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Physicochemical, microstructural and functional characterization of dietary fibers extracted from lemon, orange and grapefruit seeds press meals

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ABSTRACT

The aim of this study was to characterize dietary fibers extracted from defatted press meals of lemon, orange and grapefruit seeds. After an ultrasound-assisted aqueous extraction, the fibers were investigated for proximate composition, soluble/insoluble fiber contents, color, viscosity, degradation temperature and enthalpy, near infrared (NIR) spectra, X-ray diffraction patterns, scanning electron microscopy (SEM) images, and functional (water and oil holding capacity, swelling capacity) properties. The fibers contain 2.0–3.45 mg/g phytate, 1.36–2.26% ash, 4.95–7.95% soluble fiber, 75.95–82.24% insoluble fiber. NIR spectra showed the presence of cellulose and lignin as main components with corresponding bands. Major peak at 22–23 Å X-ray diffraction also indicated presence of cellulose crystals. Crystallinity values of 33–80% were measured. SEM images showed the microfibrillar structures present in the samples. Water and oil holding capacities (4.79–7.76 g/g and 3.44–4.19 g/g), and swelling capacities (22.44–24.55 mL/g) proved that the fibers are functional, and especially good for oil containing foods. The utilization of these new fiber sources would be important in terms of by-product valorisation. The data presented in this study may provoke further studies and/or applications of these new dietary fibers.

1. Introduction

Dietary fiber has been defined and recognized as an important component of human diet since 1950s. Although there is no universal definition yet, all definitions proclaim that fiber components of the foods are carbohydrate polymers with ten or more monomeric units and are not hydrolysed by the endogenous enzymes in human small intestine. Hence, dietary fiber may include cellulose, hemicellulose, pectic substances, gums, resistant starch, inulin, and other non-carbohydrate components like lignin, polyphenols, waxes, saponin, cutin, phytates and resistant protein. Furthermore, most fiber components may be fermented by gut microflora to some degrees (Elleuch Bedigian, Roiseux, Besbes, Blecker, & Attia, 2011; Fuller, Beck, Salman, & Tapsell, 2016; Tejeda-Ortigoza, Garcia-Amezquita, Serna-Saldivar, & Welti-Chanes, 2016). It is well accepted that dietary fibers provide some health benefits to consumers through enhancement of fecal bulk, stimulation of colonic fermentation, reduction of post-prandial blood glucose and pre-prandial cholesterol level. These physiological effects are also well associated with reduced risk of cardiovascular diseases, cancer, diabetes, respiratory disease, infections, and others (Elleuch et al., 2011; Huang, Xu, Lee, Cho, & Qi, 2015).

Dietary fibers as food ingredients have been in use for various prepared foods. Fiber addition to foods imparts not only health benefits but also some functional properties like increase in water and oil holding capacity, emulsion and foam formation, modification of texture and eating properties, stabilization of structure and extension of shelf-life. Hence, dietary fibers mainly from plant food sources and from food processing by-products have been extracted, characterized and used (Elleuch et al., 2011; O'Shea, Arendt, & Gallagher, 2012; Russo, Bonaccorsi, Inferrera, Dugo, & Mondello, 2015; Tejeda-Ortigoza et al., 2016). Different dietary fibers exhibit diverse compositional and functional properties. Beyond the most commonly utilized cereal and fruit fibers, other non-conventional sources are in search for the diversity of composition and functional properties (Elleuch et al., 2011; O'Shea et al., 2012). Good quality fibers should contain no nutritionally offensive and toxic substances, should be in concentrated and balanced (soluble versus insoluble fractions) quantity, should not have any negative effects on taste, flavor and color, should be compatible with food processing, must have expected physiological benefits, and should be easily available and in adequate price (Larrauri, 1999).

