The Determination of Fiber in Processed Cereal Foods by Near-Infrared Reflectance Spectroscopy[1]

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ABSTRACT

The use of near-infrared reflectance spectroscopy for determining fiber in foods was evaluated by predicting the neutral detergent fiber of ready-to-eat breakfast cereals. Forty cereal samples were used to calibrate a commercially available scanning instrument and to develop the prediction equation. The correlation was 0.977, and the standard error of prediction was 1.5% fiber. The prediction equation was used to predict the fiber in 50 cereal samples. The correlation was 0.955, and the standard error was 1.8% fiber. Crystalline sugar was an interference in some samples. However, the near-infrared reflectance technique shows promise as a rapid method for determining the fiber content of processed cereal foods.

Conventional analytical methods for determining fiber in foods are stepwise chemical or enzymatic procedures that require several hours. Analyses by near-infrared reflectance (NIR) spectroscopy are very rapid procedures that are desirable when large numbers of samples must be analyzed. The NIR procedures were developed for determining moisture, protein, and oil in various raw grains and oilseeds (Norris 1978, Norris and Hart 1965). A high correlation between NIR measurements and neutral detergent fiber (NDF) values was found in feeds and forages (Norris et al 1976).

In a pilot study, our laboratory determined the fiber contents of ready-to-eat breakfast cereals by the NDF method (Baker and Holden 1981). We believed that the NIR technique could be used to analyze processed foods and decided to evaluate the procedure by predicting the NDF of the dry breakfast cereals. The NIR technique was first evaluated in collaboration with K. H. Norris, Instrumental Research Laboratory, Agricultural Research, USDA. We supplied the samples of cereals with the chemical data, and he recorded the spectra on the computerized scanning spectrophotometer that he had built in his laboratory. The correlation coefficient for NDF values vs NIR-predicted values was 0.985, with a standard error of 1.36 (Baker et al 1979). At that time, we also investigated the use of a commercially available NIR filter instrument fitted with three filters optimized for fiber. However, we were unable to predict the fiber in cereals with high fat and sugar contents.

This article is a report of work with a commercially available computerized scanning instrument that is similar in principle to the research instrument built by Norris (Norris et al 1976).

MATERIALS AND METHODS

The fiber contents of the ready-to-eat breakfast cereals (Baker and Holden 1981) were determined by the NDF method as described in AACC method 32-20 (1969). The NDF value is expressed as the percent fiber in the dry sample and is the average of two determinations. The NIR spectra were recorded with a commercially built computerized scanning spectrophotometer, the Neotec 6350. The instrument and the samples were kept in a room where the temperature was controlled to maintain 22 ± 1°C to minimize effects of temperature changes on both the samples and the instrument (Norris 1978). The samples had been ground with a Wiley mill equipped with a 20-mesh screen, except for the granola-type cereals and raisin brans that would not pass through the Wiley mill screen. These were ground with a coffee mill to produce particles that were as uniform as possible. In the reflectance mode of the instrument, the ground sample (10–12 g) is packed in a cell under a quartz window and illuminated with monochromatic light. As the sample cup spins, the reflected radiation from the sample is detected by four lead sulfide cells spaced around the incident beam. The monochromator scans the sample, and the computer records the spectrum as the log of 1/R curve from 1,100 to 2,500 nm, where R equals reflectance. Data from the spectral curve were collected at 2-nm intervals. Three spectral curves for each sample (once packed in the sample cup) were averaged by the computer before recording the data.

The spectra from 40 calibration samples were stored on a floppy disk. These samples were chosen to cover the range of NDF values expected in the sample population and to represent the different types of cereals. The spectra were then analyzed by a computer regression program provided by the instrument manufacturer. This program is a stepwise multiple linear regression between the analytical value and a mathematical treatment of the reflectance data. The regression analysis determined the optimum wavelengths, ie, those giving the highest correlations to NDF and the lowest standard error. Those wavelengths were used in an equation to predict the fiber values of 50 samples.

RESULTS AND DISCUSSION

Data for the calibration samples are shown in Table I. The correlation of the NIR-predicted value with the NDF value was 0.977, and the standard error was 1.5% fiber. The regession analysis chose four wavelengths for a two-term, first-derivative prediction equation:

$$NDF = -59.961 - 22.880 \frac{d\,(\log 1/R_{2,450\ nm})}{d\,(\log 1/R_{2,204\ nm})} + 22.192 \frac{d\,(\log 1/R_{1,352\ nm})}{d\,(\log 1/R_{2,448\ nm})}$$

The use of the d log $1/R_1$ divided by d log $1/R_2$ is based on the recommendation of Norris and Williams (1977) to compensate for both particle-size effects and moisture interferences.

