Food Research International 109 (2018) 572–582

Contents lists available at ScienceDirect

Food Research International

journal homepage: www.elsevier.com/locate/foodres

Sapucaia nut (*Lecythis pisonis* Cambess.) flour as a new industrial ingredient: Physicochemical, thermal, and functional properties

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ARTICLE INFO

Keywords:
Defatted nut meal
Brazilian edible nut
Pressurized fluids
Functional properties
Emulsions

ABSTRACT

The aim of this work was to investigate the physicochemical, thermal, and functional properties of partially defatted sapucaia nut (*Lecythis pisonis* Cambess.) flours (PDSF) degreased by subcritical propane (20–60 °C; 20–100 bar) and supercritical CO_2 + ethanol (1:1 w/w) as co-solvent (60 °C; 200 bar) in comparison to the PDSF obtained through Soxhlet extraction with petroleum ether. Under the conditions studied herein, compressed propane has a minor effect on the granules' morphology (average particle size between 22 and 32 µm) or in the physicochemical characteristics of the PDSF. It caused a minimum impact on the nutritional profile of the samples; unlike, the thermogravimetric analysis revealed that there is an influence on the thermal stability of the PDSF. Functional characteristics, such as emulsifying (8–20 m^2/g), foam (6–12%), and high water (0.35–1.38 g/g flour) and oil (0.72–1.57 g/g flour) absorption capacity, were observed in PDSF. Defatted flours were found to be effective in the production of emulsions with structures that showed micrometric-sized droplets (up to 85% droplet size < 15.0 µm) with alleged stability. PDSF is a source of proteins (31–49%) and carbohydrates (17–31%), thus it can be used as an ingredient to produce foodstuff in bakery and confectionery aiming to increase their nutritional value and functional properties.

1. Introduction

Brazil has a wide variety of edible nuts and walnuts, utilized in a range of food segments including the use of raw material (*in natura*), to be used as seasonings, condiments and in the extraction of oils or beverage production. Most nuts are a rich in macro and micronutrients, as well as bioactive compounds. Furthermore, nut intake is directly related to the prevention of cardiovascular diseases and hypertension (Carvalho et al., 2015). Some underutilized Brazilian nuts, as the sapucaia (*Lecythis pisonis* Cambess.), have been studied along the past twenty years. The reports indicate a composition of nutritional importance, besides presenting great potential for the food industry (Demoliner et al., 2018; Denadai et al., 2007; Naozuka, Vieira, Nascimento, & Oliveira, 2011; Teixeira, Ávila, Silveira, Ribani, & Ribani, 2017; Teixeira, Ghazani, Corazza, Marangoni, & Ribani, 2018; Vallilo, Tavares, Pimentel, Badolato, & Inomata, 1998).

The sapucaia nut has a chemical composition which changes according to the place of cultivation and harvesting, and conditions of production, among other factors. The lipid content can reach up to 63% (Vallilo et al., 1998; Vallilo, Tavares, Aued-Pimentel, Campos, & Neto, 1999), and shows a composition rich in unsaturated fatty acids and phenolic compounds with antioxidant characteristics (Teixeira et al., 2017). Presenting low moisture content (~3 to 10%), sapucaia also stands out for its carbohydrate and protein content, which can range from 5 to 11%, and 18 to 29%, respectively. The mineral residue varies around 3%, and the fiber content is ~7% (de Carvalho et al., 2012; de Carvalho, da Costa, de Souza, & Maia, 2008; Vallilo et al., 1999). Extraction of oil from sapucaia nut has been performed from different methods using organic solvents (Teixeira et al., 2017; Vallilo et al., 1999), by cold pressing (Costa & Jorge, 2012; Demoliner et al., 2018), or using green solvents such as compressed propane and supercritical carbon dioxide (Teixeira et al., 2018), but the available studies didn't focus on the defatted residue. Researches featuring de-oiled nut residues such as physic nut (*Jatropha curcas*) (Das et al., 2011) hazelnut (Turan, Capanoglu, & Altay, 2015) and Brazil nut (Carvalho et al., 2015) shows the potential of this type of by-products.

