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Application of dietary fiber method AOAC 2011.25 in fruit and comparison with AOAC 991.43 method

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ABSTRACT

AOAC 2011.25 method enables the quantification of most of the dietary fiber (DF) components according to the definition proposed by Codex Alimentarius. This study aimed to compare the DF content in fruits analyzed by the AOAC 2011.25 and AOAC 991.43 methods. Plums (*Prunus salicina*), atemoyas (*Annona x atemoya*), jackfruits (*Artocarpus heterophyllus*), and mature coconuts (*Cocos nucifera*) from different Brazilian regions (3 lots/fruit) were analyzed for DF, resistant starch, and fructans contents. The AOAC 2011.25 method was evaluated for precision, accuracy, and linearity in different food matrices and carbohydrate standards. The DF contents of plums, atemoyas, and jackfruits obtained by AOAC 2011.25 was higher than those obtained by AOAC 991.43 due to the presence of fructans. The DF content of mature coconuts obtained by the same methods did not present a significant difference. The AOAC 2011.25 method is recommended for fruits with considerable fructans content because it achieves more accurate values.

1. Introduction

Dietary fiber (DF) is an important component of the human diet. Although it is not hydrolyzed and absorbed in the upper gastrointestinal tract, DF can be fermented in the lower gastrointestinal tract and provides health benefits when consumed regularly (Latulippe et al., 2013).

The establishment of definitions and analytical methods able to quantify all the compounds included in the DF fraction of a food is a complex process. Although several definitions have been proposed over the past 40 years, the Codex Alimentarius Commission established a definition only in 2008, defining DF as follows: "Dietary fiber is composed of carbohydrate polymers with ten or more monomeric units, which are not hydrolyzed by the endogenous enzymes in the small intestine of humans". The decision regarding the inclusion of non-digestible oligosaccharides (NDO) (DP 3–9) in the DF definition was left to national authorities (Codex Alimentarius, 2008; Codex Alimentarius, 2009).

In addition, the Codex Alimentarius Commission also recommended well-established methods of DF analysis, separating them into four groups: official general methods that do not measure the lower molecular weight fraction; official general methods that measure both the higher and the lower molecular weight fractions; official specific methods, developed to quantify individual specific DF components; and other methods (non-official methods) (Codex Alimentarius, 2009).

The general methods AOAC 985.29 (Prosky et al., 1985) and AOAC 991.43 (Lee, Prosky, & De Vries, 1992) are the enzymatic-gravimetric methods most used in determining the DF content of foods. However, these "traditional" methods do not quantify NDO, compounds present in the lower molecular weight dietary fiber (LMWDF) fraction, or resistant starch (RS) in its entirety. Thus, the development of new methods began to solve such shortcomings.

Abbreviations: AOAC, Association of Official Analytical Chemists; DF, dietary fiber; DP, degree of polymerization; HMWDF, high molecular weight dietary fiber; HMWSDF, high molecular weight soluble dietary fiber; HPLC-RID, high performance liquid chromatography coupled with refractive index detector; IDF, insoluble dietary fiber; LMWDF, low molecular weight dietary fiber; LMWSDF, low molecular weight soluble dietary fiber; NDO, non-digestible oligosaccharides; RS, resistant starch; RT, retention time; SDF, soluble dietary fiber; TDF, total dietary fiber.

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The enzymatic-gravimetric methods AOAC 2009.01 (McCleary et al., 2010) and AOAC 2011.25 (McCleary et al., 2012) are "new" general methods that quantify most of the DF components included in the definition proposed by the Codex Alimentarius, including the LMWDF fraction. The AOAC 2009.01 method quantifies the total dietary fiber (TDF), including both higher molecular weight dietary fiber (HMWDF) and LMWDF; the AOAC 2011.25 method is an extension of the previous method that quantifies TDF and its insoluble and soluble fractions separately: insoluble dietary fiber (IDF); high molecular weight soluble dietary fiber (HMWSDF); and low molecular weight soluble dietary fiber (LMWSDF).

Hollmann, Themeier, Neese, and Lindhauer (2013) compared the results obtained by the AOAC 2009.01 and 991.43 methods in the analysis of DF content of cereal-derived food products, noting that the values obtained by each method were different. Hollmann et al. (2013) highlighted the importance of comparing the DF content of other food groups using both methods in order to assess whether the results obtained by "traditional" methods need to be replaced with those obtained by "new" methods.

