This order is slightly at variance with those obtained in the detergency tests of the alkalies in combination with Santomerse No. 1. It is in fair agreement with the detergency results for the builders alone in soft water, but the correlation is poor for hard water where the detergency results exhibited no evidence of marked variation between the alkalies. It is hardly to be expected, however, that there should be exact correlation between two such dissimilar systems as graphite-mineral oil-vegetable oil and degreased burnt umber. The fact that certain of the builders exhibited high effectiveness in each of several series of tests is an indication that these materials would probably be effective for removal of soils under a variety of use conditions.

Summary

Tests made with mixtures of Santomerse No. 1 or No. 3 with TSPP at 0.05% (in synthetic sea water) indicate optimum soil removal for combinations of 80% TSPP and 20% Santomerse. At this solution concentration TSPP alone removed more soil than either Santomerse product.

Tests made in hard water at 0.075% solution concentration with Santomerse combined with various alkaline soap builders in 1:1 ratios of Santomerse No. 3 to anhydrous alkaline builders permitted arrangement of the alkalies in decreasing order of effectiveness as follows:

TSP
TSPP
TSPP—Orthosilicate (1:1)
TSPP—TSP (1:1)
Orthosilicate and metasilicate
Sodium carbonate

Further tests at the same solution concentration were made with hydrous builders on an "as received" basis but with Santomerse No. 1 to builder ratios of 67:33 or 75:25. The order of efficiency of the builders was the same as for the previous tests, and there were negligible differences between the two ratios tested.

Detergent testing of several of the alkaline soap builders indicated relatively high soil removal at low solution concentrations in comparison with Santomerse No. 1 and accounts for improved detergency of its mixtures with alkali.

Burnt umber peptization values tend to corroborate detergent findings.

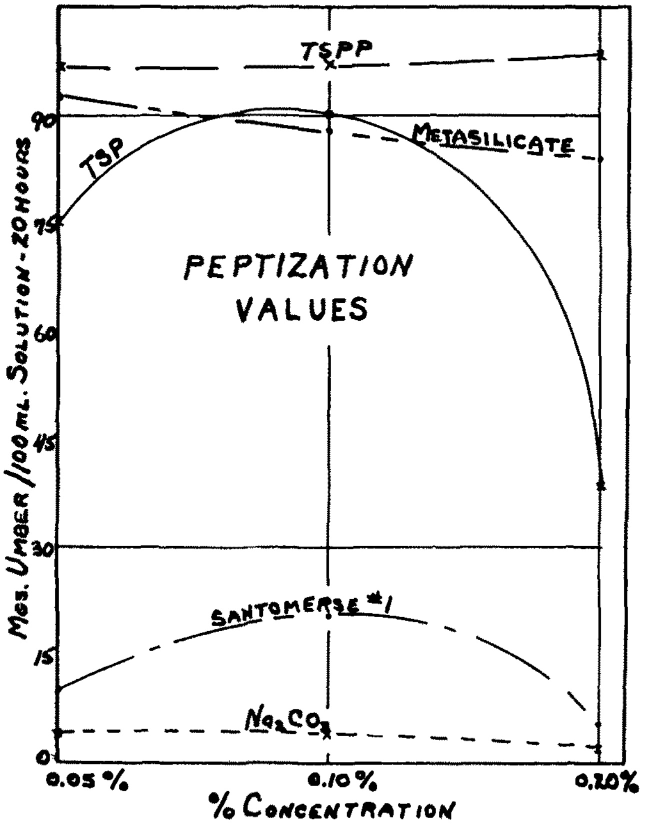

FIG. 4. Peptization values. Milligrams of Suspended Umber/100 ml. solution.

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The Gravimetric Determination of Foreign Material in Cellulose Fibers

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Summary

A METHOD is offered for the gravimetric determination of foreign material in cellulose fibers which is particularly applicable to cottonseed linters, fiber, and motes. The procedure is relatively simple and rapid, requiring only equipment and technique generally in use in the cottonseed industry's laboratories. Determinations are reproducible within significant limits. The foreign material, seed, hulls, hull bran, etc., is recovered (excepting particles finer than 50 mesh) so that it can be examined and its nature classified as to type. Use of the method would give the lint producer a dependable index to relative lint cleanliness, and the effect of modifications in seed cleaning and lint room operation aimed at improved lint quality.

