



Dietary Fibers: Analysis Methods

Fibra Alimentar: Métodos de Análises

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Resumo

O consumo de fibra alimentar (FA) é associado a uma série de benefícios e esses efeitos dependem não somente da ingestão como também da sua composição. A FA inclui polissacarídeos, como celulose, hemiceluloses, pectinas, gomas, oligossacarídeos e lignina, e podem ser agrupadas em solúveis e insolúveis. O conceito de FA foi ampliado para incluir amido resistente, inulina e fruto-oligosacarídeos. A determinação de fibra alimentar depende de métodos dispendiosos e longos que foram modificados desse novo conceito. Os métodos oficiais da AOAC permitem determinar todos os componentes presentes na fibra de um alimento, sem precisar de métodos específicos para cada componente. Estudos mostram técnicas inovadoras que garantem um menor tempo de análise, menor geração de

lixo pelo uso de reagentes e mais praticidade nas análises.

Abstract

Dietary fiber (DF) intake is associated with a number of benefits and these effects depend not only on intake as well as its composition. The DF includes polysaccharides such as cellulose, hemicellulose, pectins, gums, oligosaccharides and lignin, and can be divided into soluble and insoluble. The concept of DF was expanded to include resistant starch, inulin and fructo-oligosaccharides. The determination of DF costly and time depends on methods that have been modified for this new concept. The AOAC Official methods of determining all components present in a DF, without specific methods for each component. Studies show innovative techniques to ensure a shorter analysis time, less waste generation by the use of reagents and more convenience in the analysis.

INTRODUCTION

Dietary fiber (DF) is an essential component of a healthy diet and the health benefits associated with its regular consumption include regulating the intestinal tract and preventing diseases such as diabetes, hypertension, obesity, coronary heart disease and colon cancer (Bernaud e Rodrigues, 2013;

Ferreira et al., 2015; Macagnam et al., 2016).

Due to this importance, over the last decades, the knowledge about DF has increased considerably both in the physiological and in the analytical domain (Westenbrink et al., 2013). Fresh fruits and vegetables are an important part of the human diet as a significant source of water,

vitamins and natural sugars and are also the main source of DF (Chylinska et al., 2016).

According Food and Drug Administration (FDA, 1995), fiber-rich foods are included in the functional foods category because of their use within a balanced diet that may reduce the risk of some diseases (Ferreira et al., 2015). More than 50% of the functional foods available on the market have DF as an active component (Macagnam et al., 2016). Dietary fiber intake recommendations vary according to age, gender and energy consumption, with an adequate recommendation of around 14 g of fiber for every 1.000 kcal ingested (IOM, 2005). The beneficial effects and effectiveness of DF depend not only on intake, but also on the composition of these fibers, their organizational structure and their physico-chemical characteristics, which are directly related to their plant origin or their preparation methods (Aldwairji et al., 2014; Macagnam et al., 2016). Thus, the purpose of this review is to provide information on DF, its definitions, and methods of analysis.

DEFINITION: DIETARY FIBER

According to the Codex Alimentarius (2009), the new definition of DF, consists of carbohydrate polymers with at least 10 monomeric units that are neither digested nor absorbed by the human small intestine. In the European Union (EU), oligosaccharides with 3-9 monomeric units are also considered as DF. It is the edible part of plants or analogous carbohydrates that occur naturally or may be synthetic, obtained by physical, chemical or

enzymatic means (Catalani et al., 2003; Rainakari et al., 2016). Within the class of carbohydrate polymers include oligosaccharides and polysaccharides, such as cellulose, hemicellulose, pectin, gums, analogous carbohydrates (resistant starch and resistant maltodextrins), inulin, which may be associated with lignin and other compounds (phenolic compounds, cell wall protein, oxalates, phytates, waxes, cutin and suberin) (Elleuch et al., 2011; Bernaud e Rodrigues, 2013). An even greater definition of DF may include animal fibers (chitin, chitosan, collagen and chondroitin) and nondigestible, modified or synthetic carbohydrate polymers (Degree of polymerization ≥ 3), for example, polydextrose, fructo-oligosaccharides (Elleuch et al., 2011). Resistant starch (RS) is composed of four groups in foods: RS1 - physically inaccessible starch, which is retained in whole or in part, in the ground grains or seeds; RS2 - some types of crude starch granules (such as banana and potato) and high amylose starches (corn); RS3 – retrograded starch (or processed resulting from food processing applications); RS4 – chemically modified starches to obtain resistance to enzymatic digestion (Fuentes-Zaragoza et al., 2010).

According to the solubility of its components the DF are classified as soluble fibers or insoluble fibers and the ratio between the fractions of these fibers is important for food and functional properties. These denominations provided a simple and useful classification for DF with different physiological properties

(Macagnam et al., 2016). Soluble fibers include most pectins, gums and hemicelluloses and are responsible for increasing the exposure time of nutrients in the gastrointestinal tract, providing an improvement in digestion thereof, in particular of sugars and fats. They can be found in fruits, oat bran, barley and legumes (lentils, peas, chickpeas) (Ferreira et al., 2015). Insoluble fibers include cellulose, lignin and some hemicelluloses. They are found, in greater quantity, in wheat bran, in whole grains and their products, in roots and vegetables (Catalani et al., 2003). The main function of insoluble fibers is the intestinal due to their extreme water retention capacity, which contributes to the distension of the colon wall and facilitates the elimination of the fecal cake. By absorbing the water, any toxic agents are also absorbed, which prevents diseases such as colon cancer. The soluble fibers also act on the speed of the intestinal transit, but without increasing the absorption of water (Bernaud e Rodrigues, 2013). Table 1 shows the types of DF, groups and main sources.