Englyst et al. (2013), Brunt and Sanders (2013), and McCleary, Sloane, Draga, and Lazewska (2013) also noted differences in the results obtained by "traditional" and "new" general methods in industrialized foods, matrices with high RS content and vegetables respectively.

The evaluation of DF content in fruits using the AOAC 2009.01 and 2011.25 methods is still limited. The DF of fruit may be underestimated when analyzed using "traditional" methods, considering that the AOAC 985.29 and 991.43 methods are not able to quantify fructans (fructooligosaccharides) and other NDO from this food matrix. These oligosaccharides are considered prebiotic compounds and may often be found in fruits and others natural sources (Jovanovic-Malinovska, Kuzmanova, & Winkelhausen, 2013).

The aim of this study was to compare the DF contents of fruits analyzed by the AOAC 2011.25 and AOAC 991.43 methods. The study included two steps: the first involved evaluating the AOAC 2011.25 method under laboratory conditions, while the second involved the analysis of DF of fruits cultivated in different regions of Brazil using the AOAC 2011.25 method and comparing the results obtained with those of the AOAC 991.43 method.

2. Material and methods

2.1. Sampling

Three lots of plums (*Prunus salicina* Lindl cv. Reubennel), atemoyas (*Annona x atemoya* Mabb. cv. Thompson), jackfruits (*Artocarpus heterophyllus* Lam. var. soft) and mature coconuts (*Cocos nucifera* L. var. dwarf) were obtained from CEAGESP (the main market of São Paulo, Brazil) during their respective harvest periods. Each lot was collected from a different cultivation area (n = 3) using simple sampling with no repetitions, considering the amount sold at CEAGESP (CONAB, 2015) as a criterion. The criteria for the selection of the four fruit types were: cultivation in Brazil (Lorenzi, Bacher, Lacerda, & Sartori, 2006); consumption by Brazilian population, according to the Brazilian household budget survey (POF – *Pesquisa de Orçamentos Familiares*) of 2008–2009 (IBGE, 2011); lack of data in the Brazilian Food Composition Database; availability for acquisition in São Paulo, Brazil.

Atemoyas (4 kg/lot) were obtained in June 2014, jackfruits (10 kg/lot) in July 2014, plums (6 kg/lot) in December 2014, and mature coconuts (20 kg/lot) in March 2015. Plums, atemoyas, and jackfruits were obtained at the unripened stage.

Ripe bananas (*Musa acuminata*, AAA, cv. Nanica), cabbage, and oat bran were used to evaluate the AOAC 2011.25 method.

2.2. Sample preparation

Plum, atemoya, and jackfruit samples were kept under room conditions (22 °C, relative humidity was 80%) until they reached the ideal maturity stage for consumption, which was identified using sensory parameters: characteristic color and firmness of the fruit peel and characteristic fruity odor. Mature coconut samples were obtained in the ideal maturity stage. Subsequently, the edible part of the samples was separated, homogenized, frozen in liquid nitrogen, freeze-dried, ground into particles <60 mesh and stored at −20 °C until the analysis. Moisture content was determined by the AOAC 934.06 method (Horwitz & Latimer, 2008) using a vacuum oven (70 °C; ⩽100 mmHg).

2.3. Enzyme assay kits, standards and reagents

Commercial enzymatic assay kits were purchased from Megazyme (Megazyme International Ireland Ltd., Bray, Ireland): DF measured by AOAC 2011.25 (K-INTDF) and AOAC 991.43 (K-TDFR) methods; RS measured by AOAC 2002.02 method (K-RSTAR); fructans measured by AOAC 999.03 method (K-FRUC). The RS analysis control kits (K-RSTCL), Amberlite FPA OH^- (G-AMBOH), and Ambersep 200 H^+ (G-AMBH) resins were also purchased from Megazyme. The following carbohydrate standards and reagents obtained from Sigma–Aldrich Chemical Co. (St. Louis, MO, USA) were used: D-(+)-Xylose (⩾99%); D-(+)-Fucose (⩾98%); L-(−)-Galactose (⩾99%); D-(+)-Mannose (⩾99%); D-(+)-Glucose (⩾99.5%); D-(−)-Fructose (⩾99%); D-Sorbitol (99%); D-Glucuronic Acid (⩾98%); D-(+)-Galacturonic Acid (⩾97%); Lactulose (⩾95%); D-(+)-Sucrose (⩾99.5%); D-(+)-Raffinose (⩾98%); Stachyose (⩾98%); D-(+)-Maltose (⩾99%); Maltotriose (⩾90%); Maltotetraose (⩾95%); Maltopentaose (⩾95%); Maltohexaose (⩾65%); Maltoheptaose (⩾60%); Inulin (Chicory); Ethylenediaminetetraacetic acid calcium disodium salt (Na_2Ca-EDTA); and sodium azide. The standards used in the AOAC 2011.25 method evaluation step were prepared in 0.02% sodium azide solution and the internal standard (D-Sorbitol) was added at a 1:9 ratio. Deionized water (18.2 MΩ/cm) was obtained using the Milli-Q-plus purification system (Millipore Corp., Bedford, MA, USA).

