

these experimental samples since there is always some blending of seed of different production lots at the gin and mill and later of oils. However the results should interest oil processors desiring oils of certain iodine values for the production of special products as they offer a basis of selection of source areas, knowing the temperatures prevailing during the development of the cottonseed.

Summary

Data are reported on the variation of the iodine value of the oil from the seed of eight varieties of cotton grown at 13 locations during three years. Analysis of variance showed the influence of variety and of station-years to be highly significant statistically. Iodine value was found to be negatively correlated with the temperatures. The highest correlation was obtained for the period of maturation (35 days before the bolls open) when the oil is being synthesized. The coefficients for the relations with mean temperatures were higher than those for maximum and minimum temperatures.

Simple correlations for the relations between iodine value of the oils from seed of individual varieties and mean temperatures during two periods of boll and seed development were negative and highly significant. For the maturation period (35 days before boll opening) and the combined periods for squaring, fiber elongation, and maturation (73 days before boll opening) the average reductions in iodine value per °F. increase in temperature were found to be 0.760 and 1.172 units, respectively. Of the eight varieties investigated, temperature influenced the iodine value of the oil least for Stoneville 2B and most for Coker Wilds.

Acknowledgment

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Determination of the Methoxyl Content of Fats

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THE use of sodium methoxide as a catalyst for the rearrangement of lard, according to a recent patent (1), produces some methyl esters. Although the deodorization procedure removes practically all of these esters, it is necessary to measure the methoxyl content of the final product.

Chromotropic acid (1,8-dihydroxynaphthalene-3,6-disulfonic acid) has been used as a specific reagent for the determination of formaldehyde (2) and methanol (3). As the methyl esters present in the fat may be saponified and the methanol removed by distillation, a procedure was developed that will measure less than 0.0005% methoxyl in fat.

Experimental

A 100 g. sample of the fat is saponified in a mixture of 30 g. potassium hydroxide, 25 ml. of water, and 75 ml. of diethylene glycol. The saponification is carried out in a one-liter, one neck, round bottom flask, which is equipped with a six-inch distillation column and a condenser leading to the receiver. A dropping funnel at the top of the column is used to add acid and water to the saponification mixture.

A hemispherical Glass-Col heater is used, and agitation is produced by a magnetic stirrer. The mixture

is heated and stirred until saponification is complete, as evidenced by a homogenous solution. It is then acidified by the addition of 18 ml. concentrated sulfuric acid in 50 ml. of water, and a slow distillation is carried out until about 50 ml. of distillate have been collected. The column is washed down several times during the distillation with small portions of water added through the dropping funnel. The distillate obtained in this manner consists of a very dilute solution containing not only methanol but also steam volatile fatty acids, some aldehydes, and other materials. To remove the fatty acids and the aldehydes the solution is redistilled from a small distillation flask after adding about 1/2 g. of lime and about 1/10 g. of meta-phenylene diamine hydrochloride. A 100-ml. volumetric flask is used as a receiver. The distillation is carried almost to dryness and then water added and distilled until almost 100 ml. have been collected. The receiver is filled to the mark with water.

The oxidation and color-forming reactions are carried out in 25-ml. volumetric flasks. Aliquots of the distillate, which contain less than 0.2 mg. of methanol, are pipetted into the flask, diluted to approximately 10 ml., and oxidized by 1 ml. of a potassium permanganate solution containing 6 g. KMnO_4 , 200 ml. of

water, and 30 ml. phosphoric acid. After 2 minutes the excess permanganate is reduced by adding 5% sodium bisulfite solution. One ml. of a 10% solution of chromotropic acid (1,8-dihydroxynaphthalene, 3,6-disulfonic acid) is then added, followed by 10 ml. of concentrated sulfuric acid. The flask is cooled in an ice-water bath while the sulfuric acid is being added. The solutions are then heated for ½ hour in a boiling water bath, cooled, and diluted to the mark. To obtain a blank solution for the colorimetric measurement, a sample aliquot is carried through this same procedure with the exception of the oxidation step. In this way any material which reacts with the chromotropic acid is compensated by the blank.