Introduction

The method proposed, while applicable to various types of cellulose fibers, was developed by experimentation with cottonseed linters, primarily "second-cut"

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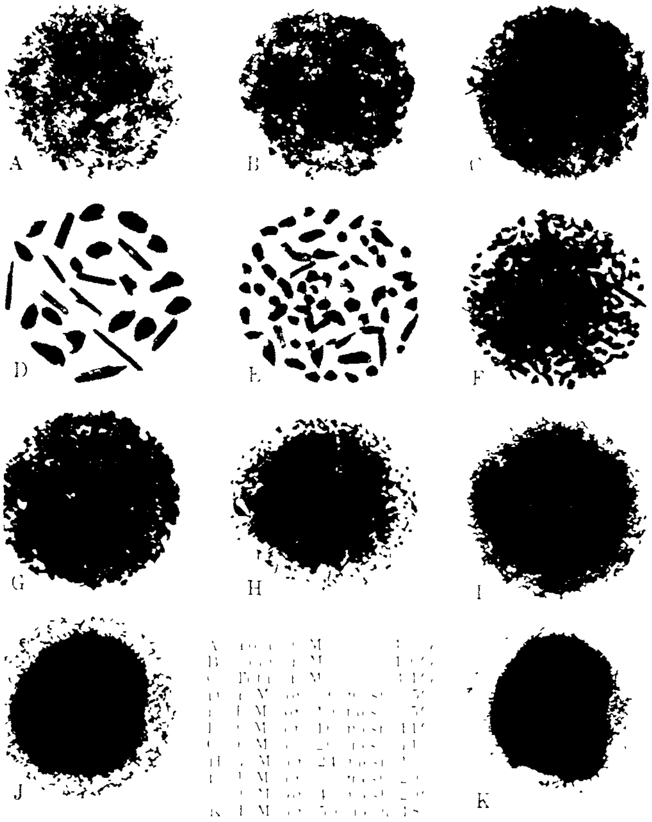
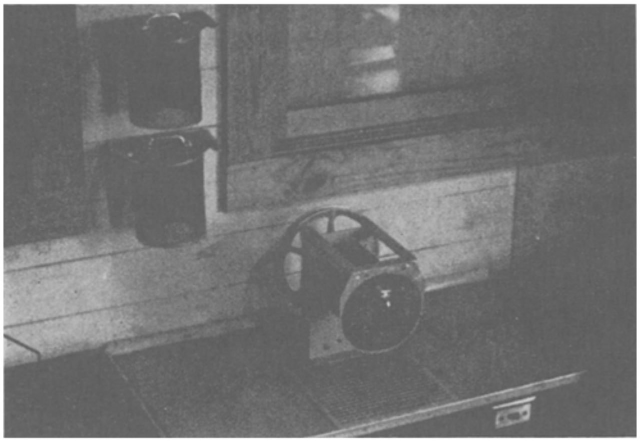


large sample was then screened on a series of sieves to the size fractions shown in specimens D through K. The percentage of the foreign material remaining on each sieve size is tabulated on the photograph. This demonstrates the relative amounts of the several types and sizes of foreign material coarser than 50 mesh as found in a series of normal second cut lint samples.



over-all time required for a series of determinations is about two hours, with about fifteen minutes of operator time per sample. Reagent cost is negligible.

Discussion

The use of this method can enable the lint producer to form a reasonably reliable estimate of the quality of lint produced, as affected by foreign material, and to judge the effectiveness of modifications in seed cleaning and manufacture aimed at improving lint cleanliness. The method is empirical in that 1, only foreign material coarser than 50 mesh is recovered; 2, the acid and friction treatment reduces the size of particles slightly; 3, leaf and shreds of stems disintegrate and are not fully recovered in size or amount; 4, corrections for moisture variation are not made. Results are, however, representative of the sample and reproducible; are not subject to personal opinion; and give reliable measurements of relative lint cleanliness. The procedure is applicable to first-cut lint, mill-run, fiber and motes, as well as second-cut lint.

The use of a 50 mesh sieve has been specified for this determination of foreign material in order to obtain relatively quick separation. Second-cut linters normally contain a considerable amount of hull particles which will pass this screen size, but this fine bran, while affecting the cellulose yield, does not have so important an effect on the cleanliness rating of the linters. If for special investigation it is desirable to recover the maximum of non-lint material in a sample of cellulose fibers, sieve sizes as fine as 100 mesh may be used. This requires protracted hand brushing, or the use of a mechanical brushing device.

