Automatic Measurement of Particulate Contaminants (Dirts) in Cotton Linters[1]

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ABSTRACT

An electronic image scanning measurement method was developed to obtain objective measurements of the area of resistant dark contaminants, broadly referred to as dirts, contained in cottonseed linters and hull fibers. The method is based upon the use of the AOCS standard cellulose yield residue. The yield residue (20 g) was bleached and formed into a 10 x 12 in. test sheet, and the projected area of visible dirts in 42 fields on each side of the sheet was meausred. Areas as small as 0.1 mm^2 in a field 30 x 41 mm can be measured. The results were converted to area, summed, and compared with the visual measurements obtained using the method of Jurbergs and Dowling. Statistical analysis gave standard deviations of ca. 8% and 25%, respectively, for new and the earlier procedures. The instrument standard deviation was shown to be ca. 1% relative. A comparison of the two methods using samples of 1099 different lots of linters gave a 0.94 correlation coefficient.

INTRODUCTION

Cotton linters, a product of the cottonseed industry, are used to produce chemical cellulose which is a raw material for such products as films, plastics, lacquers, propellants, laminants, and fine papers. Historically linters have been characterized by empirical quality factors. One such factor is the presence of foreign material originating from such sources as cotton stalks, weed plants, and cockleburs. This foreign material is termed dirts, and this is the term used in this paper. An excellent description of such dirts is given by Jurbergs and Dowling (1).

[1]Presented at the AOCS Spring Meeting, New Orleans, April 1973.

Many of the pulp uses demand the linter cellulose (pulp) be free of foreign particulate matter to obtain a specified quality in the final product. The presence of dirts in cotton linters presents, therefore, a problem of economic importance to the pulp manufacturer. To meet his cutomer's needs, information is needed about the dirts contained in the raw linters which may be expected to survive the usual manufacturing chemical treatments. This is necessary for balancing raw linter quality with the quality required in the cotton linter pulp.

An analytical method to obtain a quantitative estimate of the quality of cotton linters was published by Jurbergs and Dowling (1). That procedure was subjective, so other approaches to development of an objective procedure were considered. Among these were gravimetric procedures in which solvents, such as sulfuric acid and cupriethylenediamine, were used to dissolve the linter fiber leaving a residue of the undissolved nonlinter particulate matter. These approaches gave interesting information about the dirts but were discarded because the information could be misleading as a result of the different densities of the various dirts and the differing amounts of material dissolved by the solvent. The approach then was turned to quantifying either the number or the area of dirts in a standard sample using presently available equipment.

The Papric Dirt Counter developed at the Pulp and Paper Research Institute of Canada (2) determines the number of surface dirts in a test sheet that exceeds a given threshold size. This approach has the disadvantage that no distinction is made between large and small or gray and black dirts although the instrument can be set to provide band counts for differing thresholds. Other equipment, based upon image analysis with appropriate logic, can provide automatic direct measurement of surface dirt particles. Investigation of equipment of this type indicated that direct, objective measurements of projected area of dirts in the

