Determination of Water in Cooked Cottonseed Meats by a Modified Indirect Conductivity Method[1]

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An INDIRECT conductivity method for the determination of water in soils was described recently (3). Later the successful application of a modification of this method to cottonseed meals was reported (2). This method involves the use of alcohol-acetone-water-sodium chloride systems to determine water in various materials; the fundamental basis of the method is that the conductivity of the system will be proportional to the sodium chloride concentration, which, in turn, will be proportional to the water concentration. The use of a large excess of sodium chloride tends to mask the effect of other electrolytes that may be present in the material being tested.

First attempts to apply this method (3) to the determination of water in cooked cottonseed meats were unsuccessful due largely to ineffective stirring. This fault was corrected by the substitution of a simple, homogenizer-type of stirring apparatus. Other modifications of the original procedure were made in order to keep the method as simple and as rapid as possible. The present article describes the modified procedure and presents encouraging results from the determination of moisture in nine samples of cooked cottonseed meats.

Experimental

Apparatus. A portable model RC-1B conductivity bridge (Industrial Instruments Inc., Cedar Grove, N. J.) was used. This bridge operates on 115-volt, 50- to 60-cycle alternating current circuits. The Wheatstone bridge is supplied with 1000-cycle alternating current from an integral vacuum tube oscillator; the balance is indicated by a cathode ray tube null indicator.

A glass, dip-type conductivity cell with cell constant of 1.020 $cm.^{-1}$, determined at 35.0°C. with 0.01 *N* potassium chloride (5), was used.

Satisfactory agitation of mixtures was obtained by use of a Model 10 Oster Mixer (John Oster Manufacturing Company, Racine, Wis.).

Materials. Absolute ethyl alcohol and acetone have been described previously (3). A 5-gal. bottleful of 70% ethyl alcohol-30% acetone mixture was equipped with a long thermometer, a siphon tube connected to a 100-ml. automatic pipet, and appropriate pinch clamps and drying tubes. The pipet was calibrated and graduated to deliver 100.00 ml. of mixed solvent (corrected to 35.0°C.) over the range of 28 to 35°C.

Common table salt (100-lb. bag, Morton Salt Company, Chicago, Ill.) was used. The approximate sieve analysis of this salt is shown in Table I.

Thirty-four samples of cooked cottonseed meats from the stacks of six screw presses of the Brazos Valley Oil Mill, Waco, Tex., were taken (4) on June 28, 1955. Duplicate determinations of water by the oven-dry method (5-g. sample dried for 2 hrs. at 101°C.) were made on these samples by others (1, 6). The averages of the closely agreeing results from the duplicate determinations were used. Nine samples with a representative range of water contents were selected for this study.

TABLE I

Approximate Sieve Analysis of Morton's Evaporated, Granulated Salt

Sieve No.	% Passing
10	100
20	99.7
40	38
60	21
80	10
100	4

Procedure. A 50.0-g. portion of Morton's salt (directly from a 100-lb. bag) plus 10.00 g. of cottonseed meats plus 100.00 ml. (corrected to 35.0°C.) of 70% ethyl alcohol-30% acetone mixture were placed in a 1-pint Mason jar and agitated for 5 min. with a Model 10 Oster Mixer. The temperature rose to about 55°C. The sealed mixture was cooled for 1 min. in a running tap water bath. The agitator assembly was replaced with a plain screw cap (the agitator assembly was cleaned and used immediately in the next determination), and the mixture was allowed to settle for 4 min. while cooling in the running tap water bath. The supernate was decanted into a 6-in. test tube (inside diameter of 28 mm.). The dip-type conductivity cell was lowered and raised several times in the extract and then clamped so that the top of the extract was about 0.5 of an inch above the ports in the cell. Four measurements of the resistance were made. The deviation among these four readings was never significant so the average was used. At the time of the resistance measurements the temperatures of the various extracts fell within the range of 29.1 to 30.7°C. The average resistance in ohms was converted to specific conductance in millimhos/cm. in the usual manner by using the cell constant of 1.020 $cm.^{-1}$

This procedure was applied in duplicate to the nine cottonseed meat samples. The averages of the closely agreeing results from the duplicate determinations were used.

Comparative tests showed that there was no advantage in rinsing glassware with distilled water or drying it in an oven; consequently glassware was washed with soap solution, rinsed with tap water, drained, and air-dried.

A 10.000-g., air-exposed sample of Morton's salt was weighed 18 times during a 29-day period, and the maximum variation in weight was found to be only 0.002 g.; consequently salt directly from a 100-lb. bag was used. This practice may not be permissible in extremely humid climates or weather; however at several of the weighing times during the 29-day period, the relative humidity was near 100% but the 10.000-g. salt sample showed no significant gain in weight.