Analytical Procedure

Apparatus. Fuming Ovens and Fuming Pots as given in Rules of the National Cottonseed Products Association, No. 270 A, Section 3. One U. S. 50 mesh sieve. One (approximately) 8 mesh sieve. One 2" varnish brush.

Determination. Mix the lint sample in a Mechanical Lint Mixer as described in N. C. P. A., Rule 272, Section 1. If a mechanical mixer is not available, mix the sample by hand, reincorporating and uniformly distributing the bran.

Prepare a porous, earthenware vessel by absorbing 2½ ml. of conc. HCl into the inside walls and bottom. The acid should be distributed over the inside area of the pot and absorbed completely so that no liquid remains. Place 20 ± .2 g. of the mixed lint sample in the pot, cover, and heat in a suitable oven at 115° C. for one hour. Remove pot cover and continue heating in ventilated oven for 30 minutes. The lint should be dry and brittle, and only slightly discolored.

Transfer fumed lint to a coarse sieve of about 8 mesh and force lint through with the fingers or a small block. This speeds the subsequent brushing operation. Transfer lint and any large foreign matter which may remain on this sieve to a U. S. No. 50 sieve. Brush lint through with a brush (a 2 inch varnish brush is recommended) using a circular motion and frequent tapping of the sieve. Remove and weigh the lint-free foreign material.

5 × wt. of f. m. = % Foreign Material

The presence of a single whole seed, or a large piece of foreign material in the residue, which would indicate that the 20 gram portion was not representative, would require a second determination.

The recovered foreign material may be screened on several different screen sizes for a quantitative determination of the several types of material present.

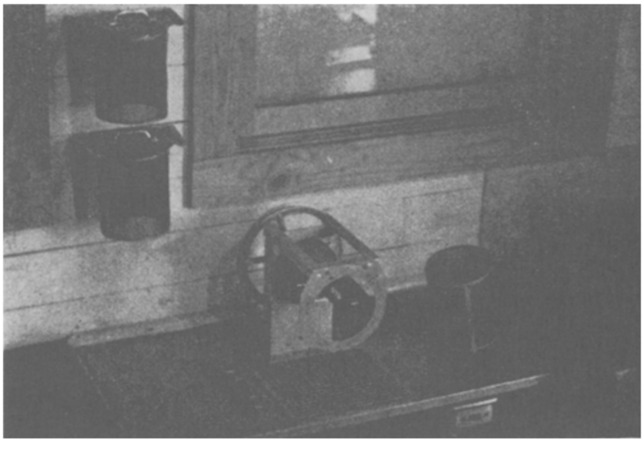
Brushing Machine

AS HAND brushing of the fumed lint through a No. 50 (or finer) sieve is tedious, a brushing machine was constructed with the collaboration of J. D. Atkinson, Jr.

The machine consists of a ½" di. horizontal shaft supported by a bearing with a pulley on one side, and extending six inches on the other. The latter extension is drilled for ⅜" cross arms, ½" and 4" from the end, which pass through the shaft and extend 1½" in line on either side of the shaft. Two copper sheets 6" x 1" are folded lengthways to form clips, into which 6" lengths of mill linter brushes with bristles trimmed to an over-all width of 2" are clamped. Two eyes are soldered to one side of each clip, so spaced that two cross arms will pass through, allowing the clip to move freely on and off.

A cylinder is made of U. S. No. 50 (or finer, if desired) wire cloth 6" in di. and 8" long. A bottom copper plate 9" di. is attached, and a false bottom is soldered in 1" above the bottom plate. The drive shaft extends through the center of a disc which is fastened to the main bearing support. The disc is 6" in diameter and 1" thick, tapered to form a tight joint with the open end of the sieve cylinder. Two horizontal arms, above and in line with the shaft, are attached to the bearing support and have attached a vertical plate with a 6⅛" circular opening.

The sieve cylinder is slipped through this opening,



which supports it at the closed end, the open end being plugged by the disc. The shaft is driven at about 250 r.p.m. The horizontal brushes, which move freely on the cross arms, press against the cylinder wall with only centrifugal force.

