Statistical Analysis of Detergency Tests With a Natural Soil

JAMES R. TROWBRIDGE, Colgate-Palmolive Research Center, 909 River Road, Piscataway, N.J. 08854

Abstract

Factorial experiments are described for detergency tests using a natural soil. The tests are carried out over several soil-wash cycles using soil rubbed onto test swatches from the surface of the skin. The significance of the results is increased by removing the variability of soiling among different individuals from the wash treatment totals and by using a log transformation on the original data. Factors investigated include wash water temperature, water hardness, concentration of surface active agent and concentration of builders.

Introduction

A detergency test which uses a natural soil and which closely simulates laundry practice has been described previously (1). A circular area of soil is applied to a test fabric by rubbing it against the skin surface. Test swatches are conveniently resoiled after laundering which allows one to obtain a number of repetitive soil-wash cycles on the same cloth. This may be important when detergent residues such as calcium salts are left on the cloth after washing. It also allows for a gradual build-up of grayness which may be necessary before small differences in performance can be determined. Although cotton swatches were used exclusively in these experiments, the method can be applied to a variety of other fabrics. Because of the variability of soiling, it has been particularly useful to employ experimental design principles in the planning of the experiment and appropriate statistical procedures in the evaluation of the results.

General Procedure

The test swatches were 6 × 4 in. cotton swatches (Testfabrics, Inc., Style No. 400 MW). These were marked with a 1¾ in. diameter circle and an identification number in indelible ink. The marked circle was centered over a 1½ in. diameter rubber stopper faced with a sponge rubber pad. A circular area of soil was applied to the fabric by rubbing it over the face and neck. Reflectance values in the range of 65 to 80 were usually obtained on the soiled swatches. Reflectance values, R_d, were read using a Gardiner Model AC-3 Color Difference Meter. The soiled area was centered over a 1½ in. diameter opening and the swatch was backed with a standard white plate ($R_d = 85$). Before resoiling swatches were sterilized by exposing them to a dilute concentration of ethylene oxide gas in a commercial sterilizer (Model No. 8, Ben Venue Laboratories, Inc., Bedford, Ohio). In previous work we have not observed any change in the launderability of the test fabrics as a result of this sterilization treatment.

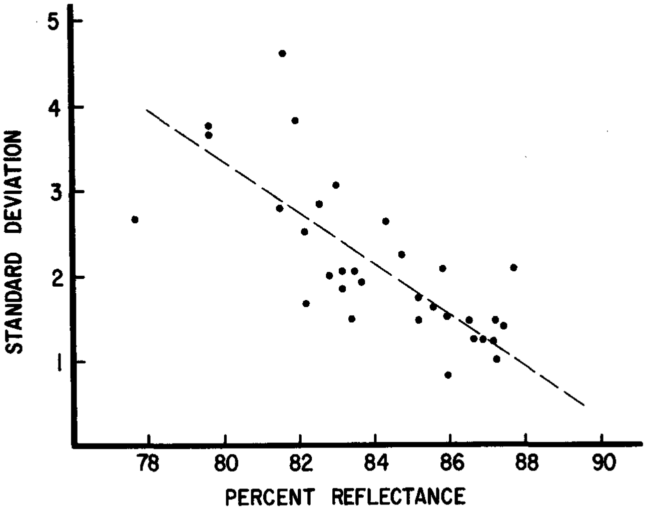

FIG. 1.

Complete 2^5 Factorial Experiment

In this experiment five factors were used at each of two levels as shown in Table I. The complete experiment contained 32 (2^5) different wash treatments with eight swatches assigned to each wash treatment. Swatches were handed out at random to laboratory personnel for soiling (usually about four swatches to each individual). Wash treatments were also run in a random order with the exception that all of the 120 F washes were done in one group followed by the 60 F washes. A total of three soil-wash cycles were obtained.

