REFERENCES

1. Schilling, H. K., I. Rudnik, C. H. Allen, P. B. Mack and J. C. Sherrill, J. Accoust. Soc. Amer. 21, 39 (1949).
2. The General Electric Co., Ltd., 1949, B.P. 628415.
3. Wiczer, 1951, B.P. 648609.
4. H. J. Rand Washing Machine Corp., 1951, B.P. 658798.
5. Lever Bros. and Unilever Ltd., 1952, B.P. 664502.
6. Powney, J., and A. J. Feuell, Research 2, 331 (1949).
7. Wagg, R. E., J. Text. Inst. 43, T.515 (1952).
8. Pinoir, R., J. Chem. Phys. 44, 254 (1951).
9. Richards, W. T., Proc. Nat. Acad. Sci., Wash. 15, 310 (1929).
10. Richards, W. T., Ibid. 17, 611 (1931).
11. Sorensen, Ch., Ann. Phys., Lpz. (5) 26, 121 (1938).
12. Sette, D., Nuovo Cimento Suppl. 6, ser. 9, No. 1 (1949).
13. Born, H., Z. Phys. 120, 383 (1942).
14. Muller, H., and T. Kraefft, Z. Phys. 75, 313 (1932).
15. Bucks, J., and H. Muller, Z. Phys. 84, 75 (1933).
16. Richardson, E. G., J. Ac. Soc. A. 4, 177 (1933).
17. Hippel, A. V., Ann. Phys., Lpz. (4), 75, 521 (1924).
18. Hippel, A. V., Ibid. (4), 76, 590 (1925).
19. Bergmann, L., Der Ultraschall und Seine Anwendungen, 6th Edition, p. 209.

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Determination of Foreign Materials of Plant Origin in Cotton Linters[1]

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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 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. 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.

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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.
2. A Williams handsheet mold (10″ × 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

TABLE I
Dirt Counts in Raw Cotton Linters

Sample	Operator 1		Operator 2	
	A	B	A	B
1	20	11	21	12
2	37	22	35	21
3	28	24	26	23
4	23	38	29	46
5	12	7	7	9
6	18	25	20	24
7	17	28	16	28
8	13	9	11	11
9	12	8	10	8
10	77	54	78	53
11	150	154	160	123
12	42	32	38	31
13	16	15	12	17
14	78	66	87	66
15	9	12	10	13
16	25	9	25	9
17	122	74	97	67
18	22	57	13	52
19	21	17	20	13
20	28	26	29	20
21	41	43	33	36
22	23	11	21	9
23	8	7	9	8
24	27	14	23	13

A and B are duplicate preparations.

Mason type jar containing 1500 ml of the bleach solution at 22 ± 2C. Stir thoroughly and bleach for 20 min. At the end of the bleaching, add, with stirring, 20 ml of the neutralizing solution. Pour the pulp slurry into the Williams hand sheet mold containing about 2 in. water over the wire. Stir well and add more water until the mold is about half full. Continue to stir the slurry as the water is being drained. It is very desirable that a sheet as uniform in thickness as possible be formed. Press the formed hand sheet through the wringer to remove the excess water, but do not dry. At this point a dirt count is made which is patterned after the Tappi (Technical Association of Pulp and Paper Industry) Standard Procedures T213 M-43 (Dirt in Pulp) and T437 M-43 (Dirt in Paper).

For the present test method "a dirt" is defined as any foreign material embedded in the surface layers of the pulp sheet which has a marked, contrasting color to the rest of the sheet. The Dirt Estimation Chart, developed for the two Tappi test procedures, contains a number of black specks of different size and shape printed on a transparent sheet of film. In order to determine the exact size of the dirt particles found in the linters sheet, the projected area of a dirt particle is compared with a similar black spot of known size on the test chart. Smaller dirts with a projected area of less than 0.3 mm^2 are disregarded. Any pieces larger than 5.0 mm^2 are counted and recorded as 5:0 mm^2 dirts. The dirt count for the sample is obtained by adding all the areas of the dirt particles observed on both sides of the sheet. This number is reported to the nearest whole number of square mm of the total projected area of dirts and represents the dirt count on the particular linters sample.

Results and Discussion

Typical dirt counts range from 5–150 as shown in Table I. Present cotton linter pulp quality demands generally require dirt count levels below 36. Counts in

TABLE II
Composition of Dirts

Sample	Dirt count	Cellulose yield	% Stalks	% Cotton-seed hulls	% Cockle burs	Location of mill
1	54	72.7	100	......	......	Texas
2	27	74.9	33.3	55.5	11.2	Tenn.
3	56	69.4	10.7	......	89.3	Miss.
4	85	73.7	95.3	4.7	......	Ariz.

