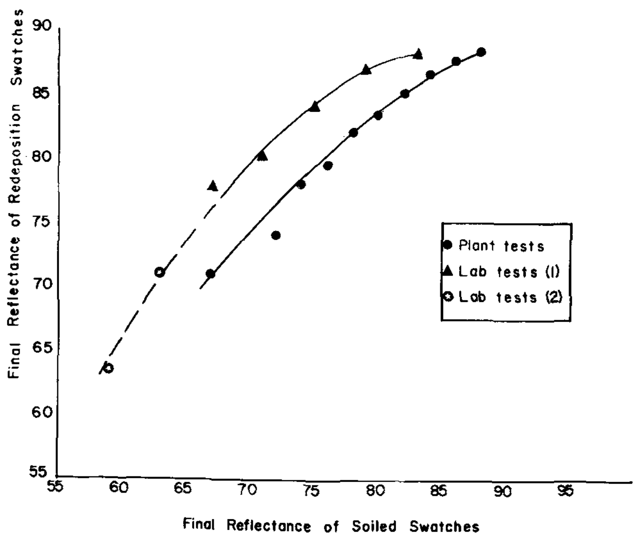

FIG. 2. Comparison of graying tests with plant tests.

TABLE II

Soil-Deposition Test Compared with Soil-Redeposition Test

Experiment	Deposition tests		Redeposition tests	
	1	2	3	4
Wt. of fabric load (g)	10.7	10.6	10.1	10.2
Total soil (mg)	66.0	136.6	66.0	136.6
Corrected for solubles (mg)	62.7	130.0	62.7	130.0
Soil suspended (mg)	51.5	99.8	39.9	75.6
Corrected for lint (mg)	45.4	93.8	33.8	69.5
% Soil suspended	72.4	72.0	54.0	53.5
% Reflectance, original	92.7	93.5	93.4	92.7
% Reflectance, final	84.0	81.3	71.2	63.5
% Graying	8.7	12.2	22.2	29.2
% Reflectance soiled swatches, final	....	.....	63.2	59.2

graying results from natural garment soil, of course, not from the rug-soil. It is interesting, therefore, to compare the graying obtained in commercial machines with the rug-soil removal under the same conditions and to compare these data with those obtained in the Launder-O-Meter. There is one common factor, removal of rug soil. Against this common variable is compared the soil redeposition in the laboratory tests with that observed in the machine tests to determine the validity of the laboratory tests. The index numbers could not be used, but the reflectance values of the soiled and unsoiled fabrics were compared after completion of the cleaning cycle. (Fig. 2). The points indicated as "Lab Test (2)" represent a second experiment run with more severely soiled samples. The individual points are average values of many individual laboratory tests or machine runs. The individual test data exhibit more scatter and overlap. By averaging the data in this way the trend curves are better defined.

It should be emphasized that these are trend curves. It is not implied that a 1:1 inverse correlation exists between soil removal and soil redeposition so that one can be used to compute the other. They are both manifestations of a common property in detergent systems; as a general principle, high soil-removal goes hand-in-hand with low graying.

The curves in Fig. 2 show another thing, that the soiled cloth and the unsoiled cloth after cleaning have not come to the same reflectance, as perhaps they should. This is possible, and we occasionally observe it in machine tests. The machine-test data approach this more closely than the Launder-O-Meter data, but this may be because the laboratory test is a "batch" cleaning process whereas the machine process has continuous filtration.

It may also be noted in Fig. 2 that the laboratory test exhibits less graying for a given degree of soil removal than observed in the machine tests. The reason for this is the effect of soluble soil in the solvent in the machine tests. This results from poor solvent maintenance by the operator and should not be attributed to any deficiency in the detergent.

