Computerization in Fabric Detergency Testing¹

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Abstract

An automated method is described which greatly speeds the calculation of fabric detergency test results. Laboratory determination of fabric detergency commonly involves replicated, bench scale washing of small pieces of cotton artificially soiled with various oil-carbon black mixtures. Reflectance measurements before and after washing give a measure of the amount of soil removed. Often, in a program involving several variables, thousands of reflectance mea-surements may be involved. By converting the electrical signal from the reflectometer to digital form, and feeding this value to a card punch, the reflectance values are systematically recorded on punched cards. Using an appropriate computer program, the reflectance changes for each test are calculated and tabulated, the saving in operator time is large and statistical examination of the data can be incorporated with the program. An example of the type of data output is given.

Introduction

YOMPUTERS ARE FINDING increasing use in all areas \checkmark of scientific endeavor. The detergency field is perhaps somewhat slow to adopt such techniques, but several instances are known, including the work of Gordon et al. involving the analysis of doubly tagged sebum soils (1) or of tagged surfactants on fabrics (2). Huggins (3) has used computer techniques in the linear regression analysis of data obtained during the evaluation of light duty liquids in dishwashing tests. At a later date, we hope to report on a general program used in our laboratories for the design and analysis of experiments involving up to six variables, useful in studying effects of composition on physical properties or performance properties. We also use a program for interpreting data from panel evaluations of softness, odor or other subjective property. The present paper describes methods for automatic

¹Presented at the AOCS-AACC Joint Meeting, Washington, D.C., March 1968. processing of reflectance measurements and subsequent calculations in fabric detergency testing.

Laboratories concerned with measurement of fabric detergency are generally faced with the determination of the amount of soil present on numerous pieces of fabric before and after washing. Although there is a trend towards the use of radioactively tagged soils (4,5), most laboratories employ oily soils containing a colored component such as iron oxide, or clay (5,6) or natural airborne particulate matter (7). Test cloths employing soils containing carbon black are the most common, and may be purchased from several suppliers. With these colored soils, reflectance measurements are used to determine the washing efficiency. Irrespective of the method used to calculate detergency from reflectance change or the details of the washing method itself, a common problem encountered also in soil retention or optical brightener studies is the frequent necessity to determine the reflectance of large numbers of fabric swatches and to perform many simple but tedious arithmetic calculations. A typical detergency program might involve the comparison of six surfactants, formulated at three concentration levels, and washed at a single temperature but at three levels of water hardness. With four types of soiled cloth, and with all washing tests performed in quadruplicate, a total of 864 fabric swatches is involved. If three of the types of test fabric are soiled on both sides (such as the commercially available U.S. Testing Co., Foster D. Snell, Inc., ACH Fiber Service, Inc., or Swiss EMPA cloths) and thus require four reflectance measurements per swatch (readings taken on each side of fabric with swatch oriented parallel to and perpendicular to the warp direction), and one is soiled on only one side (such as Testfabrics, Inc.) and requires two reflec-tance measurements, a total of 3024 reflectance measurements is needed on the washed swatches, plus the measurements necessary for the soiled swatches. The calculation of 864 average washed reflectance values (W) must be performed, involving determination of the mean of the readings taken on each swatch. From these values the corresponding



FIG. 1. Schematic diagram, automatic card punching of reflectance measurements.



FIG. 2. Reflectometer, digital voltmeter and card punch assembly.

soiled (S) values must then be subtracted. At the option of the experimenter, these reflectance changes need to be compared to a standard or calculations continued to determine detergent efficiency or soil removal such as by the Bacon and Smith (8) method.

Automatic Data Processing

In our laboratory, we have developed a procedure

to automate the processing of the numerous reflectance measurements and to calculate and tabulate the test results by means of an appropriate computer program.

A number of reflectometers are commercially available, but we have found the Gardner Precision Reflectometer, Model AUX-2, comprising a three-filter exposure head and an automatic photometric unit,



FIG. 3. Mounting of helipot to photometric unit.



Automatically Punched FIG. 4. Typical data card.

to be particularly suitable. The self-balancing feature permits rapid readings even without the card punching modification discussed below.

By means of a 10 turn, 1000 ohm helipot geared 1/1 to the drive of the photometric unit, a proportioned signal is generated for transmission to a digital voltmeter, Non-linear Systems, Inc. of Del Mar, California, Model 5005. This instrument uses a relay-operated, digital feedback voltage divider, energized by a 10 v Zener reference voltage, and creates a feedback voltage equal to the input. Output is in digital form. Modifications to the basic instrument were made by the manufacturer so as to store digits for sequential transmission to an IBM 526 Printing Summary Punch. Contact closure for the card punch, a foot switch for print control, and appropriate cable and connecting shoe are necessary.

