Detergency Evaluations to Assess Fabric Wash Products

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Fabric detergents consist of a large number of ingredients whose chemical structures and cleaning efficiency can vary greatly. The industrial advances, ecological and economic pressures in recent times are resulting in constant modifications of detergent formulas. In addition, the multitude of available raw materials, differing quality and chemical complexity makes life difficult for the formulator in his eternal search for the elusive answer on end-product performance.

Product performance test methods generally have a singular goal and that is to prove and conclusively establish that the product is of a superior grade. Quality here includes all subjectively and objectively measurable properties that play an important role in the application of the consumer product.

Detergency evaluation is one such test that is used by all fabric detergent formulators to establish product quality and cleaning efficiency.

Use of detergency evaluations in product assessment

Detergency evaluation involves mainly the following studies.

1. Soil removal by use of various surfactants, either individually or in combination.
2. Soil removal brought about by use of builders like phosphates, silicates, carbonates etc.
3. Stain removal by bleaching agents, enzymes etc.
4. Soil & stain removal by the combination of the above ingredients in a detergent base formulation or newly formulated detergents.
5. The brightening or whitening effects produced on a fabric due to the presence of optical brighteners or fluorescent whitening agents.

The above test methods can be also be modified for studying the performance of fabric detergent formulation in terms of wash conditions, fragrance performance and retention on the fabric after washing or drying etc. Evaluation of the washing effects due to washing conditions, washing temperature, mechanical factors involved during washing operations, the time taken to complete the washing process, water hardness etc., can all be assessed during these studies. Detergent evaluation studies can also be used to compare the performance of different detergents, various types of the wash processes or performance of washing machine types.

Washing process fundamentals

Washing capability of any detergent depends, on the following six W's of the washing process:

1. Water
2. Waves (mechanical energy)
3. Warmth (heat energy)
4. Wash substrate (textile type)
5. Washing agent (detergent formula)
6. Wash soils

In addition, general soil removal and cleaning during washing results from:

1. Physical washing effects due the presence of surface active agents in detergent formulations.
2. Chemical action effected by bleaching agents, bleach activators, enzymes, etc.
3. In addition to soil removal, its re-deposition is also very important to be considered during the washing process.

Types of soiled fabrics

Artificially soiled test fabrics or swatches have been used for long as an important tool for detergency evaluations. These tests effects are done with the help of one or several different types of soiled fabrics made from varying fabric materials. Different types of artificially or semi-artificially soiled fabrics can be used for conducting these tests. Some of the important types are listed below.

1. Cotton soiled with carbon black or vegetable oils.
2. Wool soiled with carbon black or vegetable oils.
3. Polyester / cotton soiled with oil
4. Cotton soiled with carbon black and mineral oil.
5. Cotton soiled with blood.
6. Cotton soiled with chocolate / cocoa.
7. Cotton soiled with red wine.
8. Cotton soiled with tea / coffee.
9. Cotton soiled with vegetable curry.
10. Cotton dyed with sulphur black.
11. Cotton soiled with blood / milk / carbon black.

Artificially soiled test fabrics tend to age and so have to be packed and stored in dark to keep it serviceable for long. Generally, multiple soiled test strips are used for testing the performance of washing. This is specially so when we have to test washing machine types. All comparative tests done should be carried out

using soiled swatches made at the same time. In case this is not followed, test results obtained will be incorrect. Thus, the quantity of test fabric prepared at one time should be sufficient to make it possible to conduct the entire test protocol of a particular project.

Use of artificially soiled test fabrics

Carbon black / oil soiling, on a substrate of cotton, wool or synthetics are the most common artificial soils used for detergency testing. The performance of surface active agents, builders, washing powder formulation, etc., is tested by use of carbon black / oil soiling as they are sensitive to mechanical and thermal actions involved in the washing process.

Proteinaceous soils like blood, milk, cocoa, chocolate etc., get denatured due to aging, heat or chemicals. This makes the soiling difficult to remove by simple washing. They are influenced by the sequence of heat applied during the washing cycle, the action of bleaches the detergent powder contains and the washing process used, as a whole. Proteinaceous soils are used as standards for evaluating proteslitic enzymes in detergents.