METHODS OF ANALYSIS OF DIETARY FIBERS

Until about 2005, the classical methods for determining the DF content in food products were the methods AOAC 985.29 and AOAC 991.43. Both methods quantify only components of high molecular weight dietary fiber (HMWDF) soluble and insoluble, being that the first quantifies directly the total dietary fiber (TDF) of a food and the second distinguishes the

soluble and insoluble fractions of the fiber (Devries, 2010 apud Macagnam et al., 2016).

The new definition of DF introduced by the Codex Alimentarius in 2008 included low molecular weight dietary fiber (LMWDF), such as inulin, fructooligosaccharides, galactooligosaccharides and polydextrose, and it is necessary to implement this definition to new methodologies. The increased use of LMWDF in food products also contributed to the confirmation that official methods AOAC 985.29 and 991.43 were becoming inadequate to quantify TDF in food (Macagnam et al., 2016; Brunt e Sanders, 2013, Westenbrink et al., 2013).

Another drawback of the classical methods is that only the RS3 type of resistant starch (retrograded starch, the RS type predominant in most food products) is measured. Thus, a number of specific AOAC methods were developed to measure the DF different components separately, which made the choice of a correct measurement of this fraction in an unknown sample extremely complex (Elleuch et al., 2011). This was not the solution because there is a considerable overlap between the methodologies, as shown in Figure 1. However, Englyst et al. (2013), observed that the values of DF obtained by the classical and specific approaches were well correlated as a whole, indicating that in many situations, they can be used for routine analysis purposes.

In 2007, an integrated method for the determination of TDF, including non-digestible oligosaccharides, was described by McCleary (2007). This process is now known as a method

for the determination of TDF (AOAC 2009.01) and measures the HMWDF fraction by enzymatic-gravimetric techniques, and LMWDF by high performance liquid chromatography (HPLC). The sample is first incubated with α -amylase at 37 °C and then the protein is digested with the protease at 60 °C. The soluble and insoluble HMWDF (which is precipitated with 78% ethanol) are determined gravimetrically. Non-digestible oligosaccharide are quantify in ethanol filtered by HPLC. In this new method AOAC 2009.01 parts of different existing AOAC methods have been combined, in order to develop a method where the deficiencies of other methods are eliminated. This method (AOAC 2009.01) is especially useful for measuring DF content in foods enriched with prebiotics because it eliminates the need to apply both the AOAC 985.29 method and e a specific method for the analysis of non-digestible oligosaccharides (Brunt e Sanders, 2013).

Vasconcelos et al. (2010) observed in yacon flour that in addition to inulin and oligofrutans were also found, glucose, fructose and sucrose, which may contribute to the determination of incomplete information on the dietary fiber composition of yacon flour determined by the enzymatic-gravimetric method of AOAC followed by HPLC. However, this association proved to be efficient in the quantification of soluble dietary fiber mainly in foods that contain high concentrations of inulin and fructooligosaccharide.

An extension of the AOAC 2009.01, known as the method AOAC 2011.25, was

developed (Mccleary et al., 2012). In this method, the HMWDF fraction is divided into soluble and insoluble, which together represent the total content. All DF components of the new definition can be measured by the two AOAC analysis methods 2009.01 e 2011.25. In Figure 1, it is possible to see that there are overlaps of some techniques, when classical and specific methods are applied and, therefore integrated methods (AOAC 2009.01 e 2011.25) are advantageous because they include all DF fractions.

The official methods of determination of DF are expensive in terms of time consumed, chemical reagents and enzymes of high cost. However these methodologies are appropriate, developed and validated through studies in international laboratories and today have become standard methods of AOAC International. In summary, the official methods simulate human digestion by subjecting the sample to three digestive enzymes, α -amylase, protease and amyloglucosidase, in their respective ideal temperatures and pH. After digestion, tests to determine the protein and ash are carried out for subtraction in the calculation of fibers content. Before this, the complete realization of the analysis requires completion of many steps, which generates a series of systematic errors and concern over the chemical waste generated (Ferreira et al., 2015).

Fourier transform infrared spectroscopy (FTIR) is a relatively new method with newer applications in fermented beverages and wine. It has numerous advantages, including ease of implementation, the small amount of sample

required, the speed and almost complete absence of reagents, besides allowing the individual quantification of compounds such as polysaccharides (Boulet et al., 2007). Ferreira et al. (2015) analyzed DF content in 80 varieties of Brazilian soybean by applying near infrared spectroscopy technique by diffuse reflectance associated with chemometric and showed, through its results, that the method is able to predict the DF content in soybean in a fast, reliable and precise way, and this technique can replace the enzymatic-gravimetric method of delayed determination of fibers. Chylińska et al. (2016) have demonstrated in their studies that determination of the pectin, cellulose and hemicelluloses content of fruits based on FTIR is possible, being a method more feasible, faster and less complicated.

CONCLUSION

Generally, the establishment and improvements observed in the methods developed for DF analysis were based on analytical methods for the isolation of these fibers focusing on their physiological aspects. In this context, several classifications of FA fractions can be found in the scientific literature and these are based on analytical methods that are used. For the purposes of product labeling for consumers, the total value of DF is really important. The study of the relationships between DF and diseases, for example, is performed from data of the individual components of DF and so researchers need updated methodologies. The official methods are able to determine all

components present in the dietary fiber of a sample, which contributes to the elimination of steps in relation to specific methods. Other innovative methods are being studied and applied with a view to shorter time, lower cost, less generation of waste through the use of reagents among other advantages.

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