2.4. Methods

All chemical analyses were performed in quadruplicate. The results (mean ± standard deviation) were expressed as g/100 g of dry weight.

2.4.1. Resistant starch

The quantification of the RS content was based on the AOAC 2002.02 method (McCleary, McNally, & Rossiter, 2002), using the Resistant Starch Assay Kit (K-RSTAR). The amount of free glucose produced after hydrolysis was quantified using an enzymatic method (glucose oxidase/peroxidase/ABTS) and the absorbance was measured at 510 nm. The total RS was calculated by multiplying the measured free glucose by a conversion factor of 0.9. Resistant Starch Control Flours (K-RSTCL) were used as reference material.

2.4.2. Fructans

The fructans content was analyzed by the enzymatic-spectrophotometric method AOAC 999.03 (McCleary, Murphy, & Mugford, 2000), using the Fructan Assay Kit (K-FRUC). The fructans concentration was indirectly determined by the reaction between 4-hydroxybenzoic hydrazide acid (PAHBAH) and sugars produced after hydrolysis. The absorbance was measured at 410 nm. Fructose (K-FRUC) was used as reference material.

2.4.3. Dietary fiber

The DF content was determined according to the AOAC 2011.25 (McCleary, 2014) and AOAC 991.43 (Lee et al., 1992) methods.

The AOAC 2011.25 method was applied using the Integrated Total Dietary Fiber Assay Kit (K-INTDF). After the filtration of the IDF and HMWSDF fraction, the LMWSDF (50 μL) was injected into a chromatograph system equipped with two Shimadzu (Shimadzu Co., Tokyo, Japan) pumps (LC-20AT), a Shimadzu autosampler (SIL-20A Autosampler), a Shimadzu refractive index detector (RID-10A), a Waters (Waters Associates, Milford, MA, USA) Guard-Pak precolumn, a Waters Sugar-Pak I column (300 mm × 6.5 mm), and Shimadzu LCsolution software. The mobile phase consisted of Na_2Ca-EDTA (50 mg/L) at a constant flow (0.5 mL/min). Glucose and D-Sorbitol were used as reference standards and LC Retention Time Standard (K-INTDF) as a retention time standard. Monosaccharide, disaccharide, and oligosaccharide reference standards were analyzed directly by HPLC-RID. Each standard was analyzed in five concentrations, while each concentration was analyzed in ten replicates. The evaluated parameters were retention time (RT) and area under the curve. The TDF obtained by the AOAC 2011.25 method was determined as the sum of IDF, HMWSDF, and LMWSDF.

The AOAC 991.43 method was applied using the Total Dietary Fiber Assay Kit (K-TDFR) and TDF was determined as the sum of IDF and SDF.

2.5. Parameters for quality assessment of the DF content by the AOAC method 2011.25

The precision of the method was determined by the coefficient of variation (CV) calculated using the mean and standard deviation (SD) values. Accuracy was determined by the percentage of recovery in terms of the obtained and expected values. The correlation coefficient (r) of the linear regression curve between obtained and expected values was adopted as a parameter to determine linearity (AOAC, 2002).

2.6. Statistical analysis

The normality and homogeneity of variances were verified using the Shapiro-Wilk and Levene's tests, respectively. A paired Student's *t*-test was used to compare the means and Pearson's test for the correlation analyses. P values <0.01 were considered to indicate significant differences. Statistical analyses were performed using the IBM® SPSS® Statistics 20.0 software (IBM Corp, Armonk, NY, USA).

3. Results and discussion

3.1. Evaluation of suitability of the DF content by the AOAC 2011.25 method under laboratory conditions

Since the laboratory wanted to be assured beforehand that the AOAC 2011.25 method adequately analyzes the DF content, three tests were performed under laboratory conditions. The method was integrally evaluated, as well as the chromatographic step separately. Food samples from the collaborative study of method validation were analyzed and the results obtained under laboratory conditions were compared with those presented by McCleary et al. (2012).