Soil-Deposition Tests. The object was to make a direct comparison of the degree of graying obtained in a soil-deposition test with that in a soil-redeposition test with the use of the same total quantity of soil in both cases. To ascertain the exact quantity of soil on the soiled swatches, use was made of the Kubelka-Munk relationship between reflectance and the quantity of material on the fabric. A calibration curve was obtained to relate the two for this soil by obtaining the oven-dry weight of a set of fabric samples, then soiling them, and reweighing them oven-dry. From the gain in weight of the samples and the reflectance readings, the calibration curve shown in Fig. 3 was

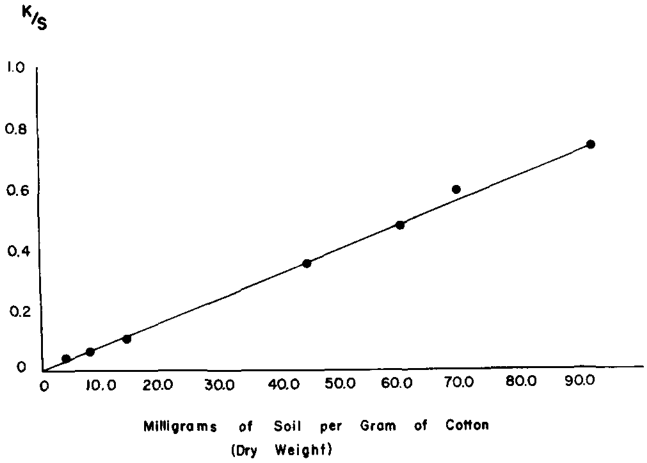

FIG. 3. Calibration curve for determining amount of rug soil on samples from reflectance data.

obtained. The cloth samples were cut on the bias (diagonally across the warp and filling) to avoid ravelling during soiling.

The reflectance of each sample in the redeposition tests was carefully determined, and the weight of the soil on each was computed. In each test, 10 g fabric loads were used, including five unsoiled swatches.

After the total quantity of soil was calculated in each test load, the deposition tests were set up to contain the same quantity of soil as in the corresponding redeposition tests. In these tests the same weight of unsoiled cloth was used (10 g) and the same solutions, etc., as in the redeposition tests. The weighed quantity of soil was added to each solution and stirred manually until uniformly mixed. The cloth samples were then added, and the canisters were put into the Launder-O-Meter along with the redeposition test canisters. The tests were thus run simultaneously. The only major experimental difference was the location of the soil at the start of the test. A minor difference which should be mentioned is that the soil was undoubtedly classified by application to the fabric by this soiling procedure; the coarser particles were excluded. However, this is part of the argument in favor of this procedure.

After completion of the tests (2 hr) the samples were rinsed, pinned to a line, and air-dried; then the reflectance readings were made. The data are summarized in Table II.

At the conclusion of the test all the solvent was recovered except the amount retained in the fabrics by capillarity. Each solution was filtered through weighed 0.2-micron millipore filter membranes to recover the suspended soil. After drying, the membranes were weighed again. Blank tests containing no soil were also run to get the amount of lint formed during the test. The soil weights were corrected for the average quantity of lint (6.1 mg from 10 g). The amount of solvent-soluble material in the soil was found to be 5%. To calculate the percentage of soil which was suspended, correction was made in the total soil for the solvent-soluble fraction. These data are also included in Table II.

The results show conclusively that graying is much more severe in redeposition tests despite the fact that the equilibrium soil-concentration is 35% greater in the soil-deposition test. Also, all of the soil was not removed from the soiled fabric. This fabric was more heavily soiled than the ones used in the other test. Nevertheless the reflectance values after cleaning fit the curve shown in Fig. 2. Table II shows that the fraction of the total soil suspended is practically constant for each type of test.

REFERENCES

1. Powe, W. C., Textile Res. J. *29*, 879 (1959).
2. Tuzson, John, and B. A. Short, Textile Res. J. *32*, 111–116 (1962).
3. Hensley, J. W., JAOCS *42*, 993–997 (1965).
4. Wedell, H., Melliand Textilber. *41*, No. 7, 1–4 (1960).

[Received July 7, 1967]