Targeting agroindustry matrices for the production of foodstuffs is crucial for the valorization of the national productive chain. Thereby, the economy is linked to the production of that raw materials, resulting in higher added value. Besides, the indirect reduction in the actions of deforestation occurs as a consequence of the valorization of the vegetable species (Teixeira et al., 2018; Teixeira, Züge, Silveira, Scheer, &

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https://doi.org/10.1016/j.foodres.2018.04.071
Received 14 March 2018; Received in revised form 17 April 2018; Accepted 30 April 2018
Available online 01 May 2018
0963-9969/

Ribani, 2016).

To exemplify, the defatted meal or flour resulting from the extraction of oil from nuts presents a high industrial potential, as a rich source of protein and carbohydrates. Instead of treating them as a residue, they are used as a new product in some companies in Brazil. Additionally, the remaining lipid content in defatted flour is a positive factor, carrying beneficial characteristics of its fatty acids to the defatted meal. These types of flours have proven to show several characteristics of industrial interest, either for use in the production of humans foodstuffs (Bhise, Kaur, & Aggarwal, 2015; Chan, Khong, Iqbal, Mansor, & Ismail, 2013; Huiping Chen et al., 2015; Ganorkar, Patel, Shah, & Rangrej, 2016; Ling, Zhang, Li, & Wang, 2016; Sawada, Venâncio, Toda, & Rodrigues, 2014; Xu, Lui, Luo, & Diosady, 2003), for animal feed (Suprayudi et al., 2016), mushroom cultivation (Pardo-Giménez et al., 2016), or even in the production of adhesives (Zheng et al., 2017). Other products such as beet-sugar pectin (Chen et al., 2016), hemicellulose and cellulose from by-products as sorghum bran, bagasse and biomass (Qiu, Yadav, & Yin, 2017) present functional properties being successfully applied in production of emulsions.

In order to have an adequate use of defatted flour for human consumption, in-depth studies on its characteristics are necessary, mainly to ascertain its functional properties, and discover its benefits and possible damages caused by the degreasing processes (e.g. sub- supercritical fluid extractions), and its applicability limitations for industry. Analytical techniques produce relevant results for industry, including thermal analysis (thermogravimetry), microscopic investigation, and colorimetric techniques, answering the diverse issues related to the studied matrix, guiding a correct application (Guimarães et al., 2012; Teixeira et al., 2016; Turan et al., 2015).

In this contex, no information regarding the functional properties of sapucaia nut defatted flour has been found in the literature. Thus, this work aimed to evaluate the physicochemical composition and functional properties of sapucaia nut flours partially defatted by pressurized fluids (propane and CO_2), as well as the influence of degreasing processes on the characteristics of the obtained products, comparing them with the flour obtained by classic Soxhlet extraction with petroleum ether.

2. Material and methods

2.1. Sapucaia nut samples

The sapucaia nut flour was partially defatted through different degreasing processes presented in details in our previous work (Teixeira et al., 2018), by using three different methods: a) subcritical extraction using propane (L1-L5, 20–60 °C, 20–100 bar); b) supercritical extraction using carbon dioxide (CO_2) + ethanol (1:1, w/w) as co-solvent (LC, 60 °C, 200 bar); c) classical Soxhlet extraction (AOCS, 1997) using petroleum ether (LS, control sample). The conditions for each process are summarized in Table 1. The partially defatted sapucaia nut flour (PDSF) samples were ground with the aid of a knife mill (MA630/1, Marconi Ltda., Brazil) for 30 s, resulting in a fine powder. All samples were vacuum packed in LDPE plastic bags and kept under refrigeration until further analysis.