Initially, the chromatographic analysis was evaluated separately. Since the LMWSDF fraction comprises a pool of NDO quantified by HPLC-RID, this test aimed to evaluate the result obtained individually by each carbohydrate. For this purpose, mono-, di-, and oligosaccharide standards were directly analyzed in the HPLC-RID. Precision, accuracy and linearity of the results obtained for each carbohydrate standard, as well as the chemical characteristics and RT of each carbohydrate are presented in Table 1. It was noted that the carbohydrate standards analyzed by HPLC-RID under laboratory conditions provided results of acceptable precision, accuracy, and linearity, according to the AOAC Guidelines for Single Laboratory Validation of Chemical Methods for Dietary Supplements and Botanicals (AOAC, 2002).

The RT standard used in the AOAC 2011.25 method enzymatic assay kit delimited the range indicated to quantify the LMWSDF. Under the established conditions, the RT limits ranged from 4.500 to 7.650 min. Carbohydrates detected within the given range had DP between 3 and 7 and molecular weights ranging from 504.44 to 1153.00 u.

Simultaneously, analysis of a food matrix (ripe banana) with added RS or inulin standards (50% of the initial content of the DF from the matrix) was done in order to evaluate the recovery percentage of the added standards (Table 2). The parameters evaluated were precision and accuracy and the results indicated that the method was performed appropriately, yielding higher values

Table 1
Chemical structure, molecular weight (MW) (u), retention time (RT) (min), precision (%), accuracy (%), and linearity of carbohydrate standards analyzed by HPLC-RID.

Carbohydrates	Chemical structure	MW	RT	Precision	Accuracy	Linearity
Inulin (fructooligosaccharides)	$(C_6H_{10}O_5)_n$	-	5.168	7.78	103	0.9999
Maltoheptaose	$C_{42}H_{72}O_{36}$	1153.00	5.467	1.49	93	0.9984
Maltohexaose	$C_{36}H_{62}O_{31}$	990.86	5.827	2.40	100	0.9977
Maltopentaose	$C_{30}H_{52}O_{26}$	828.72	6.085	3.88	107	0.9984
Stachyose	$C_{24}H_{42}O_{21} \cdot xH_2O$	666.58	6.299	3.63	112	0.9972
Maltotetraose	$C_{24}H_{42}O_{21}$	666.58	6.569	3.19	109	0.9988
Raffinose	$C_{18}H_{32}O_{16} \cdot 5H_2O$	594.51	6.910	2.43	89	0.9962
Maltotriose	$C_{18}H_{32}O_{16}$	504.44	6.939	2.38	92	0.9996
Maltose	$C_{12}H_{22}O_{11} \cdot H_2O$	360.31	8.088	1.19	94	0.9999
Sucrose	$C_{12}H_{22}O_{11}$	342.30	8.445	2.10	97	0.9942
Lactulose	$C_{12}H_{22}O_{11}$	342.30	8.743	6.24	89	0.9909
Glucose	$C_6H_{12}O_6$	180.16	9.750	0.88	109	0.9923
Xylose	$C_5H_{10}O_5$	150.13	10.555	1.68	93	0.9973
Galactose	$C_6H_{12}O_6$	180.16	10.636	2.34	107	0.9980
Mannose	$C_6H_{12}O_6$	180.16	10.850	3.36	82	0.9882
Fructose	$C_6H_{12}O_6$	180.16	11.181	0.27	102	0.9954
Fucose	$C_6H_{12}O_5$	164.16	11.706	0.73	103	0.9955
Sorbitol	$C_6H_{14}O_6$	182.17	14.683	-	-	-

Highlighted lines indicate carbohydrates detected within the range indicated by the LC Retention Time Standard (Megazyme International Ireland Ltd., Wicklow, Ireland) used in AOAC 2011.25 method.

Table 2
Dietary fiber content (g/100 g), dry weight, precision (%), and accuracy (%) of the analyses of ripe banana (*Musa acuminata*) and ripe banana with added secondary standards.

	Ripe banana	Ripe banana with added	
		Resistant starch	Inulin
Total dietary fiber	24.43 ± 0.82	37.26 ± 0.93	36.83 ± 0.18
Insoluble dietary fiber	19.04 ± 0.43	30.46 ± 0.54	18.67 ± 0.11
Soluble dietary fiber	5.39 ± 1.02	6.80 ± 0.39	18.16 ± 0.14
Precision	NA	0.43	0.77
Accuracy	NA	113	112

Results obtained by AOAC 2011.25 method and expressed as mean ± standard deviation. NA: Not applicable.

