Estimation of Crude Fiber in Dehulled Soybeans

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Abstract

The determination of crude fiber in soybeans is a lengthy and time-consuming procedure which requires extraction, digestion, and incineration and is not suitable as a routine processing-control tool. A short control procedure has been developed, employing photomicroscopy, which is based on the characteristics of soybean hull cellulose to rotate plane-polarized light. The sample is mounted on a microscope slide, treated with trichloro-acetic acid, and placed in a polarizing projection microscope; the image is compared with a series of standard photomicrographs. The amount of hulls present in the sample is measured quantitatively, and an estimation is made on the crude fiber. The crude fiber estimation on dehulled soybeans can be made in 15 min compared with 8 to 12 hr by using the official crude fiber method.

Introduction

In 1957 a Liaison Committee of AOCS and AOAC members was organized to study the crude fiber method as published by these two organizations in their Official Methods (1,2). The primary assignment of the committee was to improve the precision of the method and secondarily to reduce the operating and elapsed times of the procedure. After four years of extensive laboratory investigation and collaborative studies the committee did succeed in making some improvements in the method precision but failed to make any significant changes in the procedure that would reduce the elapsed time required to make a crude fiber determination (3). The basic technique of acid digestion, followed by alkali digestion, produces an empirical crude fiber result that cannot be reproduced by using any other technique. To give continuity to the crude fiber data that have been published and will be published in the future, the committee was forced to concede that the method, although highly unsatisfactory, must be retained as the Official Procedure.

This (AOCS Method Ba 6–61) is not adaptable to in-process control analysis where immediate results are required, particularly in the soybean dehulling operation where essentially complete removal of the hull is necessary to meet the NSPA Specification of 3.0% maximum crude fiber in 50% soybean meal (4). A project was initiated in the ADM Control Laboratories to devise a procedure that would provide production personnel with a rapid estimation of the hull fraction remaining in the meal. The microscope was chosen as the most logical approach to the problem, and micro-samples of ground whole beans were examined under varying conditions. It was discovered that the cellulose portion of the soybean hull rotates plane-polarized light when the sample is mounted in trichloro-acetic acid. The theory was advanced that a quantitative method for measuring the hull fraction could be developed by establishing a series of standard samples against which an unknown sample can be compared.

Experimental Section

To prepare the standard samples, a portion of soybeans was completely dehulled by hand-picking. The dehulled beans and the hulls were ground separately in a Mikro-Pulverizer by using a .027 herringbone screen. A screen analysis was then performed on both portions, and the hull portion was reconstituted to give a screen analysis equal to that of the dehulled portion.

A crude fiber analysis was made on the two portions, and standards were prepared by weighing and combining the two fractions to form equivalent crude fiber percentages in increments of 0.1% through a 1.0% increase in crude fiber. The crude fiber values of each of the standards were confirmed by analysis by using the official crude fiber method.

The amount of sample to be used for microscopic analysis was determined by the portion that can be conveniently mounted in a concave microscope slide and fill the viewing area in a microscope at 10 magnifications. This requires approximately 0.05 g of sample. Since the primary interest was in developing a rapid method of analysis, it was decided that the sample size should be measured by volume rather than weight. A volumetric sampler was devised by drilling a $^{5}/_{32}$–in. diameter, hemispherical depression in a flattened metal rod. This depression will hold approximately 0.05 g of lightly packed soy flour.

Trichloro-acetic preparations were made of each of the standards, and colored photomicrographs were taken of each percentage increment, including the completely dehulled portion. These were mounted for ready comparison with unknown production samples. It was found that 11 standards were too unwieldy for routine comparative analysis, and, since the precision of the official crude fiber method is approximately 0.2% between ADM laboratories on low fiber soybean meal, it was considered expedient to arrange the standard series in increments greater than 0.1%. A series of six photomicrographs was selected, as shown in Table I. These six comparative standards provide for a quick determination and easy interpolation to 0.1%.

After much experimentation with methods of viewing and comparison with standards, the projection of the unknown and the photomicrograph standards side by side on a screen was found to provide the easiest and the most accurate means of making comparisons. To accomplish this a projection microscope was used to project the unknown sample, and a film enlarger, with the photomicrographs mounted in a film strip, was used to project the comparison standards. The two projectors were mounted in a darkroom box so that both images could be projected on a horizontal screen mounted between the enlarger and microscope at desk level. With the images side by side on the screen, comparisons are made easily and accurately.