Data for the prediction samples are shown in Table II. The correlation was 0.955, and the standard error of prediction was 1.8% fiber. The bias of −0.3 means that for most samples the NIR-predicted value was generally lower than the chemical NDF value. Negative values in the NIR prediction can occur because of the standard error of prediction when NDF values are near zero. A few negative values are seen in Tables I and II. In the practical application of the NIR prediction, these values would be considered zero.

In Figs. 1 and 2 are examples of the spectral curves typical of different cereal samples. Since the cereals are complex mixtures,

[1]The research conducted by the USDA on the commercial samples as reported was limited to analyses of their fiber composition. The data are reported solely as factual information and are limited to the samples analyzed. No warranty or guarantee is made or implied that other samples of these products would have the same or similar composition. It is the policy of the USDA not to endorse those commercial products tested in research over those that were not tested. Use of company or product names by the USDA does not imply approval or recommendation of the product to the exclusion of others that may also be suitable.

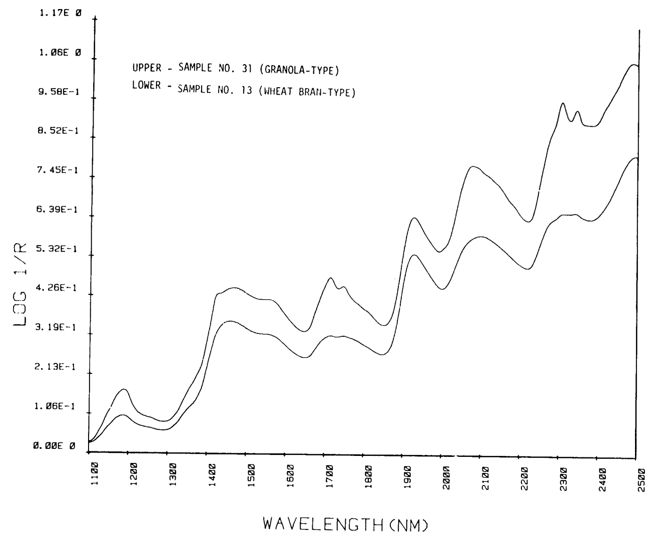

Fig. 1. The spectrum of sample 31 (Table I) is typical of granola-type cereals with prominate fat bands at 2,346, 2,308, 1,758, and 1,726 nm and evidence of free sugar at 1,440 and 2,086 nm. The spectrum of sample 13 (Table I) is typical of wheat bran-type cereals low in fat and free sugar contents.

TABLE I
Near-Infrared Reflectance (NIR) vs Neutral Detergent Fiber (NDF), Calibration Samples[a]

Sample No.	Cereal Type	NDF	NIR Prediction[b]	Difference
1	Wheat	9.2 ± 0.18	9.1	−0.1
2	Oat	3.2 ± 0.12	3.1	−0.1
3	Wheat	7.0 ± 0.08	7.2	+0.2
4	Oat-wheat	2.8 ± 0.07	3.7	+0.9
5	Granola	2.4 ± 0.07	2.1	−0.3
6	Oat-corn	1.4 ± 0.02	3.0	+1.6
7	Wheat-barley	5.4 ± 0.04	6.9	+1.5
8	Wheat-barley	6.9 ± 0.01	5.9	−1.0
9	Wheat	14.4 ± 0.03	13.2	−1.2
10	Corn	2.1 ± 0.02	−1.4	−3.5
11	Corn	1.6 ± 0.08	−1.3	−2.9
12	Wheat	1.3 ± 0.05	2.2	+0.9
13	Wheat bran	28.3 ± 0.20	27.9	−0.4
14	Wheat bran	31.5 ± 0.09	29.8	−1.7
15	Wheat	11.8 ± 0.06	13.6	+1.8
16	Corn-rice-wheat-oat	0.6 ± 0.16	0.2	−0.4
17	Corn	0.6 ± 0.10	0.9	+0.3
18	Rice	0	1.5	+1.5
19	Corn-oat	0.8 ± 0.08	0.5	−0.3
20	Oat	2.2 ± 0.06	2.4	+0.2
21	Corn	0.6 ± 0.18	−0.8	−1.4
22	Corn	0.6 ± 0.03	0.1	−0.5
23	Oat-corn	1.6 ± 0.03	2.1	+0.5
24	Oat-corn	1.6 ± 0.05	2.2	+0.6
25	Rice	0.2 ± 0.02	0.5	+0.3
26	Wheat	6.4 ± 0.05	7.6	+1.2
27	Wheat	7.6 ± 0.11	6.7	−0.9
28	Rice	0.1 ± 0.02	2.5	+2.4
29	Wheat bran-oat	13.2 ± 0.06	10.7	−2.5
30	Granola	3.9 ± 0.02	3.7	−0.2
31	Granola	3.7 ± 0.01	3.7	0
32	Granola	3.6 ± 0.07	4.6	+1.0
33	Granola	4.0 ± 0.13	4.6	+0.6
34	Granola	3.8 ± 0.27	5.3	+1.5
35	Granola	4.4 ± 0.27	5.1	+0.7
36	Corn	1.4 ± 0.04	−2.0	−3.4
37	Granola	6.9 ± 0.11	6.4	−0.5
38	Granola	3.6 ± 0.12	6.2	+2.6
39	Granola	2.9 ± 0.04	4.5	+1.6
40	Granola	5.4 ± 0.09	4.7	−0.7