Table 3
Expected and obtained values for dietary fiber content (g/100 g), dry weight, of cabbage and oat bran.

	Expected value	Obtained value
	Mean (Minimum – Maximum)	Mean ± SD
*Cabbage**		
Total dietary fiber	29.90 (26.45 – 35.66)	32.02 ± 0.78
Insoluble dietary fiber	25.74 (23.63 – 27.77)	26.35 ± 0.36
Soluble dietary fiber	3.80 (1.91 – 5.86)	5.29 ± 0.85
*Oat bran**		
Total dietary fiber	23.71 (20.17 – 29.63)	17.82 ± 0.39
Insoluble dietary fiber	11.52 (9.48 – 13.98)	10.69 ± 0.36
Soluble dietary fiber	11.29 (6.76 – 14.83)	7.13 ± 0.67

Results expected and obtained by AOAC 2011.25 method. *Samples used by the collaborative validation study of the official AOAC 2011.25 method (McCleary et al., 2012). SD: Standard deviation.

for ripe banana samples with added RS or inulin. The precision of the method was 0.43% for RS and 0.77% for inulin, while the accuracy was 113% and 112% respectively.

Finally, the AOAC 2011.25 method was evaluated by comparing values obtained under laboratory conditions with the range values presented by McCleary et al. (2012) in their collaborative study of method validation. Therefore, two of the eight food samples analyzed in the collaborative study (expected values) were also analyzed in the laboratory (obtained values). Cabbage and oat bran were the sample types chosen for analysis (Table 3). The obtained values remained within the range presented in the collaborative validation study, with the exception of the oat bran TDF value. The obtained value for oat bran TDF (17.82 g/100 g) was lower than the expected range (20.17–29.63 g/100 g) presented by McCleary et al. (2012). Although the same sample types used in this test were the same as the ones used in the collaborative study, the variation between obtained and expected values may be explained by the intrinsic variations in the DF content of the matrix.

Considering the results of the three tests performed, the AOAC 2011.25 method proved to be appropriate, providing precise, accurate, and linear results (AOAC, 2002) that were close to the expected values.

3.2. Comparison between results obtained by the AOAC 2011.25 and AOAC 991.43 methods applied to four fruits

The results (mean ± SD) presented refer to the mean values obtained by the AOAC 2011.25 and AOAC 991.43 methods for each one of the following four fruits (Table 4): atemoyas, mature coconuts, jackfruits, and plums. Fig. 1 presents a graphical comparison between the IDF, SDF, and TDF content of the four fruits (quantified using both methods) as well as the HMWDF content quantified by the AOAC 2011.25 method. HMWDF content corresponds to the sum of IDF and HMWSDF.

More specifically, the "new" integrated method (AOAC 2009.01 and 2011.25) is different from the "traditional" method (AOAC 985.29 and 991.43) because it includes the RS and the NDO in the DF measured value. The RS is quantified using enzymatic–gravimetric technique and included in the IDF fraction, while the NDO are quantified by high-performance liquid chromatography (HPLC) in the LMWSDF (Macagnan, Silva, & Hecktheuer, 2016).

The DF content obtained by the AOAC 991.43 method was shown to be appropriate to the fruits analyzed according to data available in the literature (Cruz, Lima, Abreu, Corrêa, & Pinto, 2013; Hettiaratchi, Ekanayake, & Welihinda, 2011; Lozano et al., 2009). Since the AOAC 2011.25 method had not previously been applied to the DF analysis of these fruits, there is no comparable data in the literature.

The IDF content of the four fruits, quantified by the AOAC 2011.25 and AOAC 991.43 methods, showed no significant difference (Table 4). The IDF values measured by both methods showed a strong positive correlation ($r = 0.797$, $p < 0.01$). The proximity of the IDF values quantified by each method can be explained by the fact that the fruits present a low RS content and that both methods are analyzing the same compounds. The RS content ranged from 0.04 g/100 g in mature coconuts to 1.00 g/100 g in jackfruits (Table 4).

It is well accepted in the literature that RS is analyzed as IDF, although it presents the physiological benefits of SDF. The "traditional" methods (AOAC 985.29 and 991.43) present a drawback by only measuring the RS type 3 (retrograded starch) (Westenbrink et al., 2013). The RS present in the fruits is quantified in its totality only by "new" methods (Brunt & Sanders, 2013).

In relation to the SDF fractions, the higher molecular weight fraction is measured by both methods while the lower molecular weight fraction is measured only by the "new" methods. When comparing the HMWSDF (AOAC 2011.25) and the SDF (AOAC 991.43) contents, only plum samples presented values with no significant difference between methods; the results were 8.62 and 8.23 g/100 g, respectively.