The fumed lint is put in the cylinder which, with the brushes in place, is slipped through the supporting opening and over the disc plug. The motor is started and run until lint dust ceases to fall. The outside of the cylinder is brushed or blown free of lint. A sheet of paper is placed below the cylinder, the latter is removed and dumped onto the paper, and the brushes freed of all foreign material, which is collected on the paper and all transferred to a container for weighing.

Machine brushing requires from 1 to 2 minutes against about 15 minutes for hand brushing.

Thermal Properties of Fats and Oils

IV. Some Observations on the Polymorphism and X-Ray Diffraction Characteristics of Tristearin and a Highly Hydrogenated Cottonseed Oil

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AS a preliminary step in the calorimetric examination of certain highly saturated fats, it was considered necessary to investigate the polymorphic behavior of these materials under the conditions that obtain in the calorimeter. Saturated glycerides are known to be capable of existence in a multiplicity of polymorphic forms, and these presumably differ in their heat capacities and other thermal characteristics. For identification of the different polymorphic forms, melting point determinations were supplemented by x-ray diffraction measurements. The results point to the existence of hitherto unreported crystal structures in the lower-melting forms, and are otherwise of more than incidental interest; consequently, they are here made the subject of a separate communication. In the present literature there is virtually nothing dealing with the subject of triglyceride crystal structure beyond the publications of Malkin and coworkers (2,3,6,7).

Preparation and Composition of the Samples

THE samples examined consisted of cottonseed oil, hydrogenated in the laboratory almost to completeness with a nickel catalyst, and a highly purified sample of tristearin. The estimated fatty acid composition of the cottonseed oil, before hydrogenation, was recorded in a previous communication (8). According to the data of Hilditch and Maddison (5), cottonseed oil of this composition, when highly hydrogenated, should consist principally of β-palmitodistearin and tristearin, in the ratio of about 3 parts of the former to 1 of the latter. The hydrogenated oil had an iodine value of 0.85; hence it probably contained about 3 per cent of oleodisaturated glycerides. Its content of free fatty acids was 0.30 per cent.

Samples of tristearin were prepared by reacting stearoyl chloride in 10 per cent excess with glycerol and quinoline in chloroform solution, according to the method of Averill, Roche, and King (1), and repeatedly crystallizing the product from ether containing a small amount of alcohol. Stearic acid for preparation of the acid chloride was derived from a commercial product of about 90 per cent purity (Neofat No. 1-65) and was purified by treatment with sulfuric acid, followed by water washing to remove unsaturated products, fractional distillation in the form of the methyl ester, and multiple recrystallizations of the regenerated acid from acetone. This method is believed to yield saturated triglycerides of an exceptionally high degree of purity. For the production of a very pure material the step of sulfuric acid treatment of the fatty acids is considered important. By this means traces of unsaturated fatty acids in the product are sulfonated so that they may be subsequently removed by washing with water. Traces of unsaturated C_{18} acids are difficult, if not impossible, to remove by either fractional crystallization or distillation.

TABLE I

Melting Points of Different Polymorphic Forms[1] of Tristearin, β-Palmitodistearin, and Highly Hydrogenated Cottonseed Oil (I.V. = 0.85)

	M. P., °C., of form indicated			
	I	II	III	IV
Tristearin (Clarkson and Malkin)	71.5	65.0	54.5	......
Tristearin (present investigation, Sample 1)	72.0	64.5	54.5	......
Tristearin (present investigation, Sample 2)	71.8	64.5	54.5	......
β-Palmitodistearin (Malkin and Meara)	68	64	56	50
Hydrog. cottonseed oil (present investigation)	62.3	61.0	58.5	50.5

[1] Following the scheme adopted by Daubert and Clarke (4) the different polymorphic forms are designated as I, II, etc., simply according to their melting points and without regard for the geometry of the crystal structure involved. Forms I, II, and III of tristearin correspond respectively to the β, α, and "vitreous" forms of Malkin and coworkers. The above tabulated forms of β-palmitodistearin are designated by Malkin and Meara as β, β′, α, and "vitreous" in decreasing order of melting point.

Melting Points of the Samples

By application of the technique described by Clarkson and Malkin (3), wherein solidified samples in capillary tubes are plunged into constant-temperature water baths of different temperatures, it was possible to distinguish four different crystalline modifications in the hydrogenated oil. The melting points of the oils containing these modifications are listed in Table 1, together with the melting points reported by Clarkson and Malkin for tristearin, those found in the pres-

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[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.