[a] NIR prediction vs NDF: Correlation = 0.997.
[b] Near-infrared reflectance standard error of prediction = 1.5.

each peak is a composite or summation of several peaks representing fats, proteins, carbohydrates, and water. It is difficult to see differences among the spectral curves that can be directly attributed to the major components of the cereal fiber, ie, cellulose and hemicellulose. The small differences are detected and utilized by the mathematical treatment of the data as described by Norris (1978). In Fig. 1, the upper curve for sample 31 (Table I) is typical of granola-type cereals. The lower curve for sample 13 (Table I) is typical of wheat bran-type cereals. Sample 31 shows prominent fat bands at 2,346, 2,308, 1,758, 1,726 and evidence of crystalline sugar at 1,440 and 2,086. Both free fat and crystalline sugar are potential interferences to the determination of NDF. Sample 13 illustrates the spectrum typical of a cereal sample low in both fat and sugar. In Fig. 2, the upper curve for sample 1 (Table I) is typical of wheat cereals that utilize the entire kernel. Corn-type cereals and rice-type cereals, low in free fat and free sugar, have similar curves. The lower curve is typical of cereals with significant amounts of free

TABLE II
Near-Infrared Reflectance (NIR) vs Neutral Detergent Fiber (NDF), Prediction Samples[a]

Sample No.	Cereal Type	NDF	NIR Prediction[b]	Difference
1	Rice-wheat	1.1 ± 0.05	0.5	−0.6
2	Rice	0	1.8	+1.8
3	Granola	2.2 ± 0.13	2.9	+0.7
4	Rice	0.2 ± 0	0.7	+0.5
5	Rice	0	1.4	+1.4
6	Oat	4.1 ± 0.19	7.4	+3.3
7	Corn-oat	0.6 ± 0.01	0.8	+0.2
8	Wheat	6.7 ± 0.08	7.1	+0.4
9	Corn-oat	1.4 ± 0	2.8	+1.4
10	Wheat	7.1 ± 0.12	6.2	−0.9
11	Corn-wheat	1.8 ± 0.02	1.9	+0.1
12	Rice	0	6.7	+6.7
13	Rice-wheat-corn-oat	0.9 ± 0.08	1.0	+0.1
14	Wheat bran	29.3 ± 0.46	27.1	−2.2
15	Corn-oat	1.3 ± 0.16	1.0	−0.3
16	Corn	1.2 ± 0.03	1.6	+0.4
17	Rice-wheat	0.5 ± 0.08	0.6	+0.1
18	Corn	0.4 ± 0.11	0.1	−0.3
19	Wheat	1.1 ± 0.01	0.7	−0.4
20	Rice	0	1.9	+1.9
21	Corn-rice-wheat-oat	0.5 ± 0.16	1.4	+0.9
22	Wheat	9.7 ± 0.17	11.0	+1.3
23	Corn	1.0 ± 0.26	1.2	+0.2
24	Granola	4.6 ± 0.21	5.4	+0.8
25	Corn	1.2 ± 0.05	0.1	−1.1
26	Corn-oat	1.4 ± 0.01	1.8	+0.4
27	Wheat-buckwheat	4.9 ± 0.24	3.8	−1.1
28	Corn	0.2 ± 0.03	1.0	+0.8
29	Wheat	9.2 ± 0.24	8.5	−0.7
30	Wheat	3.7 ± 0.08	6.3	+2.6
31	Corn	1.1 ± 0.12	−0.3	−1.4
32	Corn-oat-wheat	1.0 ± 0.03	0	−1.0
33	Corn-wheat-oat	0.5 ± 0.20	1.8	+1.3
34	Corn-wheat-oat	0.7 ± 0.10	2.5	+1.8
35	Corn-rice-wheat-oat	0.5 ± 0.09	1.3	+0.8
36	Corn-oat	1.2 ± 0.10	1.1	−0.1
37	Oat	3.8 ± 0.01	5.0	+1.2
38	Wheat	7.0 ± 0.14	5.1	−1.9
39	Corn	0.3 ± 0.02	−0.5	−0.8
40	Granola	4.8 ± 0.03	6.0	+1.2
41	Granola	3.9 ± 0.13	4.8	+0.9
42	Granola	4.7 ± 0.31	5.5	+0.8
43	Corn	1.0 ± 0.02	2.2	+1.2
44	Wheat	11.4 ± 0.19	11.6	+0.2
45	Wheat bran	16.0 ± 0.34	12.0	−4.0
46	Wheat bran	26.1 ± 0.09	22.8	−3.3
47	Wheat	9.3 ± 0.07	6.3	−3.0
48	Wheat	5.0 ± 0.05	2.1	−2.9
49	Rice	0.3 ± 0.03	4.3	+4.0
50	Corn	1.1 ± 0.01	0.4	−0.7

