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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.