Log Transformation of Values

The standard deviation of the R_d values within a set of eight swatches was calculated for each of the 32 treatment means. Figure 1 shows a plot of standard deviation vs. mean R_d value. It is quite apparent that the standard deviation tends to increase with decreasing reflectance value in an approximately linear manner. In order to conform more closely with the assumption of a homogeneous error required

TABLE I
2^5 Factorial Experiment

Code	Factor	Low level	High level
A	Conc. of nonionic (*n*-dodecanol + 7 E.O.)	.005%	.01%
B	Conc. of potassium pyrophasphate	.02%	.04%
C	Conc. of sodium silicate	0	.01%
D	Water hardness, ppm	50	150
E	Wash temperature, F	60	120

TABLE II
Reflectance Values After Third Wash

Treatment	R_d	Treatment	R_d
1	81.0	e	86.7
a	83.5	ae	88.3
b	82.5	be	86.1
ab	82.2	abe	87.9
c	82.8	ce	85.3
ac	83.5	ace	87.0
bc	84.7	bce	87.2
abc	83.9	abce	87.7
d	80.1	de	80.1
ad	83.4	ade	85.9
bd	83.2	bde	85.3
abd	85.0	abde	85.8
cd	77.8	cde	82.6
acd	82.7	acde	86.6
bcd	83.0	bcde	86.0
abcd	83.4	abcde	87.3

TABLE III
Significance of Factor Effects

Effect	Transformed Value	t	Effect	Transformed Value	t
Grand mean =	1.8201	......	E	–.5486	15.8[a]
A	– .2821	8.10[a]	AE	–.1251	3.60[b]
B	– .1875	5.39[a]	BE	–.0177	.51
AB	.1363	3.91[a]	ABE	.0170	.49
C	– .0295	.85	CE	–.0254	.73
AC	.0380	1.09	ACE	.0002	.01
BC	– .0837	2.04	BCE	–.0298	.86
ABC	.0125	.36	ABCE	–.0261	....
D	.2289	6.58[a]	DE	.1542	4.43[a]
AD	– .0864	2.48[b]	ADE	.0382	1.10
BD	– .1235	3.55[b]	BDE	–.0024	.07
ABD	.0578	1.66	ABDE	.0460	....
CD	– .0085	.24	CDE	–.1543	4.43[a]
ACD	– .0360	1.04	ACDE	–.0285	....
BCD	.0740	2.13	BCDE	.0253	....
ABCD	– .0123	......	ABCDE	–.0535	....

[a] Significant at .01 P.
[b] Significant at .05 P.

for analysis of variance and also to increase the significance of the treatment effects, the raw data was transformed as follows:

$$T = \log_e (91 - R_d)$$

where T is the transformed value of the original R_d reading for each swatch. An electronic computer was used for this transformation and other computations.

Treatment Effects

The factor levels in Table II are indicated following the notation described by Cochran and Cox (2) where the presence of a small letter indicates that the corresponding factor from Table I was used at the high level. The absence of a letter indicates that the factor was used at a low level and the symbol 1 indicates that all five factors were at the low level. The R_d values shown in Table II were obtained by first calculating the mean value of the transformed data and reconverting this mean by the inverse equation

$$R_d = 91 - \exp (T)$$

Table III shows the various factor effects based on the transformed data. The standard error of an effect mean ($s_m = \pm.0348$) was estimated by pooling the sum of squares of the five four-factor and one five-factor interactions with a total of 6 degrees of freedom. A value for the experimental error ($s_m = \pm.0428$) was also estimated from the within treatment swatch variance. However, the initial plan was to use the higher order factor interactions as an ±.0428) was also estimated from the within treatment do not represent independent experimental units, i.e., they are all washed at the same time.

Four of the five single factor effects were highly significant and are shown in Table IV where the mean transformed R_d values are shown.

The effect of a two-factor interaction (B × D) is illustrated by the breakdown in Table V in which the mean reflectance is shown at each of the four possible two-factor combinations.