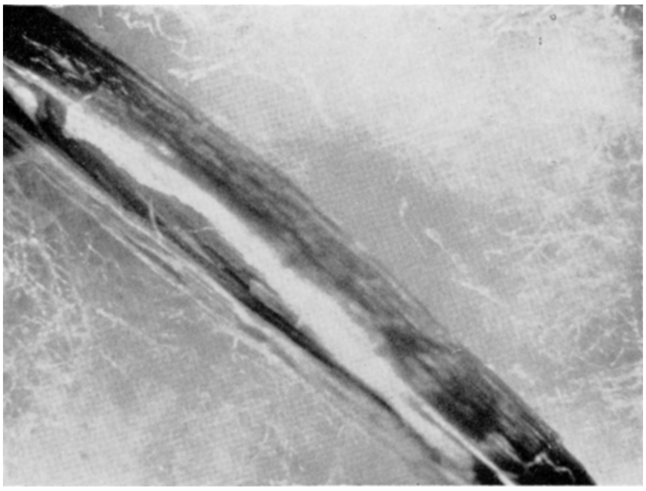

FIG. 1. Fragment of a stalk (17.5x).

the range of 80 and up represent extremely "trashy" linters. Normally the dirt counts are made on hand sheets without attempting to identify the contributing foreign material. In this work, all samples were examined in order to determine the nature of the foreign materials. Microscopic examination shows three main categories of dirts: stalks, cockle burs and cotton seed hulls. This is illustrated by the information summarized in Table II. These data represent four random linters shipments from different localities. A certain relationship is indicated between the composition of dirts and the geographic locality in which the cotton linters were grown.

Stalk materials generally are responsible for the largest portion of the total dirts, plus the majority of the large dirt particles. They are usually encountered in the form of elongated slivers (Fig. 1). These particles can originate from the stalks of the cotton plant as well as from numerous other weed plants found in a cotton field.

The next largest group, cocklebur particles, is the most spectacular group as far as the sizes and the different shapes of the particles are concerned. These particles originate from the dry fruits of the cocklebur plants (Fig. 2). The outside of the fruit coating is covered with numerous spikes. These spikes apparently break off the fruit quite easily and are encountered as small needle-like particles (Fig. 3). The middle layers of the fruit coating are heavily pigmented and particles from this layer are encountered as silvery black slivers (Fig. 4). Numerous particles

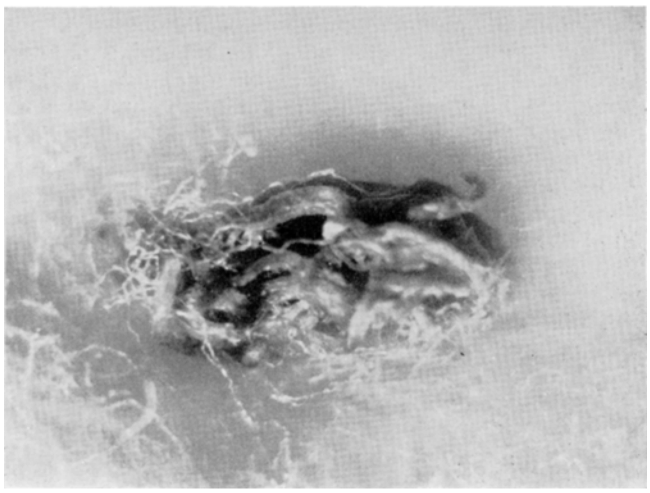

FIG. 2. Portion of the bur from a cockle bur plant (17.5x).

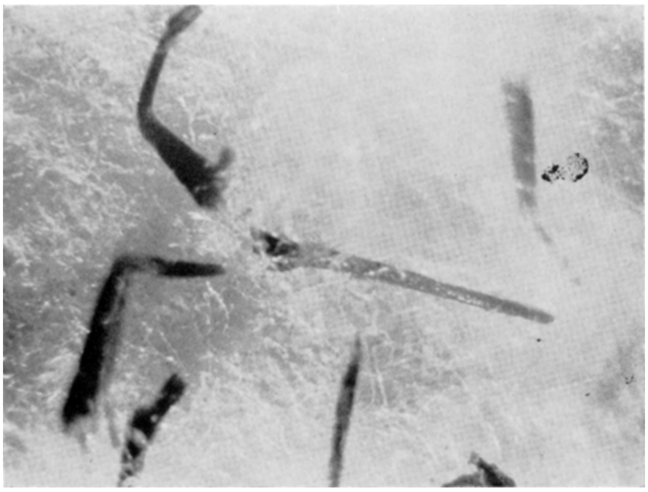

Fig. 3. Outside spikes of a cockle bur (17.5x).

originating from other parts of the cocklebur fruit coating also can be observed quite frequently. Lint grading experience at Buckeye has shown that this category of dirts is frequently overlooked in the visual estimation of foreign material.

