THE DETERMINATION OF THE LINT ON COTTONSEED AND THE CELLULOSE IN HULL FIBRE

By R. S. McKINNEY and G. S. JAMIESON

Bureau of Chemistry and Soils, Washington, D. C.

THE present price of cotton linters makes the lint content of cottonseed important in evaluating and grading the seed. Therefore an accurate and simple method for determining the lint content is worthwhile.

The use of concentrated sulfuric acid, as outlined in the present official method on cottonseed hull analysis, has many disadvantages, not the least of which is the amount of acid required and the danger to the analyst, handling many samples each day, of being burnt with the acid.

The following method was found to yield results which checked within 0.2 per cent when performed in duplicate. In a trial run, this method gave results which are comparable with plant yields. The method is simple, and the apparatus is that now used in the laboratories analyzing cottonseed. Moisture changes are taken care of in the calculation, and the amount reagents and the time required are reduced to minimum.

Method: Weigh 50 grams of cottonseed and place in a porous earthenware vessel, such as a 3-inch flower pot, in the walls of which 3 cubic centimeters of concentrated hydrochloric acid have been absorbed. Cover the pot with a watch glass or other suitable cover and place it in an oven at 130° C. for one hour. Allow the sample to cool to room temperature and reweigh on a balance sensitive to 0.05 grams. Place the sample on a 10-mesh sieve and rub off the lint with a No. 11 solid rubber stopper. Remove the last traces of lint by rubbing the sample on a towel and reweigh the sample. The loss in weight of the dried fumed seed gives the weight of dried lint on the seed. The determination is made in duplicate. It was found that this dried lint contained an average of about 2 per cent of moisture. The weight of lint on the seed can be calculated from this weight of dried lint, using the average moisture on lint as actually determined. The following is an example of the calculation:

Original sample	50.00	50.00
After fuming 1 hour at 130°C	47.40	47.50
After removal of lint.......	41.15	41.25
	6.25	6.25
	2	2
Per cent dried lint.....	12.50	12.50

$12.50 \times \frac{98}{92} = 13.3$ per cent lint. (Assuming 2% moisture in dried lint and 8% moisture in lint.)

It will be observed that this method determines the total lint content of the ginned cottonseed. For those not familiar with oil mill operations, it may be mentioned that only a portion of the lint is cut from the seed by the delinting machines and this is known in the trade as "linters." The residual lint on the seeds, which are then decorticated, serves to float the hulls above the meats in the separating shaker screens. The separated hulls are ground in an attrition mill, and the lint is separated from the hull bran by aspiration (a regulated air current). The aspirated product, as would be expected, consists of a mixture of lint and fine hull particles, known in the trade as "hull fibre."

In view of the growing importance of the commercial production of hull fibre for use as a source of cellulose for the chemical industry, it is desirable to have a method for the determination of the lint and hull (bran) content of this product.

The lint consists chiefly of cellulose. K. S. Markley (J. Am. Soc. Agron. 20, 1102 (1928) has shown that cottonseed hull bran, on the air-dry basis, is composed of 38.02 per cent of pentosans, 20.91 per cent of lignin and 45.36 per cent of crude cellulose. The cellulose is only slightly affected by boiling with 8 per cent sodium hydroxide solution, whereas the pentosans and lignin of the hulls are dissolved by this treatment. A method has recently been developed by which the proportions of lint and hull can be determined.

The effect of alkaline digestion on pure cottonseed hulls and cellulose fibre was determined. Pure cottonseed hulls were obtained by cutting them from bald cottonseed. For analysis, the separated hulls were finely ground. A moisture determination showed that they contained 9.3 per cent of moisture and volatile matter. A one-gram portion of the ground hulls was boiled for three and a half hours with 100 cc. of an 8 per cent solution of sodium hydroxide. The residue was filtered onto a tared Gooch crucible, washed free of alkali with hot distilled water, dried and weighed. It was found that cottonseed hulls lost 68.0 per cent of their weight under this treatment. This loss in weight of the hulls includes the original moisture content. This loss of the weight of hulls, due to the chemical action of the alkali, is therefore the difference between this loss in weight and the original moisture content of the hulls or 58.7 per cent. Pure cotton lint was taken as a source of cellulose fibre. It was found to contain 5.4 per cent of moisture and volatile matter. When subjected to the same alkaline treatment as that applied to the hulls, the lint lost 12.0 per cent of its weight. Since this value includes the original moisture of the lint, the loss of weight of the lint due to the chemical action of the alkali is therefore the difference between this value and the original moisture of the lint, or 6.6 per cent.

From the moisture content of "hull fibre," and the loss in weight of "hull fibre" when subjected to the alkaline treatment, the percentage of hull and lint in the hull fibre can be calculated from the following formula:

M = per cent moisture of "hull fibre" (lint + hull).
L = per cent loss in weight of "hull fibre" when subjected to the treatment described.
X = per cent of dry hull in the "hull fibre."