TABLE IV
Main Effects

Code	Factor	Low level	Rd	High level	Rd
A	Conc. nonionic surfactant	.005%	83.9	.01%	85.6
B	Conc. potassium pyrophosphate	.02%	84.2	.04%	85.4
C	Conc. sodium silicate	0	84.9	.01%	84.7
D	Water hardness, ppm	50	85.5	150	84.1
E	Wash temperature, F	60	82.9	120	86.3

TABLE V
Hardness × Builder Interaction

Water hardness	Conc. potassium pyrophosphate	
	.02%	.04%
50 ppm	84.9	85.4
150 ppm	82.4	85.0

At the lower level of builder there is a significant decrease in detergency with increase in hardness as there is insufficient builder concentration to sequester the higher level of hardness. At the higher level of builder increase in hardness has little effect. Similar two-way tables can be constructed to illustrate other significant interactions (2).

With reference to Table III it is unexpected that the only one of the 10 possible three-factor interactions which shows significance is the sodium silicate concentration × hardness × temperature interaction (CDE), since neither the main effect of sodium silicate concentration nor any of its two-factor interactions show significance. In view of the many interaction effects which are being considered in this experiment, there is a reasonable expectation that at least one estimate of interaction will show significance even if in fact no interaction is present. An additional experiment involving these three factors would be desirable in order to confirm this indication of a three-factor interaction.

Uniform Soiling Within Panelists

In an experiment with a limited number of treatments, generally not more than eight, a considerable increase in precision has been obtained by assigning to members of a soiling panel swatches from each of the wash treatments. Panelists are asked to soil all swatches as uniformly as possible by soiling the swatches several times in a random order. If this is

TABLE VI
Comparison of Seven Detergents
Reflectance After Sixth Wash

Panel members	Detergent formulations						
	A	B	C	D	E	F	G
I	84.2	83.7	84.8	85.1	83.1	82.6	84.7
II	85.9	85.6	86.0	86.2	85.5	84.9	85.7
III	82.3	79.6	81.9	84.3	76.7	82.3	83.3
IV	82.7	82.0	82.9	83.1	79.7	81.3	82.4
V	84.5	84.2	84.4	84.9	84.0	83.9	83.6
VI	83.2	83.2	84.6	84.6	82.9	83.5	84.8
VII	82.0	80.0	82.5	83.9	79.8	78.7	81.3
VIII	84.9	83.0	85.1	85.6	84.0	85.0	84.6
IX	85.9	85.4	86.0	86.3	86.0	85.6	86.0
X	85.2	84.7	84.5	85.3	83.9	83.8	83.9
Original mean	84.1	83.1	84.3	84.9	82.6	83.2	84.0
Transform mean	84.3	83.5	84.6	85.1	83.2	83.5	84.3

TABLE VII
Analysis of Variance on Original and Transformed Data

Source	Degrees of freedom	Mean square		F ratio	
		Original	Transformed	Original	Transformed
Detergents	6	6.645	.3086	7.86	13.8
Panelists	9	19.164	1.0717	22.7	47.8
Residual	54	.845	.0224		

done carefully a set of swatches can be obtained which have a standard deviation of less than 1 R_d unit. Table VI gives R_d values after 6 soil-wash cycles in which 7 swatches from each of 10 panel members were assigned to 7 different wash products. All washes were in 100 ppm hardness water at 120 F.

Mean values as obtained directly and those obtained for the transformed values based on the transformation, $T = \log_e (88 - R_d)$, are shown in Table VI for the seven detergent formulations. The advantage of using the transformed values is shown by the increase in significance which is obtained in the analysis of variance where the F value for the treatment mean square is increased from 7.86 to 13.8.

The Newman-Keuls procedure as described by Winer (3) was used to determine significant differences among the seven detergents. In Tables VIII and IX the retransformed mean R_d values for the seven detergents are listed in order of magnitude. Means which are not underlined by the same line are found to be significantly different at the 95% confidence level.

TABLE VIII
Newman-Keuls Test of Significance

Shortest significant ranges						
Range	2	3	4	5	6	7
Sign. diff.[a]	.134	.161	.178	.189	.198	.205

[a] Transformed means at $\alpha = .05$.

TABLE IX
Newman-Keuls Test of Significance
Ranked Means

R_d Values After sixth wash						
E	B	F	G	A	C	D
83.2	83.5	83.5	84.3	84.3	84.6	85.1

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