The card punch receives the digital output sequentially, and punches as instructed by a control board and drum card. By pre-arranging the test swatches in groups of four, comprising one of each type of cloth, all the reflectance measurements, for example from a single, mixed-load wash, are punched onto a card. Code numbers are punched manually to identify the sample, the concentration, the hardness and replicate, and the swatch number if desired. A new card is then automatically positioned for the next reflectance measurements.

A block diagram is given in Figure 1 and a photograph of the complete unit in Figure 2. Figure 3 shows the mounting of the helipot to the photometric unit. A typical data card is illustrated in Figure 4.

Computer Program

Measurement of the reflectances of all swatches from a test series at a particular temperature produces a deck of data cards which can be used, together with an appropriate program deck, to calculate and tabulate the test results. The program can of course be written in many ways to suit the needs of the particular company or experiment and the computer facilities available. The following program is described briefly to illustrate the type of calculation and format possible. It is not our intent at this time to propose a detergency test method, or to compare various formulations and their response to test conditions.

The program, used with an IBM 7040 computer, performs the following operations.

1) Calculates the mean value for the four (or two) reflectance values obtained on each washed swatch.

2) Calculates the similar value for each cor-

WASHED ~ SOILED VALUES FOR EACH REPLICATE REPLICATES DELETED WHEN DEVIATION FROM MEAN EXCEEDS 1.53*JOINT STANDARD DEVIATION (CHEM. ENG.V74,2/13/67)

NONIONIC A									
		UST TES	T CLOTH	TFI TES	т сготн	FDS TES	T CLOTH	EMPA TEST	Г СЕОТН
				~~~~~~~					
	CONC	. H1	H2	H1	H2	H1	H2	H1	Ha
		#			~~~~				
REPLICATE	1 C1	4.4	3.0	7,6	5.9	3.6	3.8	13.6	6.3
REPLICATE	2 C1	3,6	3.0	8.1	5.2	4.5	3.7	17.9	8.4
REPLICATE	3 C1	4,3	2.5	9.3	6+5	3.5	4.4	17.8	7.5
REPLICATE	4 C1	3.7	3.2	8.3	7.5	3.7	3.8	17.6	8.1
				********					
Mi	EANS	4.0	2.9	8.3	6.3	3.8	3.9	10.7	8.0
S	IGMAS	•4	.3	•7	•9	•4	.3	2.1	.4
S	• E• M•	.2	.2	• 4	•5	,2	.2	1.2	.3
S	IG/MEAN	10.4%	11.0%	8.8%	15.0%	11.0%	8.3%	12.6%	5.6%
S	AMPLES	4.0	4.0	4.0	4.0	4.0	4.0	4.0	3.0

FIG. 5. Detailed output of reflectance changes after rejection of outlying data points.

		UST TEST CLOTH		Г С∟ОТН	TEST CLOTH		FDS TEST CLOTH		EMPA TEST CLOTH	
		CONC.	H1	H2	H1	H2	H1	H2	H1	H2
NUNIONIC A		C1	4.0	2.9	8.3	6.3	3.8	3.9	16.7	8.0
NUNIONIC A +	•c•	C1	5.3	4.1	8.8	6+b	4.2	4.9	18.7	8.8
NUNIONIC A +	•D	C1	4.6	3.5	7.9	7.1	3.9	4.5	16.7	8.4
ANIONIC F		C1	4.9	4.3	9.6	5.7	8.3	3.9	17.7	8.8
ANIUNIC F +	'C'	· C1	3.6	4.7	8.3	6.3	9.1	5.7	17.3	8.6
ANIONIC F +	•0	C1	3.2	4.8	8.6	5.2	11.5	5.6	16.6	7.5
JUINT STD.	DEV	• C1	.65	.47	.82	•74	.52	• 34	1.83	.73

#### MEAN VALUES FOR WASHED - SUILED REFLECTANCES

FIG. 6. Summary table showing corrected mean reflectance changes.

responding soiled swatch, and determines the difference between the two mean values.

3) Tabulates this difference in reflectance for each of the four replicates, according to surfactant, concentration, test cloth and water hardness, and calculates and tabulates the mean value and the standard deviation of this mean value, both in absolute form and as a percentage of the mean.

4) Calculates and prints the joint standard deviation for all samples tested at each hardness and concentration level on each test cloth.

5) Tests the data for each set of four replicates and rejects a single value exceeding a preselected limit of error, currently 1.53 times the pooled standard deviation (9), and recalculates and reprints the data in items 3 and 4 based on the three remaining values. Figure 5 gives a partial example of this output. The first replicate value, 6.3, found for Nonionic A with EMPA cloth at hardness H2 is an example of a rejected value.