2.2. Characterization of sapucaia nut and its PDSF

2.2.1. Proximate composition and color analysis

Moisture and ash were determined by thermogravimetry (method described in Section 2.2.2), where moisture was estimated by mass loss between 30 and 150 °C, and the residue remaining at the end of the analysis at 900 °C was considered ashes (Kaspchak et al., 2017). The total N concentration was determined according to the AOCS Ba 4e-93 official method using a LECO FP-528 dry combustion Carbon/Nitrogen analyzer system (LECO, Michigan, USA). The crude protein content was calculated by multiplying the total N content by the factor 5.46. Lipid content was determined by Soxhlet (AOCS, 1997). The total carbohydrate content was estimated by difference. Energetic value was calculated by multiplying the carbohydrate and protein content by 4 kcal/g and that of lipids by 9 kcal/g. The color parameters were measured using the MiniScan XE plus colorimeter (HunterLab, Germany) and expressed in values of the CIE*Lab* color system, where L^* = luminosity, a^* = red/green coordinate, and b^* = yellow/blue coordinate. The Chroma (color saturation or intensity), and Hue angle were obtained through the formulas: Chroma = $[(a^2 + b^2)^{1/2}]$; Hue = [arc tangent (b/a)].

2.2.2. Thermogravimetric analysis

Thermogravimetric analyzes were performed on a TGA 4000 equipment (PerkinElmer Inc. Waltham, USA). Approximately 5 mg of the sample was placed in the platinum pan and then positioned in the oven where it was heated from 30 to 900 °C (10 °C/min) in a synthetic air atmosphere (50 mL/min flow). Data with sample weight changes were obtained and analyzed using Pyris™ software. The thermogravimetric curve (TG) and the derivative thermogravimetric (DTG) were analyzed with the software Origin 8.6 (OriginLab, Massachusetts, USA). The thermal stability was measured from the extrapolation of the initial temperature of the first thermal decomposition event of the respective TG curves. The initial and final temperatures of the respective DTG peaks were used as temperature limits for the instrument data analysis software.

2.2.3. Scanning electron microscopy

Scanning electron microscopy (SEM) analysis was performed using a JEOL JSM 6360-LV microscope (Jeol Company, Tokyo, Japan). Sapucaia nut and PDSF fine powder samples were fixed on copper supports using a double-sided adhesive tape and then covered with gold (Au) coating. The micrographs were obtained under vacuum and 15 kV of voltage acceleration with a magnification of 1700 ×. The area of the granules was calculated using ImageJ 1.51 s software (ImageJ for Windows).

2.2.4. X-ray diffractograms (XRD)

The X-ray diffraction patterns of the sapucaia nut and PDSF fine powder samples were investigated using a D8-Advance X-ray diffractometer (Bruker, USA) at 25 °C, employing Cu Kα radiation (λ = 1.5406 Å) from 5° to 60° (2θ), at the flow rate of 2°/min, and a step size of 0.060°.

2.2.5. Infrared spectroscopy

Fourier transform infrared spectroscopy analysis in the diffuse reflectance mode (DRIFTS) was performed using a Vertex-70 spectrometer (Bruker, USA) with a diffuse reflectance accessory at 25 °C. Data were recorded in the range of 500 to 4000 cm^{-1} wavenumbers with a spectral resolution of 4 cm^{-1}, and 1024 scans. The reflectance spectra were analyzed after the transformation of the percentage of reflectance into absorbance [log10 (1/Reflectance)] (Carioca & Ferreira, 2011).

2.3. Functional properties of the PDSF

2.3.1. Ultraviolet spectra

For each sample, a solution containing 1 mg/mL of the PDSF was prepared in a 50-mL flask, slightly shaken, and then a 2-mL aliquot was transferred to Eppendorf and centrifuged (Heraeus Fresco 21, Fisher Scientific) at 5000 × *g* for 5 min. The supernatant was then used to obtain the UV–Vis spectrum (UV-1800, Shimadzu, Kyoto, Japan) in the range of 190 to 400 nm.

2.3.2. Turbidity of the dispersions

Dispersions containing 1% (w/v) PDSF were vortexed for 2 min, and then an aliquot was transferred to a quartz cuvette, and the absorbance measured in a Spectro 3000 W spectrophotometer (Scientific Mars,