Fruit stains and organic coloured pigment soil are used for testing performance of bleaching agents. Unsoiled fabrics are used to test soil redeposition. Fluorescence due to fluorescence whitening agents or optical brighteners also requires testing in unsoiled fabrics. Many a times, multiple soiled strips are also used, if one is interested in testing all the above effects in a single test study wash cycle.

Formulating a soiling mixture

A typical standard soiling mixture has the following components.

1.	Refined coconut oil / vegetable oil	10 g
2.	Distilled coconut fatty acid	05 g
3.	Mineral oil / liquid paraffin	02 g
4.	Anhydrous lanolin	01 g
5.	Carbon soot	04 g
6.	Petroleum ether solvent	made up to 1 litre

Preparing the soiling mixture

All the above mentioned standard soiling mixture ingredients are taken in a container. It is then mixed into a homogeneous mass using a small portion of petroleum ether. Once the mixture is homogenous, the remaining portion of petroleum ether is added to make up the volume. The mixture should be stored in an airtight container to avoid change in composition of the soil due to solvent evaporation.

Soiling procedure

Shake the soiling mixture well and transfer it into the trough of a padding mangle, taking care that the guide rods are completely submerged in the soiling solution. Pass the soiling cloth through the solution three to five times. The dried soiled cloth should have a reflectance value of about 35 units. The soiled cloth is then cut into smaller pieces or swatches of 10 cms X 10 cms.

Test equipments

Special laboratory equipments like Terg-o-tometer or washing machines having washing program with well-defined temperature, mechanical and chemical actions are required to carry out tests to determine wash performance of surfactants and fabric detergents. It is important that mechanical agitation and fabric-to-fabric frictions are well controlled in laboratory conditions.

In any washing tests carried out, it is important that water hardness (24°FH / 240 ppm as $CaCO_3$, Ca: Mg ratio 2:1), period of washing (5 minutes), concentration of the wash liquor (0.75% product concentration), ratio of cloth to wash liquor (Cloth in grams to the wash liquor in ml is about 1: 50), the amount of bleaching agents, temperature of washing (30°C), the type of washing, and the load of the fabric to be used are noted down.

The type and size of the textile fabric and the soiling used can all influence performance results. In all tests, naturally soiled fabric swatches, non-soiled fresh fabric swatch, as a reference standard, should also be washed along with

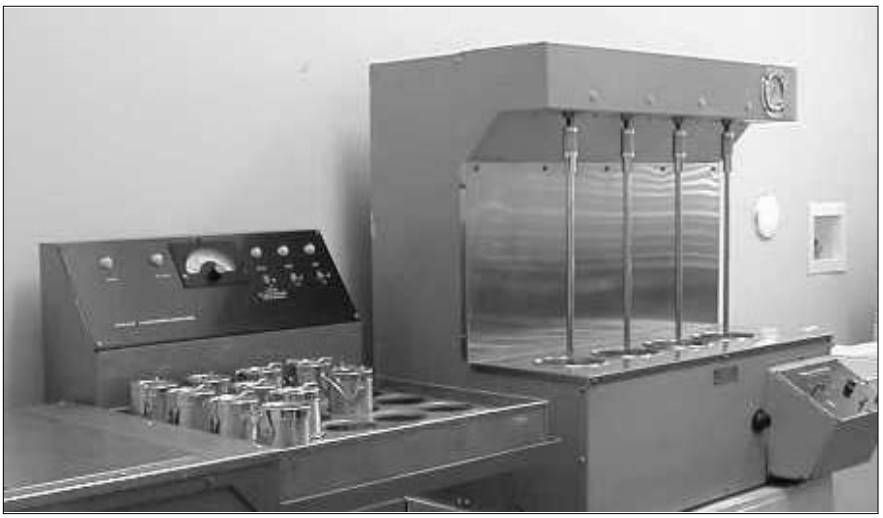

Figure 1: Terg-o-tometer for evaluating laundry products on a laboratory scale

the test or artificially soiled fabric swatches for comparative studies. The degree of whiteness of washed naturally soiled fabrics, non-soiled fresh fabric and artificially soiled fabric samples is measured using a reflectometer and detergency expressed as percentage soil removed.