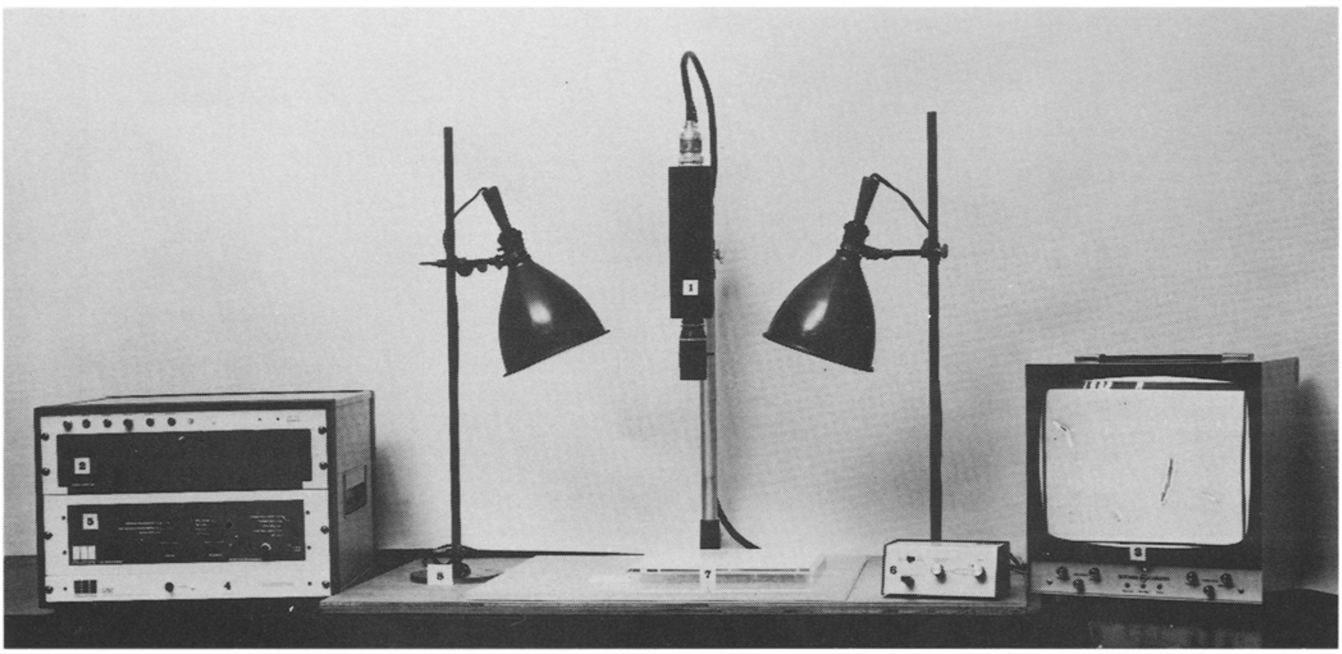

FIG. 1. The electronic dirts measurement system. The dirts contained in one camera field of a linters test sheet are shown on the television monitor. The dirts being measured, ca. 17 mm^2, are outlined in white border or appear as white objects when relatively small.

TABLE I

Comparison of Dirts Measurement

Method	Mean dirts area mm^2								
	Lot no.								
	1	2	3	4	5	6	7	8	Average
Image analysis	12.9	55.4	31.1	88.8	31.2	55.8	4.2	5.2	35.6
Visual estimation	19.5	177	64.5	410	66.5	168	6.7	4.5	114.6

sheet surface are possible. This paper describes an improved method for the grading of cotton linters. It is based upon image analysis which eliminates the subjective measurement aspects of the earlier method.

EXPERIMENTAL PROCEDURES

Materials and Equipment Used

The dirts test sample was prepared from a portion of the AOCS yield sample (Bb 3-47 Rev 1966) according to the procedure of Jurbergs and Dowling.

Bleach solution was prepared from commercial sodium hypochlorite by acidifying with sulfuric acid and diluting with water to 0.52 ± 0.02 g/liter available chlorine and acidity equivalent to 0.26 g/liter sodium hydroxide.

Bleach neutralizer solution was prepared by dissolving 250 g laboratory grade sodium thiosulfate and 20 g sodium hydroxide in 500 ml water and diluting to 1 liter. The solution is ca. 1 N in thiosulfate and 0.5 N in sodium hydroxide. Test sheets were prepared in a 10" x 12" Williams handsheet mold.

A particle measurement computer system (Millipore Corp., Bedford, Mass., ΠMC) consisting of: (A) a television camera equipped with a 15 mm extension tube and 75 mm Bolex Switar lens; (B) camera control unit, (C) monitor, (D) basic module (computer), (E) size measurement and entire field measurement logic, and (F) control box for communicating with the system was used to measure, by image analysis, the dirts in a test sheet (Fig. 1). A digital display of dirts area measurements was used to indicate the projected area of dirts contained in the surface of test sheets. Field illumination was provided by two 250W ECA 3200 K lamps to obtain 1500-1800 ft candle when measured with a photographic light meter resting in the center of the camera field. The camera head was supported in a vertical position with means to adjust its height to provide a field size of 30 x 41 mm. Test sheets were presented to the camera on a mechanical stage which permits measurement of 42 successive nonoverlapping fields/side.

Reference standards were used to obtain routine checks on the instrument imaging and measurement systems. An imaging standard was constructed by placing 0.1, 0.2, 0.3, and 0.5 grayness chips removed from a photographic grayness standard (Eastman Kodak, Rochester, N.Y.) on a background approximating the average test sheet grayness, ca. 0.1. The instrument distinguished by outlining and measured at the 0.2-0.3 level. A size standard was prepared from the 4.0 mm^2 black spot removed from the Dirt Estimation Chart (3). The average instrument measurement for 10 readings distributed across the top, middle, and bottom of the field allowed calculation of a conversion factor between instrument units and dirts area. The variation of successive measurements was less than ± 1.0%. Measurements at different positions in the field did not exceed ± 2.0% of the value at the center of the field. A photographic dirts area standard was made by photographing representative linters dirts removed from linters test sheets and distributed within one camera field on a typical test sheet. The dirts in the photograph were measureed successively to obtain a working value. The variation in the established value did not exceed ± 2%.

