Dietary Fiber Analysis

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Insufficient intake of dietary fiber (DF) has been associated with the incidence of various Western diseases over the past two decades (1,2). With increased awareness of physiological benefits of dietary fiber, there is a need to provide the public with the dietary fiber content of foods they eat and lists of dietary fiber sources to help them adopt dietary guidelines. In order to make tables of dietary fiber content, various food samples should be analyzed by using appropriate analytical methodology.

The following considerations should be taken into account when selecting an analytical method:

(1) Definition of analyte: A chemist should be able to provide analytical values that are consistent with the definition of the analyte we are working with. Dietary fiber is generally defined as the remnants of plant components resistant to hydrolysis by the human alimentary enzymes (2,3) which includes non-starch polysaccharides (NSP), resistant starch (RS) and lignin. The method we are working with has been based on this definition.

(2) Advantages and disadvantages of several analytical approaches. Currently, DF methods are generally categorized into enzymatic-gravimetric and enzymatic-chemical methods, including colorimetric, GLC and HPLC. Enzymatic-gravimetric and enzymatic-colorimetric methods are advantageous since these methods are simple, inexpensive, fast, and robust enough for routine analysis. They do not require high capital investment or highly trained personnel. However, they do not provide a detailed profile of DF components. Colorimetric methods in conjunction with additional fractionation techniques may provide information on major DF components, such as hexoses, pentoses and uronic acid constituents. Colorimetric methods in general require a reference method for reliable data interpretation, due to their nonspecific color reactions of reducing sugars with chromogens. The GLC or HPLC type methods offer potential advantages of characterizing neutral sugars present in DF. However, the reproducibility of neutral sugar values measured by these methods have not been proven through collaborative studies. These methods are time consuming and expensive. They require high capital investments and highly trained personnel.

(3) The methods applications: A chemist should define how they are going to utilize the data. For example, is the data going to be used for nutrition

labeling, nutrient database, construction, or quality control purposes; or will this data be used to identify active components in nutrition research, et cetera. Based on the factors listed above enzymatic-gravimetric methods are generally accepted as the best choice for Q.C. and nutrition labeling purposes. For nutrition research, the choice of methods should be left open. Currently there is no ideal method for nutrition research purposes. This topic has been recently reviewed by Asp and his colleagues (4), and Lee and Prosky (5).

This current review will focus on official methods approved by the AOAC for labeling and quality control purposes. Table 1 lists the currently approved AOAC methods (6-13). The total dietary fiber (TDF) method, 985.29, was adopted as official first action in 1985, and received official final action in 1986, with a method change in 1988(6-8). The scope of the method has been expanded to give separate values for soluble and insoluble dietary fiber (SDF/IDF). The insoluble dietary fiber method received official first action in 1991 (9-10). Currently, the SDF method by direct measurement is being reviewed by the AOAC (11).

In 1990, Lee proposed modifications to the method 985.29, which received AOAC official first action (The method 991.43) on total, soluble, and insoluble DF determinations in 1991 (12-13). This modification has been developed to improve job efficiency and to reduce analytical costs. The potential of this modification to further improve the precision of the method was also investigated. All methods are based on the same principles.

The flow diagram of the AOAC methods 985.29, 991.42 and 991.43 is as follows:

Duplicate samples of dried foods, fat extracted if containing >10% fat, undergo sequential enzymatic digestion by heat stable a-amylase, protease, and amyloglucosidase to remove starch and protein. For TDF determination, the enzyme digestates are treated with alcohol to precipitate SDF before filtering. TDF residues are washed with alcohol and acetone, then dried and weighed. For IDF determination, the enzyme digestates are filtered, residues remaining in crucible are washed with warm water, dried and weighed. For SDF determination, combined filtrate and water washings are precipitated with 4 volumes of 95% ethanol. All the dried DF residues are corrected for protein, ash and blank for final calculation of SDF and IDF values.

Table 2 lists the method performance of the 1988 version of the Prosky TDF method. It showed good precision across the products when the DF level is above 3%. In the study of 1991-1992 (9,10), the IDF method showed excellent precision (Table 3). The method 991.43 used similar principles of the AOAC methods 985.29 and 991.42 with minor modifications. These modifications include:

(1) Use of MES-TRIS buffer instead of phosphate buffer.

(2) We also eliminated 1 pH adjustment step for protease action. As with all organic buffers, the pH of the MES-TRIS buffer changes with temperature. If we start with pH 8.2 at 24 o C, it reaches pH 7 at 85-90 o C and pH 7.5 at 55-60 o C. By using this buffer system, the heat stable a-amylase is incubated near its optimum pH 7 at ~90 o C. It is no longer necessary to adjust the pH for protease digestion at 60 o C.