The temperature rise that occurred on stirring with the Oster Mixer resulted in a pressure increase in the jar; consequently gloves and safety glasses were used in handling the warm jar. This precaution was fol-

[1] Describes data obtained in research conducted cooperatively by the Cotton Research Committee of Texas and the Texas Engineering Experiment Station.

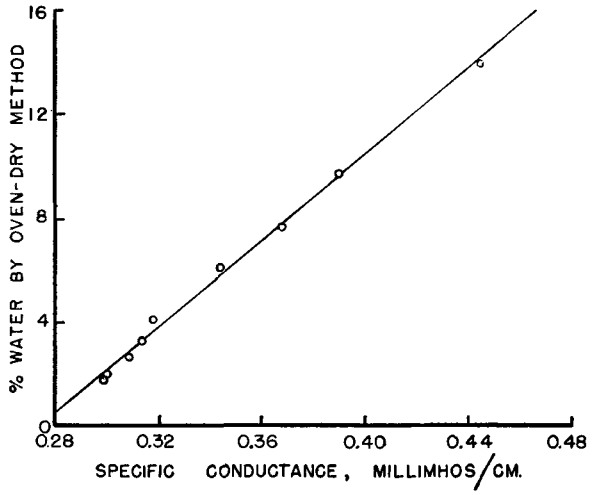

FIG. 1. Relationship between percentage of water by oven-dry method and indirect specific conductance for 9 cooked cottonseed meat samples.

lowed rigorously even though no explosions of jars ever occurred.

After this study was completed, an examination of all of the resistance readings showed, in all cases, that the average of the first two values did not deviate significantly from the average of the four readings. As a result, it appears that two readings are sufficient for a reliable average.

Results and Discussion

The indirect specific conductances and the percentages of water by the oven-dry method for the nine cottonseed meat samples are plotted in Figure 1. Statistical treatment (7) of the data represented in Figure 1 yields the following results: (1) equation of the regression line,

$$\% \text{ water} = (82.31)(\text{millimhos}) - 22.53;$$

(2) correlation coefficient, 0.998; (3) standard deviation from the regression line, 0.27% water. The regression line is shown in Figure 1.

These results show that the accuracy of the method is good, especially in view of the facts that percentages of water by the oven-dry method were used as criteria of accuracy and that no temperature corrections were applied to the indirect specific conductances. Probably the former source of error was more serious. In connection with the latter source of error, several measurements of the temperature coefficient of conductance of alcohol-acetone-water-sodium chloride systems were made, and an average value of about 0.6%/°C. was found. This value is considerably smaller than the coefficient of 2%/°C. that is generally accepted for aqueous systems so temperature control is less critical for the indirect conductivity method. In view of the small temperature coefficient and of the small temperature range of 1.6°C. involved in the resistance measurements, it was not considered worthwhile to make temperature corrections on the indirect specific conductances. Anyway the final practical answer to temperature corrections involves the incorporation of a temperature compensation feature on the Wheatstone bridge.

In order to evaluate the precision of the method, resistance determinations were made on 10 replicate samples of each of two of the cottonseed meat samples. The results and statistical data (7) resulting therefrom, given in Table II, show that the precision of the method is good.

For a consecutive series of determinations the average time required per determination was about 9 min. This time could be reduced to about 5 min. by using two Oster Mixers.

The results of the studies of accuracy and precision show that the modified indirect conductivity method offers promise as a simple, rapid laboratory control procedure for the determination of water in cooked cottonseed meats.

The intimate mixing and temperature rise resulting from agitation with an Oster Mixer are both beneficial in that both help toward transferring water from the cottonseed meat sample to the mixed solvent and subsequently saturating the alcohol-acetone-water system with salt. As shown by the results, very nearly reproducible temperatures can be obtained by cooling the warm mixture for 5 min. in a running tap water bath.

Results of studies of the original method with soils (3) indicate that simple agitation suffices to transfer all of the water from the soil to the mixed solvent. Results reported herein for the modified method with cottonseed meats indicate that only a part of the water is transferred even though the mixing is much more intimate and at a higher temperature. The reason for this difference, of course, is that the water is bound more strongly to the cottonseed meats. Even though the extraction of water from the cottonseed meats is incomplete, the degree of extraction under well-defined conditions is reproducible as proved by the results of this study. In this connection it should be noted that if the extraction procedure is varied, then the slope and intercept of the equation of the regression line will also vary.