Y = per cent of dry lint in the "hull fibre."

$$58.7\,X + 6.6\,Y = 100\ (L—M)$$

$$X + Y = 100—M$$

$$\text{Per cent hull} = X\left(\frac{100}{100\text{-}M}\right)$$

$$\text{Per cent lint} = Y\left(\frac{100}{100\text{-}M}\right)$$

Example: A sample of "hull fibre" which contained 6.1 per cent of moisture lost 26.2 per cent of its weight when subjected to the above-described alkaline treatment.

$$58.7\,X + 6.6\,Y = 2010 = 100\ (L - M)$$

$$X + Y = 93.9 = (100 - M)$$

X = 26.7 per cent of dry hull
Y = 67.2 per cent of dry lint

$$\text{Per cent hull} = 26.7\left(\frac{100}{93.9}\right) = 28.4$$

$$\text{Per cent lint} = 67.2\left(\frac{100}{93.9}\right) = 71.6$$

THE OXIDATION OF FATS IN STORAGE

By GEORGE R. GREENBANK

Research Laboratories, Bureau of Dairy Industry,
U. S. Department of Agriculture

THE rate of oxidation of fats in storage is influenced by the following factors: (1) The kind and amount of unsaturated fatty acid constitutents of the glycerides; (2) those constituents which act as antioxidants and prooxidants; (3) the conditions to which the fats are subjected.

In the present discussion an attempt will be made to show the relative effect of each of these factors on determining the susceptibility of various fats to oxidation.

The term "tallowiness" will be employed to denote those flavors and odors which are the result of oxidation while the term "rancid" will be used to denote other flavors and odors except "fishiness," which results from the normal hydrolysis of a fat.

Normal Constituents of Fats

Free fatty acids—Of the normal constituents affecting oxidation the amount of free fatty acids is the most easily controlled. It has been known for many years that fatty acids are prooxidants. Harris (1) finds that the wet method of rendering fats causes a high free fatty acid content. He also observes that lard of low free fatty acid remains sweet even when kept in storage for a long time. Rogers and Gray (2) observed that butter made from sweet cream kept better in storage than butter made from sour cream. This difference in keeping quality is probably due to the lower free fatty acid content of the sweet cream butterfat. Butter made by this method may be kept in storage for months without much deterioration.

To determine the effect of fatty acids on oxidation, comparable quantities of different acids were added to 10 cc. samples of fresh butterfat. The samples were exposed to air in the absence of light. As soon as one of the samples had lost its color, all samples were removed and the extent of the oxidation was determined. Table I shows the progress of the oxidation.

Unless otherwise stated the fat employed in this discussion is butter fat.

TABLE I.—CATALYTIC EFFECT OF FATTY ACIDS

Sample	Color	Relative extent of oxidation*
Control	+ + +	0.0
" + butyric acid	+ +	1.0
" + caproic acid	+	1.2
" + oleic acid	— (bleached)	12.0

*Determined by Kreis test (10).

In view of this catalytic action of the fatty acids in the oxidation of fats an attempt was made to improve the keeping quality through a reduction of the free fatty acid content by neutralization and washing.

Dilute NaOH was first employed, using phenolpthalein as an indicator. After neutralization, the butterfat was thoroughly washed and dried, and found to be slightly acid. This acidity was probably due to further hydrolysis of the glycerides. This fat resisted oxidation for only one hour at 95° C., while the control resisted oxidation four hours at the same temperature.

Neutralization with $Ba(OH)_2$ and $Ca(OH)_2$ was employed, but the results were similar. Total or partial removal of the free fatty acids from a fat through treatment with alkalies and washing seemed to lower the keeping quality. This explains to some extent the loss of keeping quality in alkali refining of cottonseed oil.

In an attempt to isolate the antioxidants in cottonseed it was found that the only extract having any antioxygenic value was water soluble and acidic. Therefore, in alkali refining this substance would be lost.

An attempt was then made to remove some of the acids through steam distillation. Accordingly, 400 cc. of butterfat were treated by passing steam through the fat. At intervals of 15 minutes, samples were taken and the acidity determined. As soon as two consecutive samples gave the same acidity the treatment was discontinued. Continued treatment would result in hydrolysis of the glycerides.

A number of samples prepared in this manner were dried and sealed under high vacuum (0.1 mm. Hg). One of these samples after exposure to direct sunlight for three months, and subsequent storage in diffuse light of the laboratory for three years, showed a negative Kreis test. The untreated samples, stored under similar conditions were completely bleached and tallowy in three weeks. The free oxygen necessary to produce such a change could not have been present in the latter under the conditions. A source of oxygen must, therefore, be assumed (5). Oxidation in vacuum may be explained by assuming the existence of loosely bound oxygen compounds. These labile compounds probably break down in storage liberating free oxygen which attacks the oleic acids radicle at the double bond. This loosely bound oxygen may be the source of the active