Citrus processing yields peels, seeds and pulps as valuable by-products which can comprise 50% of fresh fruit weight (El-Adawy, Rahma, El-Bedawy, & Gafar, 1999). There are studies about dietary fibers extracted from citrus albedos and flavedos (Chau & Huang, 2003;

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Gorinstein et al., 2001; Figuerola, Hurtado, Estévez, Chiffelle, & Asenjo, 2005; Marin, Soler-Rivas, Benavente-Garcia, Castillo, & Pérez-Alvarez, 2007; Russo et al., 2014, 2015), but there is no study about dietary fibers extracted from citrus seeds. Citrus seeds were analyzed (Russo et al., 2015), and it was found out that they contain substantial amounts of flavonoids, limonoids and dietary fiber, and represent important source for nutraceutical bioactive compounds.

It was discussed that citrus processing wastes are abundant, inexpensive and undervalued bio-sources with potential pollution problem (Russo et al., 2015; Tejeda-Ortigoza et al., 2016). Hence, we started valorisation studies for citrus seeds collected as wastes of juice factories. After cold press oil extraction, we got the defatted citrus seed meals as the subject material of this study. Hence, the aim of this study was to extract dietary fibers from the defatted press meals of lemon, orange and grapefruit seeds, to characterize the extracted dietary fibers for common physico-chemical, microstructural, thermal and functional properties, and to compare the effects of seed treatments (solvent extraction, microwave heating and enzyme treatment) applied prior to cold oil pressing on the dietary fiber properties. Almost all parts of this study are for the first time in the literature and novel, and may provoke further studies and even industrial interests to citrus seed dietary fibers.

2. Materials and methods

2.1. Materials

In this study, the defatted press meals of lemon (*Citrus limon* L.), orange (*Citrus sinensis* L.) and grapefruit (*Citrus paradisi* L.) seeds were used as the starting materials to extract the dietary fibers. At the start of this study, lemon seeds (Kütdiken variety) from Limkon Food Industry and Trade Inc. (Adana, Turkey), orange seeds (Dörtyol variety) from Anadolu Etap Penkon Co. (Mersin, Turkey) and grapefruit seeds (Beyaz variety) from Frigo-Pak Food Co. (Bursa, Turkey) were gifted us. Each seed variety was cold pressed against its treatment group to gain the seed oil and oily press cakes. The control groups of all three seeds were roasted at 150 °C for 30 min in an oven (Inoksan PFE, Bursa, Turkey), then conditioned to 10% moisture, and cold pressed. The treatment group for lemon seeds was prepared as follows: solvent extraction of oil was carried out by first drying the finely ground seeds to 5% moisture, and then hexane extraction of oil (1:2.5 = seed: hexane, w/v) at 45 °C in water bath by mixing 12 h at 140 rpm, and the process was repeated 3 times. The microwave treatment of orange seeds was applied with 360 W energy for 30 min total time in 3 min apply-3 min cease operational mode in a microwave oven (Beko MD 1505, Turkey), before cold pressing. The treatment group of grapefruit seeds was carried out by grinding the seeds and then incubating them with 0.06 U/g seed naringinase (Rham 142) and 0.033 U/g seed hesperidinase (Rham 143) enzymes in 100 mM KH_2PO_4/K_2HPO_4 buffer solution (pH 7.5) at 65 °C for 4 h. After the incubation, the seeds were heated to 150 °C for 30 min to inactivate the enzymes and to adjust the moisture level before cold oil pressing. The prepared seeds were cold pressed with a lab scale press machine (Koçmaksan ESM 3710, İzmir, Turkey, 12 kg seed/h capacity, single head, 2 hp, 1.5 kW power) in two separate batches for the two replicates of the study. The press conditions were 30 rpm screw rotation speed, 10 mm exit dies and max 40 °C exit oil temperature. All collected press meals were first defatted by hexane extraction (1:4, w/v, 2 h, 190 rpm stirring at room temperature) 3 times, and then the wet meals were forced air-oven dried at 60 °C for 1 h, and under hood for overnight. The defatted press meals were ground (Retch Grindomix GM 300, Germany), placed into zipped refrigerator bags and kept at −20 °C until dietary fiber extraction.

The megazyme enzyme (α-amylase, protease and amyloglucosidase) kit (Megazyme International, Bray Co., Wicklow, Ireland) was used for dietary fiber analysis. All chemicals and standards used were of analytical grade and purchased from Merck Co. (Darmstadt, Germany) and Sigma Chem. Co. (St. Louis, USA).

