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An improved method for rapid quantitative analysis of the insoluble dietary fiber in common cereals and some sorts of beans

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ABSTRACT

An improved rapid analysis for determining the content of insoluble dietary fiber (IDF) in common cereals and some sorts of beans is described in this paper. The procedure includes starch gelatinization in water bath for 20 min at 100 °C and 2.5% (w/w) α-amylase hydrolyzed reaction followed by neutral detergent wash and acetone extraction. Compared with 1.5 h for filtration (estimated) and 18 h for the enzymatic hydrolysis required by the typical American Association of Cereal Chemists (AACC) method, the filtration and enzymatic treatment procedures in the improved method was completed within 15 min and 1.5 h, respectively. The length of time for the filtration and the enzymatic hydrolysis for the improved method was significantly shortened from 19.5 h (AACC method) to 1.75 h. In addition, orthogonal array design (OAD) has been applied to optimize parameters of the improved method. The recovery yield of microcrystalline cellulose was 97.75% (w/w), in agreement with the result obtained using the typical AACC method, demonstrating the reliability of the improved method. Furthermore, several common cereals and beans were employed to validate the accuracy and universality of this improved method.

1. Introduction

Dietary fiber is generally defined as a mixture of carbohydrate polymers containing oligosaccharides and polysaccharides, for instance, cellulose, hemicelluloses, resistant starch, gums, pectin and inulin etc (Elleuch et al., 2011). In terms of digestibility in the small intestine, dietary fiber can be categorized into two groups: soluble dietary fiber, for example, pectin and gum, which are digested into carbohydrate and absorbed in the small intestine; and insoluble dietary fiber (IDF) such as cellulose, hemicellulose and lignin etc, which are unable to be digested and are poorly metabolized in the small intestine (Englyst et al., 2007). The latter offers numerous physiological health benefits in humans, including reduction of postprandial blood glucose, pre-prandial cholesterol levels, coronary heart disease, and some sorts of cancer (Mann and Cummings, 2009). Given that health concern motivates most consumers to purchase high-insoluble fiber foods, the analysis of dietary fiber grabs the attention of food scientists. In the literature, the conventional methodology for the analysis of dietary fiber can be classified into three categories: enzymatic-gravimetric method (Elleuch et al., 2008; McCleary et al., 2012; Prosky et al., 1988; Vergara-Valencia et al., 2007); enzymatic-chemical methods (Englyst et al., 1994; Goni et al., 2009; Manas et al., 1994; McCleary and Monaghan, 2002); nonenzymatic-gravimetric method (Southgate et al., 1978). Among the mentioned methods, the enzymatic-gravimetric method is the most suitable for nutrition labeling and quality control purposes since nonenzymatic-gravimetric methods fail to recover a massive portion of dietary fiber and the operation of enzymatic-chemical methods generally requires expensive equipment such as colorimetric or GLC/HPLC (Andrews, 1996).

However, the enzymatic-gravimetric method does suffer from the time-consuming procedure of filtration and the enzymatic hydrolysis unable to meet the current requirements of the food industry. Hence, a dilemma between growing demands for the analysis of insoluble dietary fiber in industry and time-consuming methods in the literature gives momentum to the development of a rapid and robust analysis of insoluble dietary

Abbreviations: AACC, American Association of Cereal Chemists; Disodium EDTA, disodium ethylenediaminetetraacetate dehydrate; Na_2HPO_4, disodium hydrogen phosphate; IDF, insoluble dietary fiber; OAD, orthogonal array design; NaH_2PO_4, sodium dihydrogen phosphate; AOAC, the Association of Official Analytical Chemists.

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fiber. In this report, an improved and rapid method was designed by us, based on the modification of AACC method 32-20 approved by the American Association of Cereal Chemists (AACC, 1999) and the Association of Official Analytical Chemists (AOAC) 985.29 – "total dietary fiber of food samples determine by the enzyme-weighted method" (AOAC, 1992). Further, this improved analytical approach was tested and validated by several common sorts of cereals and beans and the final analytic results have been compared with those using the conventional method (AACC, 1999).

2. Materials and methods

2.1. Materials

Sodium lauryl sulfate (ACS grade); Disodium ethylenediaminetetraacetate dihydrate (Disodium EDTA), (ACS grade); Sodium tetraborate, decahydrate (ACS grade); Disodium hydrogen phosphate (ACS grade); 2-ethoxyethanol (ACS grade); Phosphoric acid (ACS grade); Anhydrous sodium sulfite (ACS grade); Petroleum ether (ACS grade), Sodium dihydrogen phosphate (ACS grade); Acetone (HPLC grade); microcrystalline cellulose (ACS grade); were purchased from Sigma–Aldrich (Shanghai, China). α-amylase (VI-A type, product No. 3880) was obtained from Sigma–Aldrich (St Louis, MO, USA). All cereals and beans were purchased from a local grocery store in Wuhan, China.

