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Detergent Effects of Ultrasonics

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Abstract

The effect of ultrasonic treatment using 300 and 650 kc fields, respectively, on the detergent power of five products has been investigated. Factorial experiments were devised varying the different parameters such as textile fibers or fabric, intensity of the field, nature and concn of the detergent and length of treatment. The ultrasonic treatment proved highly efficient compared to the laboratory washing machine.

Introduction

COME EFFECTS of ultrasonic energy on detergency \mathbf{O} have been reported in the past (1-5). The object of this investigation was not so much to establish whether such treatment was commercially feasible but to shed some light on the still elusive details of detergent action.

Apparatus and Technique. In order to study dirt removal from fibers the chopped fiber technique of Powney & Feuell (6) was used, the fibers being soiled with the mixture described by Wagg (7).

For ordinary washing experiments the laboratory washing machine (6), henceforth to be called "posser" was employed. In case of subsequent ultrasonic treatment, wool and cotton, but not viscose fibers, had to be broken up in the posser (pre-possing) by treating 0.5-2.5 g fibers with 400 ml water for 10 min at room temp in the posser. This pre-possing, as will be shown, has no washing effect. For ultrasonic treatment a gentle stirring in the detergent solution, while the field is on, is necessary. As will be shown, this has only a negligible washing effect. After treatment the fibers were rinsed in water. The reflectances of the pads of washed fibers were measured by means of an EEL P.R.S. reflectometer, using a porcelain tile as standard. The reflectances are then expressed as percentage of this standard, which itself has 81% of the reflectance of MgO. It will be shown that the temp dependence of the washing effect is negligible so long as the Krafft point is exceeded. This last condition affects high titer (H.T.) soap only. For ultrasonic treatment, the initial temp was that of the room (for H.T. soap it was 42C), rising as the treatment proceeded.

The ultrasonic energy was generated by either a 300 or 650-kc quartz crystal of 5 cm diam, radiating vertically into a vessel containing the aqueous deter-

TABLE 1	
Reflectances	
Initial Values	

Fibers	Soiled	Unsoiled
Wool	32	85
Viscose	32	102
Cotton	36	105

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gent solution. The total ultrasonic power has been measured calorimetrically (9,10) by radiation pressure (11-13), by the height of the fountain (24), with an ultrasonic proble, by the hot wire method (14-18) and by the heat adsorption method (8). The most reliable and most reproducible method, albeit one giving an upper value owing to some dielectric heating, was the calorimetric method.

Experimental

Shown in Tables I and II are data for the reflectance values of the fibers, and the possing or mechanical washing of them.

Data for the necessary gentle stirring of the fibers shows in Table III, both Tables II and III showing the effect of no detergent and of mechanical action on the fibers.

Detergent solution used:

- 1) 0.2% sodium oleate solution by itself.
- 2) 0.1% H.T. soap solution containing 0.15%sodium metasilicate.
- 3) 0.1% Lissapol N solution containing 0.15% sodium metasilicate and 0.0015% sodium carboxymethyl cellulose (CMC).
- 4) 0.1% Santomerse solution containing the same admixtures as 3).
- 5) 0.1% Teepol solution containing the same admixtures as 3) and 4).

Percentages are calculated on the commercial product. H.T. soap contains 90% active compound. For the rest an analysis is given by Wagg (8).

The effect of temp upon soil removal from two fibers washed in the posser shows in Table IV: No temp effect was found.

Cotton Fibers. A statistical evaluation of detergency using cotton fibers was performed. Raw data show in Table V, and the analysis in Table VI.

A three factor experiment with two replications was done. The three factors were: 1) Detergent used at five levels, D1,D2,D3,D4,D5; 2) Length of treatment at two levels (five min and 20 min, respectively, T5, (T20); and 3) Kind of treatment at two levels (posser and 30 kc ultrasonics at 30 w power, W_p, W_u). R₁ and R_2 are the replications.

The standard deviation of means of two is 1.36, and the difference of means is 1.92. For significance at the 0.05 level, the means of two reflectances taken at random must differ at least by 4.

TABLE 11												
Possing	of	Fibers	in	400	\mathbf{ml}	Water	for	10	Min	at	$20\mathrm{C}$	

Fibers	R (Before)	R (After)
.25 g Wool	32	33
5 g Viscose	32	33
5 g Cotton	37	37

No washing effect.

1 6

TABLE III Reflectance Values After Gentle Stirring in the Ultrasonic Vessel at 60C for 20 Min (No Ultrasonic Engery)

Detergent:	1		2	2		3		4 ,		5	
	Before	After									
Fiber			·								
Cotton	36	37	36	40	36	39	36	39	36	39	
Viscose	32	34	32	38	32	34	32	34	32	34	
Wool	32	34	32	38	32	34	32	34	32	34	

The data show negligible washing effect.