Preparation of Sample Test Sheet

A 20 g portion of the cellulose yield residue was mixed with 1500 ml bleach solution in a 2 qt glass jar and bleached for 20 min at 22 ± 2 C. Residual chlorine was neutralized with 20 ml thiosulfate solution, and the slurry was poured into the sheet mold. Water was added to fill the mold ca. half full, while mixing by hand to obtain a

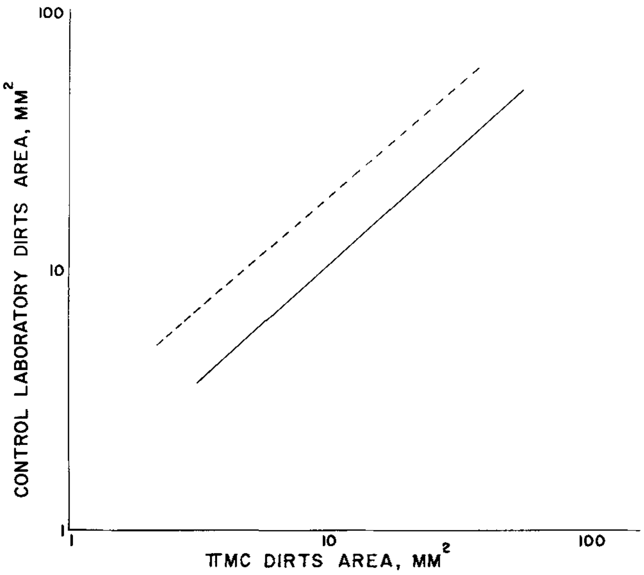

FIG. 2. Area of dirts as determined on wet linters test sheets by the visual method vs. area as determined by the electronic Particle Measurement Computer (ΠMC) system on, —wet test sheets, and –on the corresponding dry test sheets.

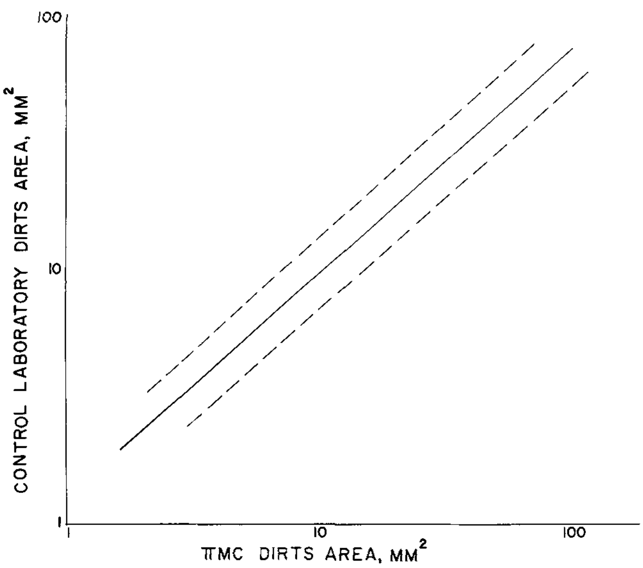

FIG. 3. Regression line showing correlation between the visual method and the electronic method of dirts measurement with standard deviation limits, ± 0.1540 logarithm. — dry sheets, —— wet sheets.

TABLE II

Comparison of Wet and Dry Test Sheets

Statistical treatment	Visual estimate Wet	Image analysis Wet	Image analysis Dry
N	70	70	70
Log $\bar{x}$	1.5353	1.5792	1.2969
Regression equation log y =		0.9066 (log x)+0.1037	0.8609 (log x)+0.4188
Standard error of log y		0.1338	0.1853
Correlation coefficient, percent		94.06	88.26

uniform dispersion of fibers. Water then was drained away only until the surface assumed a glassy appearance. Final improvements to handsheet uniformity were made by lightly patting the liquid surface with palm of hand or suitable object. Remaining free water then was drained away, and the sheet was couched and pressed to ca. 30-35% dryness. The sheet then was placed upon a blotter, ca. 12 x 15 in. for measurement of dirts.

Dirt Measurement

The ΠMC system should be warmed up at least 10 min prior to use. The operating mode requires that the lamps be positioned to provide 1500-1800 ft-c field illumination, height of camera be adjusted to provide a field size 30 x 41 mm, camera lens be open to f 1.9 and brought to sharp focus in the plane of the test sheet, control box be set for black particles and mode to automatic, size measurement module objective power be set to 100, readout mode to sum successive measurements, and entire field measurement plug-in set to total area. The 10 x 12 in. test sheet is supported on a white blotter on the mechanical stage to begin measurements at one corner of the sheet. With the test sheet aligned on the mechanical stage, the projected area of dirts of the first field is measured by depressing the count/measure switch of the control box. The stage is moved to successive fields of the grid until all 42 fields, 7 rows of 6 fields, in the first side of the sheet are measured. The sheet is turned over, and the measurement of 42 fields is repeated on the second side. The total projected area of dirts measured in the 84 fields, equivalent to ca. 70% total sheet surface area, is displayed on the monitor masthead.