[a] NIR prediction vs NDF: Correlation = 0.955.
[b] Near-infrared reflectance standard error of prediction = 1.8. Bias = −0.3.

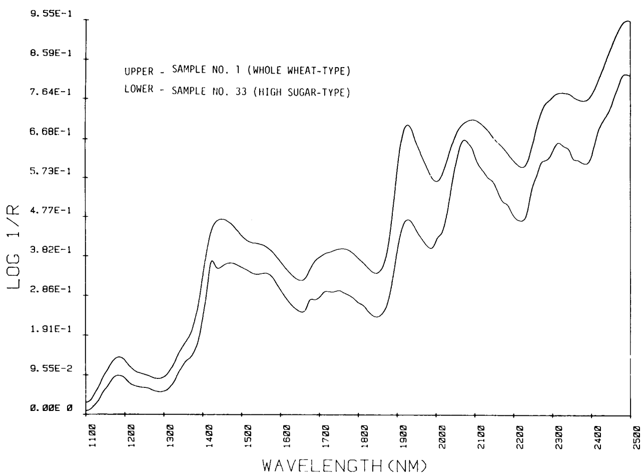

Fig. 2. The spectrum of sample 1 (Table I) is typical of whole wheat-type cereals low in fat and free sugar contents. The spectrum of sample 33 (Table II) is typical of cereals with a high content of free crystalline sucrose, and illustrates the interference of free sugar.

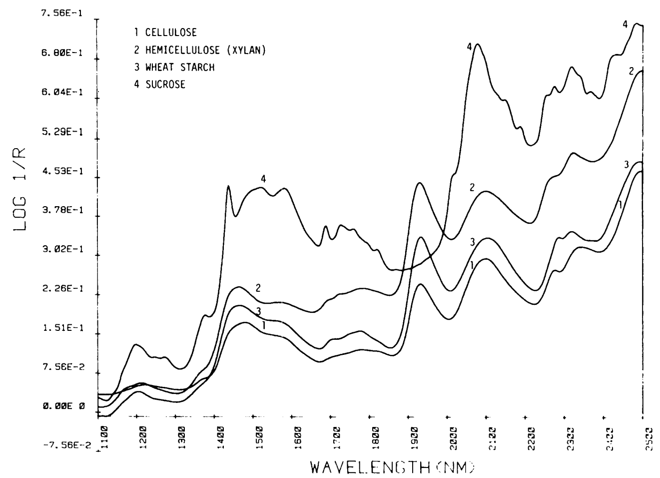

Fig. 3. Spectra of cereal components illustrating the similarity of the curves of cellulose, hemicellulose, and starch, and the interference of sucrose in the crystalline form.

crystalline sucrose. Sample 33 (Table II) contains 48–49% sucrose as determined by Li and Schuhmann (1980). In Fig. 3 are the spectra of cellulose and hemicellulose, the two major components of fiber found in cereals. The spectrum of wheat starch is shown to illustrate the spectral similarities of the polysaccharides of wheat starch, cellulose, and hemicellulose. This emphasizes the difficulty of identifying the bands where cellulose and hemicellulose can be measured. Small differences seen in the 2,250–2,400-nm region may make it possible to determine starch in future work. The spectrum of crystalline sucrose illustrates the interference of sugar. Some of the bands in the sucrose spectrum correspond to those in the spectrum of sample 33, eg, 1,440, 1,688, 1,732, 2,080, 2,188, 2,274, 2,318, 2,428, and 2,484 nm. The wavelengths in the prediction equation are in the areas where interferences of fat and sugar are minimal. The wavelengths at 2,450 and 2,448 nm avoid sugar interferences at 2,480 and 2,428 nm. At 2,204 nm part of the carbohydrate peak is used but avoids sugar interference at 2,188 nm. At 1,352 nm the sugar interference at 1,440 is avoided. The regression program did not choose wavelengths in the 2,250–2,400-nm region, probably because of interferences from both fat and sugar.

The NIR technique shows promise for determining fiber in foods. The remaining problems are not of such magnitude that they cannot be solved by further research and perhaps changes in instrument design. The NIR procedure requires only minutes and has the potential for automation. Both of those characteristics make it attractive for analyzing large numbers of samples.

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