TABLE II

Precision of Resistance Determinations at 29.3 to 29.7°C. by the Simplified Procedure

Run No.	Percentage of water by weight	
	2.7	7.7
	Resistance, ohms	
1	3292	2762
2	3300	2775
3	3318	2778
4	3312	2756
5	3299	2770
6	3305	2755
7	3300	2762
8	3309	2768
9	3299	2770
10	3318	2759
Av., ohms	3305.2	2765.5
Av. dev., ohms	7.24	6.70
Stand. dev., ohms	8.78	7.89
Stand. dev., % water	0.07	0.08

The object of a project under consideration is to calibrate and graduate a small, portable, battery-operated, 1000-cycle conductivity bridge so that it will read directly in percentage of water with bridge temperature compensation. A pipet-type conductivity cell will be used with this modified bridge.

Summary

The modified indirect conductivities and oven-dry moisture percentages were determined on nine sam-

ples of cooked cottonseed meats (1.8 to 13.9% water). Statistical treatment of the data yields the following results: (1) equation of the regression line,

$$\% \text{ water} = (82.31)(\text{millimhos}) - 22.53;$$

(2) correlation coefficient, 0.998; (3) standard deviation from the regression line, 0.27% water.

Ten replicate determinations made on each of two samples containing 2.7 and 7.7% water showed standard deviations of 0.07 and 0.08% water.

These results show that the accuracy and precision of the modified indirect conductivity method are satisfactory for practical applications. For a consecutive series of determinations the average time required per determination was about 9 min. This time could be reduced to about 5 min. by using duplicate sets of stirring apparatus.

Acknowledgment

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Solvent Cooking of Cottonseed Meats for Extraction[1]

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SINCE 1946 the cottonseed processing industry has undergone considerable technological change. The conventional hydraulic process has been replaced in many oil mills by screw pressing (3, 8), direct solvent extraction (9), or a combination of the two generally referred to as prepress solvent extraction (9, 12). One of the recently commercially developed processes, which solved many of the problems encountered in applying direct solvent extraction to cottonseed, is filtration-extraction (1, 2, 5). An important feature of this process is the cooking and crisping of the raw, rolled meats prior to extraction which results in the granulation of the meat particles and reduction of gossypol (0.03–0.05% in the final meal) while maintaining a reasonably high protein solubility. The cooking-crisping operation is conducted in the same type of conventional equipment as is used for preparing cottonseed for hydraulic pressing. This method of preparation also makes possible the rapid solution of the oil while slurrying the rolled, cooked, crisped meats in the extractor (2) and the efficient removal of the resulting miscella on a horizontal rotary vacuum filter (2).

"Solvent cooking," as described in this paper, combines the cooking, crisping, and the slurrying steps in a single operation. The raw, rolled cottonseed meats are mixed in the solvent, together with sufficient moisture (containing added chemicals), and then the resulting slurry is subjected to a combined heating and azeotropic distillation, with further mixing, to reduce the moisture content to an optimum value.

Data are presented to show the influence of various conditions and chemicals in the "solvent cooking" operation on the granulation and extraction of the oil in the prepared meats and the reduction of free gossypol and maintenance of high alkali protein solubility in the final meals.

[1] Presented at the annual fall meeting of the American Oil Chemists' Society, Philadelphia, Pa., Oct. 10–12, 1955.

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Experimental

Material and Equipment. One prime lot of cottonseed was used to obtain the data reported in this paper. The solvents used were commercial grades of hexane and heptane.

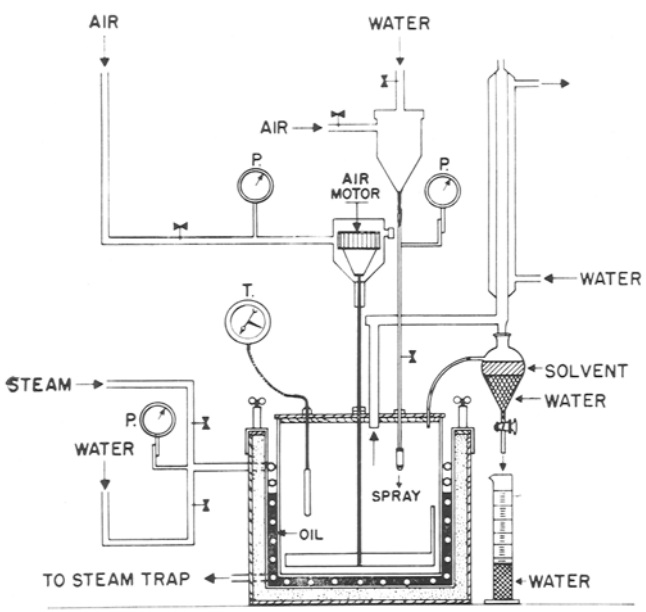

FIG. 1. Solvent cooker.

Figure 1 shows a drawing of the apparatus in which the solvent-cooking of cottonseed flakes was conducted. A coil immersed in an oil bath and surrounding the outside and bottom of the cooking container is connected to both steam and water lines for heating and