Neutral detergent was prepared initially by mixing 18.61 g of $C_{10}H_{14}N_2$ $Na_2O_8 \cdot 2H_2O$ with 6.81 g of $Na_2B_4O_7 \cdot 10H_2O$ in 150 mL of water at 60 °C until completely dissolved. Afterward, 30 g of sodium dodecyl sulfate, 10 mL of 2-ethoxyethanol in 700 mL of hot water and 4.56 g of Na_2HPO_4 in 150 mL of hot water were gently introduced into the mixture in that order. The phosphate buffer solution (0.1 mol/L, pH7.0) was prepared by adding 38.7 mL of disodium hydrogen phosphate (0.1 mol/L Na_2HPO_4) and 61.3 mL of sodium dihydrogen phosphate (0.1 mol/L NaH_2PO_4). 2.5 g of α-amylase was subsequently dissolved into 100 mL of phosphate buffer solution (pH7.0) for further usage.

2.2. Apparatus

The electric oven and incubator were purchased from Bo newsletter industrial Co., LTD medical equipment factory (Shanghai, China). A beaker flask with condensing unit, coarse glass-frit (ASTM 40–60 μm), thermostat water bath, dryer, and filtering device were kindly provided by Glass Instrument Factory (Beijing, China). Grinding apparatus, extracting equipment with condenser and hot plate were obtained from Nanjing Feiqi Industry and Trade Co., LTD (Nanjing, China).

2.3. Analytical methods

2.3.1. Sample preparation

The samples were carefully ground and stored at room temperature. Subsequently, the sample was extracted three times by 25 mL of petroleum ether per g of sample, followed by filtration with 20–30 mesh paper (1 mm).

2.3.2. Sample gelatinization and α-amylase hydrolyzed reaction

One gram filtered sample was weighed into a conical flask with 45 mL of distilled water and incubated in the water bath at 100 °C for 20 min, and then cooled down to room temperature. Five mL of α-amylase solution was gently mixed with the gelatinized sample, and the suspension was stirred at 300 rpm for 90 min at 37 ± 2 °C.

2.3.3. Sample reflux extraction

After the hydrolysis reaction, the sample was transferred to the extraction device with 100 mL of neutral detergent and 0.50 g of anhydrous sodium sulfite at 100 °C for 60 min.

2.3.4. Filtration and acetone extraction

The filtration residue from the previous extraction was removed to a glass sand core filter, and washed by 300 mL of hot water (100 °C) several times. Afterward, the residue was soaked in 20 mL of acetone in a glass sand core filter for 10 min, and then filtered by vacuum. Subsequently, 5 mL of acetone was employed to wash the residue carefully. After storage at 110 °C for 6 h, the glass sand core filter was moved to a desiccator for 1 h and weighed at room temperature.

2.4. Insoluble dietary fiber determination

The content of the insoluble dietary fiber was calculated as follows:

$$\text{IDF}\,\% = (m_2 - m_1)/m$$

IDF: percent of the insoluble dietary fiber, %
m_2: weight of glass filter and residue after drying, g
m_1: weight of glass filter after drying, g
m: weight of original sample, g

2.5. Statistical analysis

Averages and standard deviations (SD) from at least three measurements of each sample were reported. All determinations were performed in duplicate. Data are expressed as mean ± SD. ANOVA and Student's *t* test were employed to analyze the effect of vacuum filtration of enzyme solution on IDF and validation of IDF using the improved method, respectively, at the significance level of p-value <0.05 using the SAS software (SAS Institute Inc, Cary, NC, USA).

3. Results and discussion

3.1. The effect of vacuum filtration of enzyme solution on IDF

Corn flour was selected as the substrate to examine the effect of vacuum filtration of the hydrolysis solutions treated by α-amylase since corn is a typical model containing insoluble dietary fibers (Pandya and Srinivasan, 2012). Vacuum filtration is a time consuming procedure generally involved in the determination of IDF for a majority of methods (AACC, 1999; Goni et al., 2009; McCleary et al., 2010; Vansoest et al., 1991). The purpose of employing vacuum filtration in the determination of IDF is to remove some sorts of proteins solubilized by neutral detergent from samples. However, vacuum filtration for handling some highly viscous samples is a time-consuming procedure. Therefore, it was hypothesized that vacuum filtration could be redundant for the improved method.

As described in the experimental section, 2.5% (w/w) α-amylase was mixed with the gelatinized sample at 300 rpm at 37 ± 2 °C for 90 min. Subsequently, the enzyme solution underwent vacuum filtration at different ratios of 0, 10%, 30%, 50%, 60%, 70%, 80%, respectively. Other procedures followed the protocol in the experimental section. Table 1 showed that there was no significant difference ($P > 0.05$) in the determination of IDF in samples treated with vacuum filtration at different ratios. This result indicates that vacuum filtration can be negligible during the procedure of this