6) Summarizes in a shorter table (see Fig. 6), the mean reflectance changes (W-S), as corrected above to eliminate wild values, together with the new joint standard deviation.

7) For each test cloth, assigns a value of 100 as the rating for a preselected reference sample at a designated washing condition, and proportions all other experimental values up or down, according to the ratio of their reflectance change to that of the reference. Thus, the reflectance change is converted from an absolute value to a percentage of that observed with the reference. Figure 7 is a tabulation of these ratings. 8) Calculates and tabulates these ratings averaged over all test cloths, and over all conditions for a given surfactant, and recalculates to a new value of 100 for the reference detergent (Fig. 8.)

9) Examines the data from 6 for consistency of effect of each experimental variable, using a nonparametric method, Wilcoxen's signed rank test (10), and indicates each detergent which is statistically different from the standard. This final tabulation is shown in Figure 8.

It will be observed that the last sample is the only one not stated to be significantly different from the reference, although it has the highest overall rating. This rating is largely the result of the high value found for FDS cloth at H1, but, because in four cases involving the other cloths the sample has a lower rating than the standard, the sample does not show a sufficiently consistent effect, and thus does not pass the significance test.

### Advantages of Computer Calculation

Although the cost of the reflectometer and digital voltmeter is considerable, of the order of \$5,000, and the availability of the computer must be justified on other grounds, the system has numerous advantages. In the 864 swatch example mentioned above, we estimate a time saving of at least 30-40 man hours in reading reflectances and calculating and tabulating the results. Our computer calculation required about 3.6 min, at a charge of about \$10. The results of a large program can be available within 3 or 4 hr after the reflectances are measured, and with much

			UST TEST CLOTH		TEST CLOTH		FDS TEST CLOTH		EMPA TEST CLOTH	
	с -	0NC.	H1	H2	H1	H2	H1	H2	H1	H2
NUNIONIC A		C1	137.1	100.0	133.6	100.0	98.7	100.0	208.9	100.0
NUNIONIC A +	'c'	C1	182.1	139.2	141.4	105.6	107.2	124.8	233.2	109.3
NUNIONIC A +	•0•	Cl	156,1	118.0	127.2	114.0	100.3	115.9	208.7	105.2
ANIONIC F		C 1	167.6	146.1	154.4	91.6	212.8	99.5	221.2	109.1
ANIONIC F +	•c•	Cl	121.5	159.9	132.8	101.3	233.1	146.4	216.3	106.7
ANIONIC F +	•D•	C 1	108.7	163.5	138.2	83.6	295.1	143.6	207.2	93.5

RATINGS FOR EACH TEST FABRIC

FIG. 7. Rating relative to a preselected standard.

OVERALL PRODUCT RA	ATING OVER	CONCENTRATIONS,	HARDNESS,	AND	CLOTHS
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NONIONIC A	122•3	100.0a)	REFERENCE PRODUCT
NONIONIC A + *C*	142•9	116.8	SIGNIFICANT DIFFERENCE AT THE 95 PERCENT LEVEL.
NGNIONIC A + 'D'	130.7	106.8	SIGNIFICANT DIFFERENCE AT THE 95 PERCENT LEVEL.
ANIONIC F	150+3	122.9	SIGNIFICANT DIFFERENCE AT THE 95 PERCENT LEVEL.
ANIONIC F + *C*	152+3	124.5	SIGNIFICANT DIFFERENCE AT THE 95 PERCENT LEVEL.
ANIONIC F + "D"	154•2	126.1	

SIGNIFICANCE DETERMINED BY WILCOXSON'S SIGNED RALK TEST (ENGR. STAT. BY BOWKER + LIEBERMAN PP 182-185)

a) Ratings relative to new value of 100 for reference product.

FIG. 8. Overall product ratings and statement of significance.

less chance for errors than when hand calculated and tabulated. Any suspect test values can be detected and redetermined while samples are still readily available. The ease of calculation makes possible the inclusion of a few additional tests into a program rather than stripping it to the bare minimum because of the tedious methods normally involved. If desired, output from the computer can be card punched, and the cards subsequently rearranged in any desired manner, such as in order of increasing detergency, or merged with previous results.

When the data are on punched cards, and subject to the limitations of programmer and computer, any desired type of sophisticated calculation can be made. For example, regression equations can be calculated, or the results can be automatically plotted, if a data plotter is available with the computer.

We believe that there are many benefits to be realized from the adoption of automated data handling

and calculating techniques, and highly recommend such procedures to anyone involved in large detergency test programs.

#### ACKNOWLEDGMENT

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