NOTE

A Modification of the Crude Fiber Test for Application to Flour

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In recent years, the crude-fiber test has been criticized for not truly representing the nondigestible fraction of foods, feeds, and other raw materials. Acid and neutral detergent fiber, cellulose, and lignin values provide more meaningful information about the digestibility of the material. Nevertheless, the crude-fiber test remains the industry's only official method for the reporting of nondigestible carbohydrates. Feed companies are obliged to meet legal specifications regarding the crude fiber content of their merchandise. Wheat flour is not routinely tested for fibrous components, because the level of these in white flour is too low to significantly affect the digestibility of breads or other products baked from it. Occasionally, investigational purposes require that the fiber content of flour be determined. A recent series of investigations was completed at the Canadian Grain Research Laboratory with the objective of identifying wavelengths for developing calibrations for the determination of ash in flours by near-infrared reflectance spectroscopy. Both the crude fiber and the oil contents were regressed against flour ash to determine the degree of correlations between these characteristics, all of which increase in flour as a result of bran contamination. During these investigations, a modification to the test procedure was developed that significantly improved the efficiency of the crude fiber test in its application to flour.

The testing of wheat flour for crude fiber by the standard wet chemical method (AACC 1976) involves preliminary extraction of most of the lipids, acid digestion of most of the carbohydrate, followed by an alkaline digestion, which removes the protein, any remaining oleaginous material, and some additional carbohydrate. Testing is complicated by the fact that, after acid digestion, a large amount of colloidal material remains that inhibits the filtration necessary before passing to the alkaline digestion step. Filtrations of the acidic suspension can be prolonged to several hours, which affects the accuracy of the method. Filtration can be improved by reducing the sample size to 1 g or less, but the amount of fiber in flour is so small that this practice increases sampling error. A simple modification is described that simplifies the test and improves overall precision. The acid suspension is neutralized with a small amount of very strong alkali, which is added in an amount sufficient to provide the correct alkalinity for the second digestion while not appreciably altering the volume. Earlier work (Hullab and Epps 1963) indicated that small deviations from the prescribed concentrations and volumes of the two digesting solutions exert no significant effect upon the results of crude fiber analyses.

MATERIALS AND METHODS

We weighed 2–3 g of flour into a Berzelius beaker 800 ml tall and added 2–3 boiling chips (or glass beads) and 200 ml of 0.2549*N* sulfuric acid. After it reached the boiling point, the mixture was boiled under reflux on the crude fiber boiling apparatus; the usual precautions were taken to prevent material from creeping up the sides of the beaker. The mixture was removed from the heat, and 10 ml of sodium hydroxide solution that contained 407.42 g/L was added. The mixture was then boiled on the apparatus for an additional 30 min. Then the solution was filtered through a tared, sintered silica crucible (or a silica gooch with a fiberglass filter), washed free of alkali with boiling water, and the residue transferred to the filter crucible. The crucible was dried for ~1 hr at 100–130°C, cooled in a desiccator, and placed overnight in a muffle furnace at 600°C. The residue was cooled and reweighed. The difference in weight was equivalent to the weight of the crude fiber.

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TABLE I
Precision of Analysis of Three Materials for Crude Fiber by Standard and Modified Methods

Material	Modified Method Mean (%)	Modified Method Standard Deviation (%)	Standard Method Mean (%)	Standard Method Standard Deviation (%)
Straight run flour	0.23	0.04	...	...
Whole wheat meal	2.79	0.14	2.59	0.08
Barley straw	35.44	0.56	36.01	0.92

RESULTS AND DISCUSSION

Accuracy was evaluated by "spiking" flours with finely divided bran powder (sifted through a 200-mesh stainless steel screen). Bran additions were made to three different flours, a very pure first middlings flour, a straight run flour, and a first break flour. Fiber was determined in the original flours by the modified method and in the bran powder by the standard method. Increments of bran powder were added to the subsamples of the flours, and the flours were mixed well and analyzed for fiber by the modified method. The results were compared to the theoretical (calculated) fiber contents. The mean difference between calculated and observed fiber contents was +0.010% with a standard deviation of ±0.024%. The coefficient of correlation was 0.992.

Precision was assessed by analysis of a straight run flour, whole wheat meal, and barley straw, all of which were ground on a Cyclotec grinder. Results are summarized in Table I. In addition to the satisfactory accuracy and precision obtained by the modified method, the analytical procedure was shortened, which made its execution more convenient. Filtrating the oil extract and working the acid free normally occupy about 30 min for a series of six samples, including transfer of the fibrous residue to the original beaker for alkaline extraction. For white flours, this time may be increased to 2–3 hr because of the very slow filtration. The time between acid and alkali extraction is consistently shortened to 5 min by the neutralization step.

LITERATURE CITED

AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1976. Approved Methods of the AACC (8th ed.). Method 32-10, approved October 1981. The Association, St. Paul, MN.

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