(3) We use a smaller volume of buffer and acid which has resulted in reduction of total filtration volume by 20%.

Table 4 summarizes results of the preliminary methods comparison study. These chemists had extensive experience with the unmodified procedure, but no previous experience with the modification. As shown in Table 4, the two methods generated similar mean TDF values. Precision of the unmodified method 985.29, was found to be satisfactory. The modification had a tendency to show even better precision for many products. Overall, chemists in both laboratories agreed that the modification was more convenient.

Based on this preliminary study, the main collaborative study has been conducted. As shown in Table 5, the measured TDF values were in excellent agreement with the TDF by summing SDF plus IDF values. Both measured and calculated TDF values reflected excellent precision. Both standard deviations (SD) and relative standard deviations (RSD) were low on all the products tested. For both soluble and insoluble DF determinations (Table 6), the method performance was also found to be excellent. As with TDF, both SD and RSD are low for this SDF analysis method. This indicates that it is not necessary to estimate SDF content by the difference between total and insoluble DF.

To summarize the results of the studies (6-13) on method performance evaluation:

(1) The overall precision of the SDF, IDF and TDF determinations is excellent. (Methods 985.29, 991.42 & 991.43.)

(2) The TDF values calculated by summing SDF and IDF were in agreement with the TDF measured directly.

(3) The modification (991.43) does not alter mean DF values when compared to the unmodified methods (985.29 and 991.42).

(4) These methods for SDF, IDF and TDF determinations can be successfully used to generate reliable values for Q.C., research, labeling and database construction purposes.

Table 1. The Status of AOAC Official Dietary Fiber Methods

Methods		References	Approval Status
TDF	985.29	Prosky et al; Ref. 6-8	1985, First Action 1986, Final Action 1988, Method Change
IDF	991.42	Prosky et al; Ref. 9-10	1991, First Action
SDF	992.--	Prosky et al; Ref. 11	The method is being reviewed.
TDF/SDF/IDF	991.43	Lee et al; Ref. 12-13	1991, First Action

Table 2. Precision Measures for AOAC TDF Method 985.29*a

	$\bar{x}$, % TDF	SR	RSDR, %
Rice	1.0	0.95	95.0
White Wheat Flour	3.1	0.42	13.6
Soy Isolate	1.6	1.38	88.0
Rye Bread	6.6	0.81	12.2
Potatoes	6.8	0.94	13.9
Oats	11.3	1.62	14.3
Wheat Bran	43.9	1.18	2.7
Corn Bran	87.1	1.41	1.6

*a. Dry Weight Basis; Prosky, L. et al. (1988) JAOAC 71, 1017-1023.

Table 3. Performance of AOAC IDF Method 991.42*a

	X, % IDF	SR	RSDR,%
Apples	55.57	2.53	4.55
Apricots	44.92	3.69	8.22
Figs, Calimyrna	43.07	7.92	18.40
Figs, Mission	33.61	4.06	12.09
Peaches	39.53	2.44	6.16
Prunes	46.18	8.98	19.44
Raisins	49.18	9.49	19.30
Barley	4.30	0.62	14.33
Rye Flour	11.81	1.02	8.62
Soy Bran	65.24	2.40	3.68
Wheat Germ	15.67	0.96	6.13
Beans, Butter	17.36	1.96	11.31
Beans, French	25.64	1.51	5.87
Beans, Kidney	16.33	1.04	6.39
Brussels Sprouts	30.23	2.39	7.89
Cabbage	21.60	1.68	7.79
Carrots	32.29	3.68	11.39
Chick Peas	16.69	2.80	16.80
Okra	24.15	3.28	13.57
Onions	13.32	1.57	11.79
Parsley	34.39	4.69	13.64
Turnips	21.38	3.55	16.61

*a. Dry Weight Basis; Prosky, L. et al. (1992) JAOAC 75, 360-367.

Table 4. Comparison of Two TDF Methods - Preliminary Study

LAB		$\bar{x}$, TDF		S.D.*a	
		AOAC 985.29	AOAC 991.43	AOAC 985.29	AOAC 991.43
C	Oat Flour	10.8	10.7	0.60	0.35
C	Oat Cereal A	11.5	11.1	0.39	0.14
C	Oat Cereal B	7.9	7.6	0.29	0.18
C	Oat & Barley Cereal	14.6	14.5	0.26	0.14
CT	Rye Bread	6.7	6.6	0.33	0.05
CT	White Wheat Four	2.8	2.8	0.08	0.09
C	Apple	11.9	12.5	0.91	0.36
C	Carrot	26.6	26.4	0.77	0.60

*a. Dry Weight Basis; N=6.