The last group of dirts, fragments of the cotton seed hulls, apparently is a result of the processing practices at the oil mill. This group is a minor contributor to the dirt count. In the majority of the cases, these particles appear as tiny fragments in the raw linters and are destroyed by the cooking and bleaching processes. Particles counted as dirts (0.3 mm^2 and above) represent considerably larger pieces of hulls, mainly cut off in a circular form from one end of the cotton seed. These are not completely destroyed (or bleached) and are encountered as black or dark brown circular particles (Fig. 5).

A specially designed experiment was employed to measure the test variability. Samples from 24 different shipments (lots) of raw cotton linters were used in the experiment. These samples were obtained according to the requirements and regulations of the standard cellulose yield test. That is, each bale in a lot was sampled and the individual samples were blended in a mechanical mixer to obtain a composite sample representing the particular lot or shipment of linters. Duplicate cooks were made from each composite sample. A 20-g aliquot from the resulting pulp was bleached and formed into a hand sheet. The dirts on both sides of the sheets were counted by two different operators.

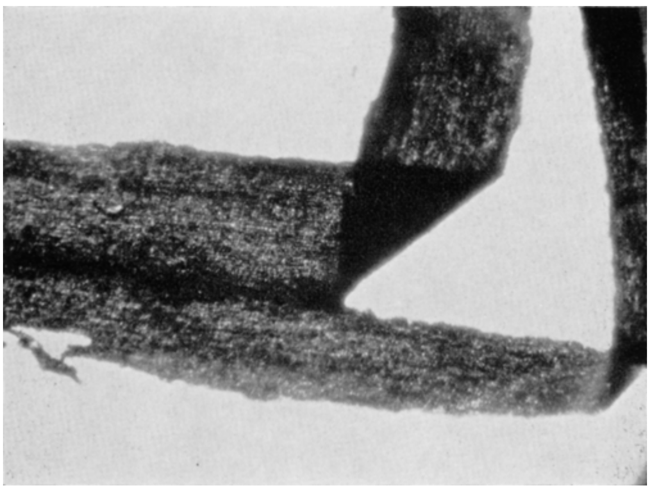

Fig. 4. Fragment of the middle layer (mesocarp) from a fruit coating of a cockle bur (35x).

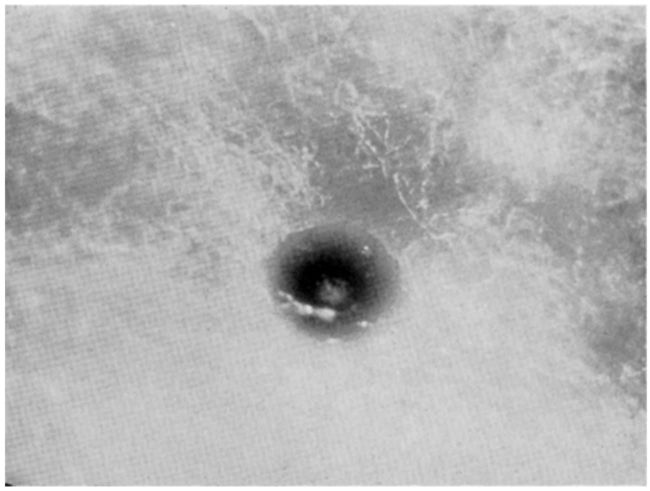

Fig. 5. Fragment from a cotton seed hull (17.5x).

TABLE III

Test Variabilities of the Dirt Count Method

Variability	%
Total	42
Between cooks within lots	39
Between operators within cooks	13

In order to calculate the test variability, it was necessary to determine the true nature of the distribution of the dirts among different lots of cotton linters. It was found that the natural distribution was logarithmic and the logarithmic transformation was therefore employed for the analysis of variance. The results show in Table III. With the logarithmic transformation, the geometric rather than the algebraic mean is obtained. The geometric mean for the dirt counts of the cotton linters samples (as listed in Table I) was found to be 23.5. The standard deviations (see Table III) are expressed as percentages of this mean.

Normally for a Poisson or logarithmic distribution one would expect a simple relation between the mean and the standard deviation. However, the data obtained in this work did not follow the general rule exactly. The standard deviation for the samples with low dirt counts was considerably smaller than the standard deviation for all of the samples. The standard deviation increased to a certain point with increasing dirt level. A further increase in the dirt level then caused the standard deviation to decrease. Such a relation between the mean value and the standard deviation is not completely understood. It is possible that this is because there is more than one type of population (either according to sources or sizes) of dirts present, and to the effect of the counting statistics upon the final results.

The calculated total test variability of ca. 40% for all 24 samples is higher than that usually encountered in chemical analysis, but is definitely a significant improvement over foreign material estimation by direct visual observation. The one-sided upper 95% confidence limit for the method was found to be 1.78 times the mean. The results show that the major contributor to the test variability is the variation between cooks of the same sample. The variability is closely connected with the non-uniform distribution of dirts within the composite samples representing each lot. The variability between counts observed by different operators is very low.

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