TABLE IV Temp Effect on Washing. Liquor Ration (L.R.) Always 1:200. Time of Treatment 20 Min

Detergent:	[]	1	2	2	:	3	1 4	1	1	5
Temperature :	20°	60°	20°	6 0°	20°	60°	20°	60°	20°	60°
Fiber Viscose Cotton	61 87	$\frac{62}{86}$	62 88	$\frac{62}{88}$		60 90	56 83	56 83	$\frac{51}{78}$	$\frac{52}{78}$

TABLE V Cotton Fiber Experiment

Detergent	Ultrasonic				Detergent Ultrasonic						sser	
solution	5 N	Ain	20 Min		5 Min		20 Min					
1 2 3 4 5	72 84 78.5 75 52.5	$72 \\ 85 \\ 85 \\ 74.5 \\ 58$	85.5 87 95.5 86.5 70.5	$83 \\ 88 \\ 94 \\ 86.5 \\ 78$	70.5 77 77.5 73 65.5	70.5 76 76 72 65.5	86 87 90 83.5 78.5	$86 \\ 88 \\ 91.5 \\ 83.5 \\ 77.5$				

TABLE VI Statistical Analysis Cotton Fiber Experiment

Source of variance	Sums of squares	Degrees of freedom	Mean squares	F
w	6.40	1	6.40	
т	1512.90	1	1512.90	408.89
D	1521.70	4	380.41	102.81
$W \ge T$	2.50	1	2.50	
$T \ge D$	89.52	4	22.38	6.08
W x D	177.90	4	44.48	12.02
WXTXD	56.68	4	14.17	3.83
Residual	74.00	20	3,70	
Total	3441.60	39		

TABLE VII Wool Fiber Experiment

Detergent		Ultra	sonic		Posser				
solution	5 N	lin	20 Min		5 Min		20 Min		
1 2 3 4 5	68.5 73.5 74 40 37	74.5 76 70 38.5 36.5	$79.5 \\ 83 \\ 76 \\ 38.5 \\ 40.5$	$80.5 \\ 82.5 \\ 76 \\ 41.5 \\ 36.5$	79.58062.558.544.5	78.5 80 62.5 57.5 44.5	$83 \\ 87 \\ 68.5 \\ 62 \\ 51.5$	$85 \\ 87 \\ 67.5 \\ 61.5 \\ 49.5$	

TABLE VIII Viscose Fiber Experiment

Detergent solution	Ultra	sonics	Posser		
1 2	90 88.5 90 86.5 79.0	$90 \\ 89.5 \\ 90.5 \\ 86.5 \\ 72.0$	$\begin{array}{r} 61.5 \\ 60.5 \\ 60.5 \\ 53.5 \\ 51.5 \end{array}$	$62.5 \\ 62.5 \\ 59.5 \\ 58.5 \\ 51.5 \\ \end{array}$	

TABLE IX Effect of Ultrasonic Variation

Detergent solution	30) kc	650 kc		
1	92.5	$94.0 \\91.5 \\91.5 \\92.0 \\78.0$	90.0	90.0	
2	91.5		88.5	89.5	
3	91.5		90.0	90.5	
4	89.0		86.5	86.5	
5	75.0		79.0	72.0	

Of the main effects W (posser or ultrasonics) is not significant and posser and ultrasonics give the same performance. T (time) and D (detergent) are both highly significant.

Two first order interactions are significant, i.e., the length of treatment does not have equal effect on all detergents, nor is their order of efficiency quite the same for the posser and ultrasonic treatment.

Wool Fibers. The same factorial experiment was performed with wool fibers and instead of metasilicate, 0.04% sodium sesquicarbonate was used.

Standard deviation of means of two is 1.108, that of the difference of means 1.57. The means of reflectances taken at random must differ by at least 3.25 for significance at the 0.05 level. All the main effects and the W (washer) x D (detergent) interaction are highly significant. Ultrasonic washings in this case are inferior to posser cleaning.

Viscose Fibers. In this factorial experiment the ultrasonic treatment was at 650 kc and 60 w. The oleate contained 0.15% metasilicate. There were only two factors with two replications. W at two levels (posser and ultrasonics), D at five levels, the length of treatment being 20 min throughout.

Standard deviation of means of two is 1.4, that of the difference of two means is 2.01. For significance at the 0.05 level the difference of two means taken at random must be at least 4.5. Both main effects of washer and detergent are highly significant. Ultrasonics is much superior to the posser and the order of detergent effectiveness is the same in both (W x D interaction not significant).