The achievement of the SDF fraction depends on many critical steps that can directly affect the SDF measurement. Type and pur-

Table 4
Dietary fiber (AOAC 2011.25 and AOAC 991.43), resistant starch (AOAC 2002.02) and fructan (AOAC 999.03) content (g/100 g), dry weight, of plums (*Prunus salicina* Lindl.), atemoyas (*Annona x atemoya* Mabb.), jackfruits (*Artocarpus heterophyllus* Lam.), and mature coconuts (*Cocos nucifera* L.).

Fruit	Moisture	AOAC 2011.25				AOAC 991.43			Resistant starch	Fructans
		IDF	HMWSDF	LMWSDF	TDF	IDF	SDF	TDF		
Atemoya	72.56 ± 1.84	16.17 ± 2.02^{a}	6.59 ± 2.32^{c}	1.16 ± 0.46	23.92 ± 4.02^{e}	13.83 ± 1.32^{a}	3.76 ± 1.01^{b}	17.59 ± 0.37^{d}	0.41 ± 0.14	8.12 ± 1.70
Coconut	44.17 ± 1.29	12.75 ± 1.11^{a}	0.89 ± 0.08^{c}	1.28 ± 0.67	14.92 ± 1.75^{d}	13.08 ± 0.60^{a}	0.29 ± 0.19^{b}	13.37 ± 0.61^{d}	0.04 ± 0.01	0.06 ± 0.02
Jackfruit	76.77 ± 3.98	10.85 ± 1.30^{a}	2.08 ± 0.48^{b}	10.61 ± 0.94	23.55 ± 2.29^{e}	9.46 ± 1.39^{a}	4.13 ± 0.48^{c}	13.59 ± 1.46^{d}	1.00 ± 0.43	6.45 ± 0.95
Plum	87.42 ± 2.28	8.74 ± 2.23^{a}	8.62 ± 0.63^{b}	4.94 ± 1.12	22.30 ± 2.66^{d}	9.13 ± 1.46^{a}	8.23 ± 1.15^{b}	17.36 ± 2.28^{c}	0.42 ± 0.08	7.18 ± 1.77

IDF: insoluble dietary fiber; SDF: soluble dietary fiber; HMWSDF: high molecular weight soluble dietary fiber; LMWSDF: low molecular weight soluble dietary fiber; TDF: total dietary fiber. Results expressed as mean ± standard deviation. n = 3 (3 lots from different cultivation areas. Each lot was composed by 10 plums, 9 atemoyas, 3 jackfruits or 10 mature coconuts). The different letters in the same row and fraction indicate a significant difference between methods ($p < 0.01$).