Calculations

The area of dirts for the sample is reported as mm^2 of total projected dirts area. This value represents the dirts quality of the particular sample. Dirts area mm^2 = total instrument units/conversion factor.

RESULTS AND DISCUSSION

Image Analysis vs. Visual Estimation

A comparison of the 2 analytical methods was made using 8 lots of linters selected to provide 2 lots for each of 4 levels of dirts as estimated by the visual method, ca. 5, 10-50, 50-75, and ca. 100 plus. One handsheet was made from each of duplicate yield cooks of the linter samples representing the 8 lots. Triplicate preparations of the set of 16 handsheets provided 48 test sheets. Single visual estimates were made on each sheet while wet. Duplicate instrument measurements were made after the sheets had dried. The raw data were evaluated by regression analysis with best fit of the data being obtained with common logarithm transformation. Because past estimates of linters quality have been based upon the visual method, correlation of the two methods was of interest in the utilization of linters. The results are summarized in Table I. Correlation between the two methods was ca. 94%.

Wet vs. Dry Sheet in Dirts Measurement

A test was run to compare the precision of using dry or wet test sheets directly in the image analysis procedure. This would be consistent with the earlier procedure in which the area estimates were made immediately following the preparation of the test sheet. Advantages to be gained using wet test sheets are savings in test time, elimination of errors caused by variations in areas of dirts resulting from shrinkage during drying, and elimination of changes in optical density of some dirts that occur with drying.

A broad base for this comparison was available in the use of the control laboratory routine test sheets of the daily linters receipts. On completion of the routine tests, the wet sheets were tested on the ΠMC system. The sheets were allowed to dry and were remeasured the following day. The analysis of the data thus obtained is shown in Table II. The wet vs. dry sheet comparison was made using the control laboratory routine visual estimate as a common reference base. A graphical representation of the data (Fig. 2) shows the effect of sheet drying upon dirts measurement. Area measurements with wet sheets are ca. twice that with dry sheets, but the more important finding is the improved precision that is obtained with the wet test sheets. For this reason the procedure for further work was standardized on the use of never dried sheets.

Precision of ΠMC Dirts Measurement

The reproducibility of the instrumental measurement was obtained using wet routine dirts test sheets. Test samples (13) covering a wide range of dirts, 68-617 mm^2, were selected for measurement. Each sample sheet was measured by scanning 42 camera fields on each side. The measurements for each side were recorded separately. On completion of the first measurements, each sheet was returned to the camera stage, and the measurements were repeated. The variation in duplicate measurements from their mean was calculated for each side of the 13 sheets. An average variation of 1.13% was obtained from the 26 comparisons. The data are shown in Table III. Differences can be seen in the amount of dirts in the two surfaces of the sheets, and this is the reason that measurements of both

TABLE III

Dirts in Wet Quality Control Linters Test Sheets, Equivalent mm^2[a]

Sample no.	Side 1 (mm^2) Test 1	Side 1 (mm^2) Test 2	Side 2 (mm^2) Test 1	Side 2 (mm^2) Test 2
1	89	88	75	76
2	269	268	320	312
3	20	21	56	53
4	30	29	38	38
5	99	101	92	90
6	78	78	87	88
7	82	84	58	88
8	116	123	119	120
9	87	86	106	110
10	37	38	44	40
11	54	55	64	64
12	291	288	332	322
13	94	89	92	92

[a]Deviation from average of pairs ± 1.13%.

sides must be made to obtain a true rating of the linter quality.

Application of Image Analysis in Linters Dirts Control

A test of the suitability and reliability of the automatic electronic procedure to determine cotton linters quality was made by using the ΠMC system in parallel with the control laboratory routine visual method for testing daily linters receipts. During this period a total of 1099 lots representing over 100,000 bales of linters were tested by both procedures. The data, treated by regression analysis, are shown graphically in Figure 3. Standard deviation limits are indicated by the broken lines. The regression equation is log y = 0.8839 (log x) + 0.1039 where y = control laboratory dirts area and x = ΠMC dirts aea. The standard error for log y = 0.1540. A 94.0% correlation between the two procedures was obtained.

The new instrumental method for measurement of dirts in cotton linters has been put into routine service to replace the visual estimation method in this laboratory. Because of its possible value to others, instrument calibration procedures have been defined which should make it possible to calibrate other similar ΠMC systems for this type of service. Two camera systems, loaned by Millipore Corp., were installed in the Buckeye ΠMC system, replacing its camera system and were checked using the calibration procedure. Results were obtained which are comparable to those obtained with the complete Buckeye ΠMC system.

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