Washing technique

Detergent powders

Several swatches of soiled fabrics are washed during a single wash. The washing cycle is repeated at least a couple of more times using a new set of soiled fabrics to check repeatability of washing performance. This is necessary as within each wash cycle and also between wash cycles, variations are generally observed in soil removal characteristics in standard soiled fabrics due to uneven mechanical thermal and chemical action during the washing process. When small pieces of soiled cloth are used in tests they should be fixed to the textiles washed. However, in case larger soiled fabrics are used for testing, they can be washed directly as such, without any fixation. After washing is completed, the fabric samples are dried and ironed at a fixed temperature.

Detergent bars

The above method by use of Terg-o-tometer or washing machines is more suitable for detergent powders and liquids. In case of detergent bars, a wash-down procedure under controlled laboratory conditions that simulates the various physical forces involved in the fabric cleaning process, when using a detergent bar or a laundry soap cake, according to Indian wash conditions, is designed and followed.

This is necessary so that a correct whiteness measurement is obtained for comparative studies. Sample detergent bar / laundry soap cake are rubbed down in a reproducibly controlled manner on the wetted test fabric. The operator then performs controlled kneading and squeezing operations and measures the reflectance or the whiteness obtained in the washed, dried and ironed fabrics to conclude the superiority of a detergent bar or laundry soap cake sample.

As in the case of detergent powders, multiple tests are done and the mean or average value observed is taken as the final test result. Without doubt, repetition of tests to confirm results for final average value depends on the skill and dexterity of the operator.

Reflectance measurements

Reflectance measurements on washed fabrics are carried out using a reflectometer. A filter that absorbs all UV light is placed between the light source of the reflectometer and the washed soiled fabrics so that fluorescence of the fluorescence whitening agent is eliminated in the measurements. The reflectance values are measured using a tristimulus blue filter or a filter having $\lambda = 460$ nm.

A minimum of four single reflectance measurements readings — two in front and two in the back side of the washed fabric sample — should be made and the resultant average measurements are considered for all calculations and for drawing inference on the experiment carried out. Soil removal and cleaning efficiency on soiled fabrics washed under stipulated conditions are assessed by reflectance measurements. As obvious, higher the reflectance measurement values better the soil removal or cleaning efficiency of the process.

Practical evaluation & consumer tests

Practical wash evaluation is normally done using commercial washing machines in naturally soiled fabrics. In contrast, laboratory testing is carried out in artificially soiled fabrics. Laboratory based wash tests are carried out to get results that are comparable to washing in realistic conditions. However, it is near impossible to replicate the complete spectrum of wash conditions that occurs in actual practice. Detergency evaluation measurements by visual analysis are without doubt better, as instrumental method of evaluation and interpretation is difficult and subject to questioning. This is particularly true from the consumer point of view, as they are more comfortable with actual in-use washing under realistic situations.

Product performance and consumer acceptability will be achieved only when consumers subject the formulation to their own individual set of wash conditions at their homes. In this respect, practical consumer tests will help draw conclusions to identify product weakness and shortcomings in a more meaningful manner.

Experience shows that consumer in-use tests are more valuable than a laboratory test results. Ultimately product judgement must be based on not only wash results, but also on all other relevant factors that affect product in-use characteristics.

In-use practical evaluations are time consuming and associated with high labour and material costs. However, the formulator cannot omit product assessments under fabricated in-use conditions, if the product has to meet consumer needs and fulfil expectations.

Under these circumstances, detergency evaluation and product assessment laboratory procedures are necessary to obtain useful information on product quality and its performance. Results obtained through these tests, although not identical to real life conditions, do provide valuable clue to formulators during product development and thereafter.