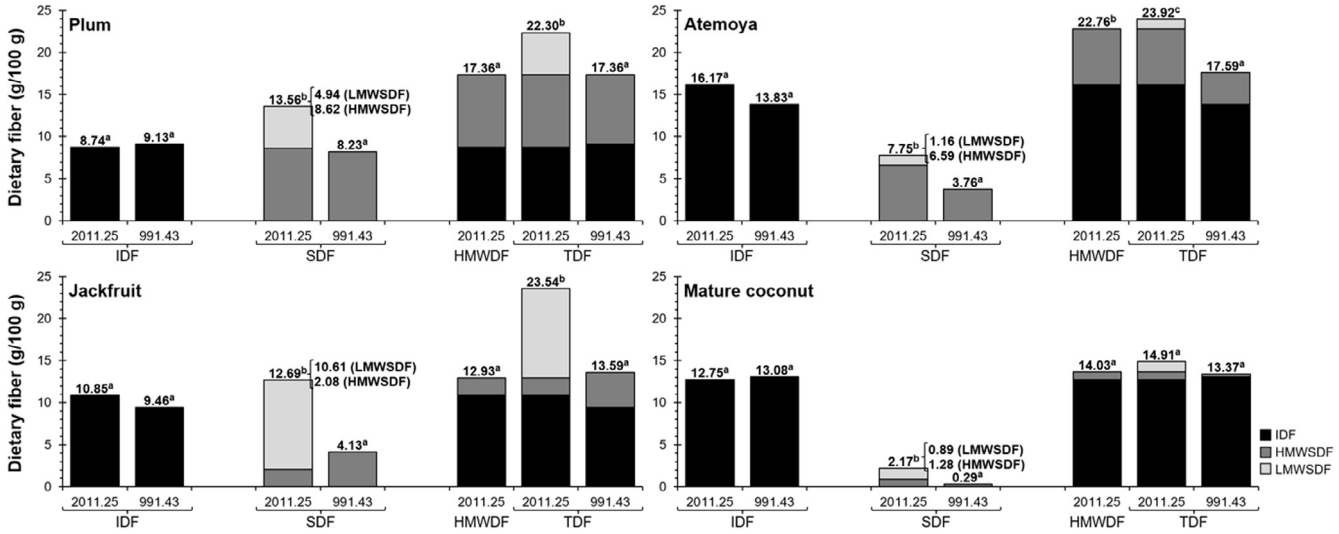

Fig. 1. Insoluble dietary fiber (IDF), high molecular weight soluble dietary fiber (HMWSDF), and low molecular weight soluble dietary fiber (LMWSDF) content (g/100 g) of plums (*Prunus salicina* Lindl.), atemoyas (*Annona x atemoya* Mabb.), jackfruits (*Artocarpus heterophyllus* Lam.), and mature coconuts (*Cocos nucifera* L.), dry weight, analyzed by the AOAC 2011.25 and AOAC 991.43 methods. HMWDF: high molecular weight dietary fiber. The different letters in the same fraction indicate a significant difference between methods ($p < 0.01$).

ity of the enzymes, incubation time and temperature, precipitation conditions, the temperature (60 °C) and concentration (78%) of the ethanol solution are some of the critical factors. The AOAC 2011.25 and 991.43 methods use different enzymes, as well as different times and temperatures for incubation (Lee et al., 1992; McCleary et al., 2012). These factors may have affected the SDF quantification, resulting in final values with significant differences.

Once the LMWSDF content cannot be quantified using the "traditional" method (AOAC 991.43). The LMWSDF present in the fruits was quantified using only the AOAC 2011.25 method; its values ranged from 1.16 g/100 g in atemoyas to 10.61 g/100 g in jackfruits.

Due to the low content of RS, the HMWDF values (AOAC 2011.25) for plums, jackfruits, and mature coconuts showed no significant difference when compared with the TDF as quantified by the AOAC 991.43 method (Fig. 1). The atemoyas showed a significant difference between the HMWDF (22.76 g/100 g) and TDF (17.59 g/100 g) values. As shown in Fig. 1, the IDF values obtained by both methods showed no significant difference, indicating that the difference between the HMWDF and TDF values resulted from the atemoyas' high content of HMWSDF (6.59 g/100 g) when compared with the SDF quantified by the "traditional" method (3.76 g/100 g).

When comparing the TDF content quantified by both methods, significant differences were observed in the values obtained for plums, atemoyas, and jackfruits; the data was congruent with the fructans content observed. The fructans content was high in plums (7.18 g/100 g), atemoyas (8.12 g/100 g), and jackfruits (6.45 g/100 g), while it was low in mature coconuts (0.06 g/100 g).

The positive correlation observed between the content of fructans (AOAC 999.03) and SDF (AOAC 2011.25) ($r = 0.748$, $p < 0.01$) (Fig. 2A), as well as the correlation observed between the HMWSDF (AOAC 2011.25) and the SDF (AOAC 991.43) contents ($r = 0.794$, $p < 0.01$) (Fig. 2B) indicate that fructans were included in the DF as quantified by the AOAC 2011.25 method. The TDF analyzed by the AOAC 991.43 method showed a stronger positive correlation with the HMWDF ($r = 0.745$; $p < 0.01$) quantified using the AOAC 2011.25 method than with the TDF ($r = 0.507$; $p < 0.01$) quantified