TABLE I
Crude Fiber Results of Comparison Standards

Std. No.	Equivalent % Crude fiber
B	2.6
I	2.7
III	2.9
V	3.2
VII	3.5
IX	3.8

To run an unknown sample of dehulled beans or 50% flakes, the sample is ground in a Mikro-Pulverizer by using a .027 herringbone screen. The sample rod is dipped into the ground sample, and the excess is struck off with a spatula in such a manner that the sample in the depression is only lightly packed. Two drops of isopropyl alcohol, followed by two drops of a saturated solution of trichloro-acetic acid, are added, and the soybean meal is gently dispersed with a slender glass rod. The slide is then placed in the projection microscope, and comparison is made against the comparison standards projected by the enlarger. Three separate slides are prepared on each unknown sample, and an average of the three values is reported as the estimated crude fiber result.

Results and Discussion

This equipment has been set up at each of the ADM soybean plants and is being used routinely by the operators to ascertain the efficiency of the dehulling operation. Table II shows a series of results obtained by an ADM plant control laboratory by using the microscopic and Official crude fiber methods. Table III shows the results of a series of check samples sent at intervals to six laboratories which are equipped to run microscopic analysis for soybean hulls. The first analysis shown for each laboratory was run by the laboratory on receipt of the sample; the second analysis was run on the same samples approximately one month later. Crude fiber determinations on these samples, using the AOCS Official Method Ba 6–61, are shown in Table IV.

It should be emphasized that this method determines quantitatively only the hulls remaining in the dehulled soybeans and that the crude fiber result is only an estimation. For this estimation, the amount of crude fiber inherent in the endosperm was set at 2.6%. This is an approximate rather than an exact figure, and samples have been analyzed with an endosperm crude fiber of 2.4% and less. When such samples are encountered as unknowns, the estimated fiber by the microscopic method will give higher results than results obtained by the Official Method.

TABLE II

Comparison of Microscopic Crude Fiber with Official Fiber Method

Sample	Microscopic	Official
50% Prod.	2.6	2.5
50% Prod.	2.6	2.4
48½ % Prod.	3.6	3.5
50% Flakes	3.8	3.95
48½ % Prod.	2.4	2.55
48½ % Prod.	3.5	3.3
50% Prod.	3.0	2.85
48½ % Ship.	3.35	3.3
50% Prod.	2.6	2.5
50% Ship.	2.7	2.6

TABLE III

Check Sample Analysis by Microscopic Crude Fiber Sample

Lab.	Anal. No.	1–20	1–27	2–3	2–8	2–10
A	1	3.2	3.1	2.9	2.7	3.5
B	1	3.35	3.2	3.0	2.7	3.5
	2	3.2	3.2	2.9	2.7	3.2
C	1	3.6	3.2	2.9	2.5	3.5
	2	3.4	3.2	3.0	2.5	3.45
D	1	3.4	3.3	3.2	2.9	3.6
	2	3.2	3.1	3 0	2.7	3.4
E	1	3.2	3.1	2.9	3.0	3.4
	2	3.3	3.1	3.1	2.9	3.3
F	1	3.3	3.4	3.1	2.8	3.6
	2	3.4	3.2	3.0	2.9	3.3
Average		3.32	3.19	3.0	2.75	3.43

TABLE IV

Soybean Meal Check Sample by Official Crude Fiber Method

Lab.	Anal. No.	1–20	1–27	2–3	2–8	2–10
A	1	3.39	2.85	2.79	2.33	3.32
	2				2.53	
B	1	3.45	3.30	3.00	2.61	3.40
	2	3.30	3.31	3.00	2.60	3.40
C	1	3.45	2.90	2.75	2.40	3.40
	2	3.55	2.87	2.90	2.50	3.60
Average		3.43	3.04	2.89	2.50	3.42

REFERENCES

1. AOCS Official Method Ba 6–61.
2. AOAC Official Method, 10th ed., 22.038.
3. Holt, K. E., J. Am. Official Chemists *45*, 578–584 (1962).
4. NSPA Trading Rules, 1966–67, Rule 2, Sec. 3C.

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