Although the detergent effect is highly significant, an inspection of the data indicated that this was because Teepol was worse than the other four detergents, among which there seems to be no significant difference. Leaving out the Teepol experiments leads then to the following information: The detergent (D) effect is still very significant, partly because the residual is smaller because much of the variance has been caused by the Teepol results.

This also can be shown when two ultrasonic treatments are compared: One by 650 kc 60 w the other by 300 kc 65 w with two replicates.

The statistical analysis, excluding the Teepol results, shows that the W x D interaction is not significant. Both main effects are very significant. 300 kc at a somewhat higher power is better than 650 kc at a lower power. The detergent effect remains very significant even when the Teepol result is left out, though the bulk of the variance was really contributed by the Teepol values. These examples show the advantage of statistical analysis as against qualitative inspection of the results.

It is concluded that ultrasonic cleaning has an effect on detergency comparable with, or superior to, the very energetic mechanical posser treatment.

ACKNOWLEDGMENTS

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Determination of Foreign Materials of Plant Origin in Cotton Linters¹

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Abstract

The present article describes a test procedure which can be used for determination of foreign materials of plant origin in raw cotton linters. Laboratories that perform the cellulose yield test require very little additional equipment to carry out this test. A portion of the dry cellulose sample, remaining after the completion of the AOCS Official Cellulose Yield Method Bb 3-47 is bleached, formed into a hand sheet and the total projected area of the visible dirts is determined on both sides of the hand sheet. The determination of the dirts is patterned according to principles used in Technical Association of Pulp and Paper Industries Standard Procedures T213 M-43 and T437 M-43.

There are three main groups of foreign materials encountered : stalks, cockle burs and cotton seed hulls. The numbers and distribution of these particles vary with the general geographic locality and individual shipments of cotton linters. The test procedure described considers only those dirt particles which survive the major purification steps in the manufacturing of pulp from linters, and are undesirable from the quality point of view of the finished product. Prior to the development of the test only visual grading estimations have been used for this purpose.

Introduction

FROM THE PULP manufacturer's point of view, the quality of raw action limit quality of raw cotton linters always has been an important parameter, but a difficult one to assess. The cellulose yield test is the only quantitative analytical test procedure available for the determination of lint quality. The other attributes of lint quality, and the amount of foreign material in particular, are estimated qualitatively by visual grading. This subjective procedure leads to wide variations in the estimates of foreign material assigned to a particular lint by different graders. This measure of lint quality has been reasonably adequate in the past. However, the quality requirements for cotton linter pulp have increased steadily and have indicated the need for a quantitative procedure for quality estimation. Coincident with increased quality demands in linter pulps, the field trash level of cotton linters has increased in the past few years. This increase has occurred because of the widespread use of mechanical harvesting methods.

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This development is an additional reason for use of objective procedures for specifying and measuring quality levels.

This article describes a test procedure used by The Buckeye Cellulose Corp. in grading linter shipments with regard to foreign material. The emphasis is placed on dirts which are likely to survive the industrial linters purification processes and are incorporated as undesirable impurities in cellulose derivatives such as plastics or films.

Test Procedure

Laboratories that perform the cellulose yield test will require very little additional equipment to install this test. A portion of the cooked sample remaining after the completion of the standard AOCS cellulose yield test is bleached, a hand sheet formed, and the total projected area of the visible dirts is determined on both sides of the hand sheet. Such a procedure closely resembles the major purification steps of the pulp producer.

Apparatus and Reagents. If the test is carried out independently from the cellulose yield test, all the apparatus and reagents described in the standard method are necessary. Various additional pieces of equipment and reagents are also required. If the test under discussion is carried out on linters samples remaining after the cellulose yield test, these additional items are needed :

1. Mason type glass jars of two quart capacity.

A Williams handsheet mold $(10'' \times 12'')$.

3. A wringer with two rubber press rolls for dewatering the handsheets.

4. A Dirt Estimation Chart. This chart can be obtained from the Secretary of the Technical Assoc. of the Pulp and Paper Industry, 360 Lexington Ave., New York 17, N. Y.

5. A fluorescent table lamp.

6. Bleach Solution. This solution is made from sodium hypochlorite acidified with H_2SO_4 . The final solution contains 0.52 ± 0.02 gpl available chlorine and 0.32 gpl acid (equivalent to alkalinity of 0.26 gpl as sodium hydroxide)

7. Neutralizing Solution. Dissolve 250 g sodium thiosulfate $(Na_2S_2O_3 \cdot 5H_2O - "hypo")$ in 500 ml water. Add 20 g sodium hydroxide and dilute to one liter. The solution is approx 1N in thiosulfate and 0.5N in sodium hydroxide.

Procedure. Transfer a 20-g portion of the air dry cellulose yield sample of cotton linters to a two-quart

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