Table 5. The Performance of the AOAC TDF Method 991.43

	$\bar{x}$ (g/100g)*a		RSDR,%
	Measured	Calculated (SDF+IDF)	Measured
Calculated (SDF+IDF)			
CEREAL PRODUCTS			
Barley	12.2	12.1	6.9
6.7			
High Fiber Cereal	33.7	33.0	2.8
2.7			
Oat Bran(Pretrial)	16.9	16.9	12.2
8.8			
Soy Bran	67.1	67.6	1.6
1.4			
FRUITS & VEGETABLES			
Apricots	1.1	1.1	2.2
4.2			
Prunes(Pretrial)	9.3	9.4	4.3
3.2			
Raisins	3.1	3.1	5.0
7.0			
Carrots	3.9	3.9	3.2
3.2			
Green Beans	2.9	3.0	2.6
4.0			
Parsley	2.7	3.0	5.2
7.5			

*a. As-is Basis; Lee et al. (1992) JAOAC 75, 395-416.

Table 6. The Performance of the AOAC SDF/IDF Method 991.43

Insoluble DF		Soluble DF		
RSDR,%		$\bar{x}$(g/100g)*a	RSDR,%	$\bar{x}$(g/100g)*a
	CEREAL PRODUCTS			
	Barley	5.0	12.2	7.0
8.2				
	High Fiber Cereal	2.8	20.4	30.5
2.3				
	Oat Bran(Pretrial)	7.2	15.9	9.7
12.0				
	Soy Bran	6.9	8.7	60.5
1.3				
	FRUITS & VEGETABLES			
	Apricots	0.53	7.3	0.59
2.9				
	Prunes(Pretrial)	5.1	6.7	4.2
6.0				
	Raisins	0.73	21.7	2.4
4.9				
	Carrots	1.1	16.5	2.8
5.7				
	Green Beans	1.0	10.6	2.0
4.2				
	Parsley	0.6	15.0	2.4
9.9				

*a. As-is (fresh weight) Basis; Lee et al. (1992) JAOAC 75, 395-416.

References

1 Burkitt, D.P. Epidemiology of cancer of the colon and rectum. Cancer 28:3, 1971.

2 Trowell, H.Ischemic heart disease and dietary fiber. Am. J. Clin. Nutr. 25:926, 1972.

3 Trowell, H.C., Southgate,D.A.T., Wolever,T.M.S., Leeds,A.R., Gassull, M.A., Jenkins, D.J.A. Dietary fiber redefined. Lancet 1:967, 1976.

4 Asp, N.-G., Schweizer, T.F., Southgate, D.A.T. and Theander, O. Dietary fiber analysis. In: Dietary fiber- A component of food: Nutritional function in health and disease. T.F.Schweizer and C.A. Edwards, Eds., Springer-Verlag, London pp 57-101, 1992.

5 Lee,S.C. and Prosky,L. Dietary fiber analysis for nutrition labeling. Cereal Foods World 37, 1992, in press.

6 Prosky, L., Asp, N.-G., Furda, I., DeVries, J.W., Schweizer, T.F., and Harland, B. Determination of total dietary fiber in foods and food products: Collaborative study. J. Assoc. Off. Anal. Chem. 68:677, 1985. (1985)

7 Prosky, L., Asp, N.-G., Schweizer, T.F., DeVries, J.W., and Furda, I. Determination of insoluble, soluble and total dietary fiber in foods and food products: Interlaboratory study. J. Assoc. Off. Anal. Chem. 71:1017, 1988. (1988)

8 AOAC Method 985.29. Total dietary fiber in foods. Enzymatic- gravimetric method. Official Methods of Analysis of the Association of Official Analytical Chemists, 15th ed. The Association: Arlington, VA, 1990.

9 Prosky,L., Asp. N.-G., Schweizer, T.F., DeVries, J.W. and Furda, I. Determination of insoluble and soluble dietary fiber in foods and food products: Collaborative study. J. Assoc. Off. Anal. Chem. 75, 360-367, 1992.

10 AOAC Method 991.42. Insoluble dietary fiber in foods and food products. Enzymatic-gravimetric method, phosphate buffer. Official Methods of Analysis of the Association of Official Analytical Chemists, 15th ed., 3rd suppl. the Association: Arlington, VA, 1992.

11 Prosky,L. Personal written communication, 1992.

12 Lee, S., Prosky, L. and Devries, J.W. Determination of total, soluble, and insoluble dietary fiber in foods: Collaborative study. J. Assoc. Off. Anal. Chem. 75, 395-416, 1992.

13 AOAC Method 991.43. Total, soluble, and insoluble dietary fiber in foods. Enzymatic-gravimetric method, MES-TRIS buffer. Official Methods of Analysis of the Association of Official Analytical Chemists, 15th ed., 3rd suppl. Association: Arlington, VA, 1992.