2.2. Dietary fiber extraction

Finely ground and defatted press meal and ultrapure water were mixed at 1:20 (w/w) ratio to start the extraction process. The mixture was first ultraturraxed at 13.500 rpm speed for 5 min (Yellow line D125 basic) to disperse the meal completely in the aqueous medium. This process was repeated for 30 min total time. Then, the dispersion was placed into the ultrasound chamber (Sonics VCX750, Connecticut, USA) and amplitudes of 70% corresponding to 280 W, 20 kHz were applied for 20 min with pulse durations of 5 s on and 25 s off. The temperature of the medium was continuously monitored by a thermocouple, and never exceeded 40 °C. Finally, the mixture was filtered through 100 mesh screen, and the solids remaining on the top of screen were washed with hot (80 °C) water five times before vacuum drying at 50 °C for 3 days. The dried mass was ground to fine particles, and collected as the extracted seed dietary fibers. The name abbreviations used throughout the paper for the extracted fibers are as follows; CPLS-DF: cold pressed lemon seed dietary fiber, SELS-DF: solvent extracted lemon seed dietary fiber, CPOS-DF: cold pressed orange seed dietary fiber, MTOS-DF: microwave treated orange seed dietary fiber, CPGS-DF: cold pressed grapefruit seed dietary fiber, ETGS-DF: enzyme treated grapefruit seed dietary fiber.

2.3. Physicochemical properties

The colors of the dietary fibers were measured by Minolta CR-400 (Osaka, Japan) colorimeter calibrated against white tile on at least 10 random points of samples placed in a petri dish. The dietary fiber dispersions (10%, w/w) were prepared and their pH was adjusted to 7.0 by 1 N HCl or NaOH, before their viscosities were measured at 30 and 60 °C by circulating water bath attached to Brookfield DV II. Pro viscosimeter with Rheocalc software (Brookfield Eng. Lab., Inc., MA, USA) and no. 18 spindle at 10 rpm measurement speed, and apparent viscosities were recorded as centipoise (cP) values.

One g of each fiber sample was dried at 110 °C for 30 min in an Ohaus MB45 moisture analyzer (Switzerland) for moisture content determination. Total ash contents of the fibers were assessed by the Ba 5a-49 method (AOCS, 1984), and presented on dry weight basis (dw).

The phytic acid content of the dietary fiber samples was measured by AOAC method 986.11 (AOAC, 1986). Each dietary fiber sample (2g) were mixed with 40 mL HCl (2.4%, v/v) at 25 °C for 3 h at 150 rpm rate, before filtering the slurry on Whatman no. 1 paper. The filtrate was diluted with 25 mL Na_2EDTA-NaOH solution (1:1, v/v). Dowex® AG 1 × 4 chloride resin (100 − 200 μm) was used to prepare the ion-exchange column, and the column was washed with 15 mL of 0.7 M NaCl solution and 15 mL of water. Then, the diluted filtrate was transferred into the column, and eluted with 15 mL water and 15 mL 0.1 M NaCl, and these eluates were discarded. The final elution was done with 15 mL 0.7 M NaCl, and was collected. Finally, 0.5 mL H_2SO_4 + 3 mL HNO_3 was added to the final eluate and wet burning was applied according to Kjeldahl method. The phytate salts were dissolved in 100 °C water bath after adding 10 mL distilled water. Then, 2 mL molybdate and 1 mL sulfonic acid solutions were added and completed to 25 mL with distilled water to finish the reaction in 15 min. Finally, spectrophotometric absorbance was measured at 640 nm (Agilent 8453 UV–Visible Spectrophotometer, Waldbrann, Germany). Standard curve was prepared with potassium acid phosphate, and the phytate content of the samples (mg P/g) was calculated.

2.4. Soluble and insoluble fiber analysis

The total, insoluble and soluble dietary fiber contents (% dw) of the extracted citrus seed dietary fiber samples were determined by the enzyme-gravimetric method 991.43 (AOAC, 2000) with the commercial enzyme kit Megazyme. The results were also corrected for remaining protein (by Kjeldahl method) and ash content. The total fiber contents