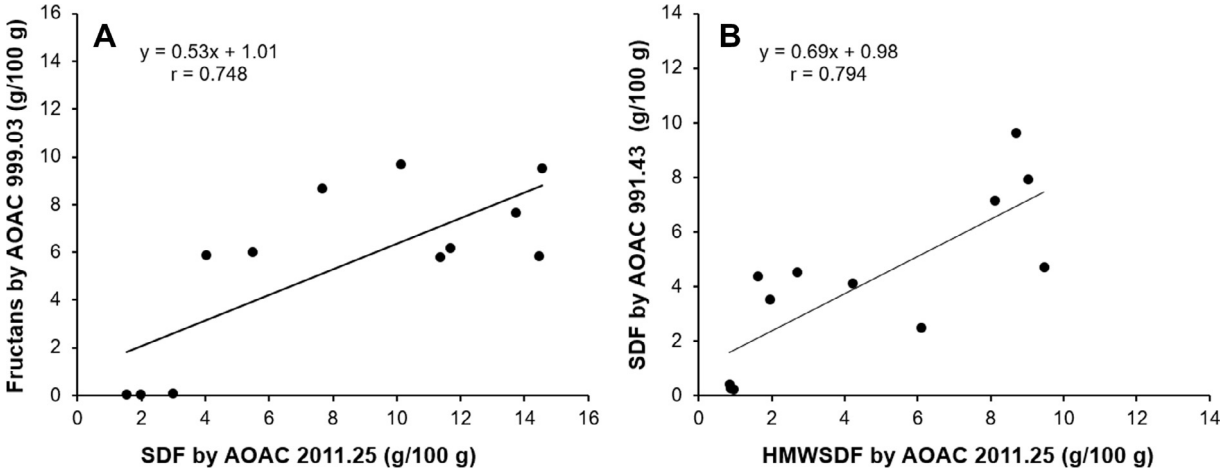

Fig. 2. Correlation between dietary fiber (DF) fractions determined by different analytical methods: (A) fructans by the AOAC 999.03 and soluble DF (SDF) by the AOAC 2011.25 method; (B) high molecular weight soluble DF (HMWSDF) by the AOAC 2011.25 and SDF by the AOAC 991.43 method.

by the same method. These correlations also suggest the fact that the fruits had a low RS content, corroborating the results obtained in terms of the IDF and RS.

As far as the IDF:SDF ratio when applying the AOAC 2011.25 method, the IDF corresponded to 39.19% of TDF in plums and 85.46% of TDF in mature coconuts. When applying the AOAC 991.43 method, the DF content of the same fruit comprised 52.59% and 97.83% of IDF respectively. The change in the IDF:SDF ratio can be attributed to the inclusion of the LMWSDF fraction, that ranged from 4.85% (atemoya) to 37.05% (jackfruit).

Recent studies have compared the DF values obtained by the "traditional" (AOAC 985.29 and 991.43) and the "new" integrated AOAC methods (AOAC 2009.01 and 2011.25) (Brunt & Sanders, 2013; Hollmann et al., 2013; Westenbrink et al., 2013).

Hollmann et al. (2013) observed differences in the DF values from cereal-derived food products when measured by the AOAC 2009.01 and AOAC 991.43 methods. When compared the values obtained using each method, the HMWDF contents obtained by the AOAC 2009.01 method were closer to the TDF content obtained using the AOAC 991.43. The TDF content quantified by the AOAC 991.43 method was also lower than the one obtained by the AOAC 2009.01 method.

The importance of compare the values obtained by both methods was highlighted by Hollmann et al. (2013). They have argued that it is important to undertake similar research for other food groups in order to identify possible correlations and to evaluate whether certain groups should be analyzed again.

Comparative studies between "traditional" and "new" methods have also been performed with industrialized foods (Englyst et al., 2013), foods with high RS content (Brunt & Sanders, 2013), and vegetables (McCleary et al., 2013). In the study of these food groups, the "new" methods provided higher values in comparison to "traditional" methods; this difference was justified by fact that the RS present in the samples was only included in the DF quantified by "new" methods.

In contrast to the results presented by Hollmann et al. (2013), Englyst et al. (2013), Brunt and Sanders (2013) and McCleary et al. (2013), the present study determined – through a comparison of the AOAC 2011.25 and AOAC 991.43 methods – that the RS did not influence the DF quantification in the fruits that were analyzed, since it was present in a small amount (<1.00 g/100 g). However, the results obtained by the present study suggest that the inclusion of fructans (fructooligosaccharides) in fruits' DF values when quantified by AOAC 2011.25 method was an important advantage. Only fruits that had a considerable content of these compounds showed higher DF values using the AOAC 2011.25 method.

Apart from vegetables and other food groups (Biesiekierski et al., 2011; Judprasong, Tanjor, Puwastien, & Sungpuag, 2011), fruits can naturally present a wide variance of fructooligosaccharides (Jovanovic-Malinovska et al., 2013) and fructans contents (Muir et al., 2007). Therefore, the results obtained in the present study highlight the importance of the analytical method choice in DF analysis.

4. Conclusions

The content of dietary fiber analyzed by AOAC 2011.25 method was higher than that determined using the "traditional" method (AOAC 991.43) for fruits with a considerable fructans content, such as plums, atemoyas, and jackfruits.

The use of the AOAC 2011.25 method to determinate dietary fiber is relevant for fruits with a high content of lower molecular weight carbohydrates, since it provides more accurate results.

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