The optical density of the solution is determined at 570 millimicrons by any suitable instrument. Equally good results were obtained by the Beckman D. U. Spectrophotometer and a Coleman Junior. By reference to a standard curve (Figure 1) the amount of methanol in the sample aliquot is determined. The grams of methanol divided by the sample weight represented by the aliquot and multiplied by 100 gives the percentage of methanol in the fat.

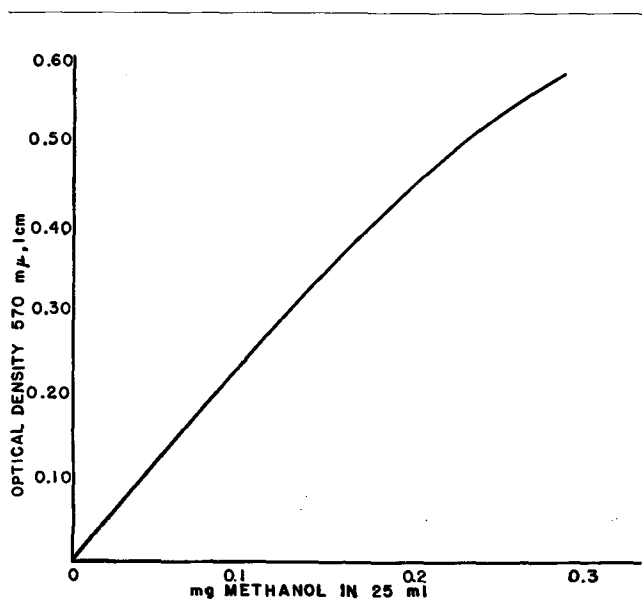


FIG. 1. Standard Curve

Aceituno Seed Fat¹

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THE aceituno tree, *Simarouba glauca* DC., which belongs to the family Simaroubaceae, grows to a height of 15 meters, is dioecious, and produces fruit 37 mm. long and 20 mm. wide, greenish yellow in one variety and violet to almost black in another. Because the fruit resembles olives in shape, size, and color, the tree is called aceituno, aceituno silvestre, or aceitillo in Central America (5, 7, 8). The fruit,

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The standard curve is determined in solutions containing aliquots of the sample distillate. Measured amounts of methanol are added to a series of flasks which contain an aliquot of the sample solution. The oxidation and reactions with chromotropic acid are carried out and the optical density of the solutions is determined by the use of the sample aliquot which contains no added methanol as a blank. In this way the effect of any unknown material in the sample distillate is internally compensated.

Table I shows some results obtained by this procedure.

TABLE I
Results of the Determination of the Methoxyl Content of Fats

Sample	Methoxyl added	Methoxyl found
	%	%
Lard.....	0	0
Lard + 0.01% methyl oleate.....	0.0011	0.0010
Lard + 0.01% methyl oleate.....	0.0011	0.0012
Lard + 0.01% methyl oleate.....	0.0011	0.0011
Rearranged lard before deodorization.....	0	0.21
Rearranged lard after deodorization.....	0	0.0006

The solvent used in the saponification step must be checked for methanol and formaldehyde content. Materials such as ethylene glycol, propylene glycol, cellosolves, and polyethylene glycol were found to give very high reagent blanks. However diethylene glycol gives a very low blank and therefore may be used in the procedure.

Also it was found that the commonly used antioxidants did not interfere with the determination.

Summary

A method for the determination of the methoxyl content of fats has been developed using chromotropic acid. The fat is saponified and the methanol recovered by distillation, oxidized, and reacted with chromotropic acid. The optical density of the colored solution at 5,700 Å is proportional to the methoxyl present.

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which contains a sweetish pulp with an astringent aftertaste, is a favorite, especially with children, and is sold in the markets.

The seed is ovoid in shape, about 20 mm. long and 12 mm. wide, deeply veined, and easily broken. The kernel is greenish in color and has an intensely bitter and persistent taste.

The seeds contain about 30% kernels and 70% shells. The kernels contain between 55% and 65% fat and 14% moisture. No difference in yield of fat has been observed between the white and "black" varieties.

The tree grows wild from Mexico to Panama, and a related species is found in the Caribbean area (2, 6).