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Effect of pulsed electric fields and high voltage electrical discharges on polyphenol and protein extraction from sesame cake

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ABSTRACT

The study evaluates the effect of pulsed electric fields (PEF) and high voltage electrical discharges (HVED) on the extraction of sesame cake compounds. Different energy inputs were applied and, afterwards, PEF and HVED were used as pre-treatments to diffusion. Diffusion kinetics were evaluated in diverse ethanol concentrations and temperatures. The extracts were analyzed for polyphenols, lignans and proteins. According to the results, the disintegration index and the polyphenol and protein contents increased with the energy inputs, until 83 kJ/kg was reached. During diffusion, both pre-treatments improved extraction yields of polyphenols and lignans through different solvents. For diffusion in different temperatures, PEF and HVED had a significant positive effect on the extraction of proteins and polyphenols when compared to the control sample. Results show that the use of PEF or HVED can reduce the use of organic solvents and the need for high temperatures to improve diffusion.
Industrial relevance: There is an increasing demand by the industry for the development of extraction procedures that reduce or eliminate the need for organic solvents and high temperatures. Also, food producers are aiming to find new application for by-products from the industry. PEF and HVED assisted extraction damages the product cells, increasing diffusion from the product to the solvent. The concentration of polyphenols and protein in the solvent after PEF and HVED treatments shows that the use of these techniques is relevant for an industrial use, since solvent amount and extraction time and temperature can be reduced.

1. Introduction

Sesame (*Sesamum indicum L.*) is an oilseed, composed of, approximately, 50% lipid and 20% protein. This seed is considered as a valuable source of nutrients and is an important crop around the world due to its high oil content (Beltrão, Souza, & Pereira, 2001). Sesame seed cake is a solid product obtained after the oil removal from sesame seed, usually by cold pressing. The cake obtained from sesame is composed approximately of 35.6% protein, 7.6% crude fiber and 11.8% ash and has an 83.2% dry matter content (Ramachandran, Singh, Larroche, Soccol, & Pandey, 2007). The production of sesame cake is estimated to be very high, considering that worldwide sesame oil production was of 1 million tons (FAO, 2011). In the oil industry, sesame seed cake and other cakes are considered to be by-products, being commonly used as cattle feed in several producing countries. However, with new technologies these residues can be recovered and value added to be a source of nutraceuticals, proteins and fibers.

Sesame seed is rich in lignans, which are antioxidants and one of the most important functional compounds of this seed. Sesame cake has been discovered to be rich in lignan glucosides, which are hydrophilic antioxidants and thus, are not extracted with the oil. Among the glucosides, generally, sesaminol triglucoside is the major component, followed by sesaminol diglucoside (Moazzami, Andersson, & Kamal-Eldin, 2006a,b). Studies regarding the physiological activities of sesame cake have shown that this product can act on the prevention of obesity and hyperglycemia (Bigoniya, Nishad, & Singh, 2012) and reduce cholesterol levels (Visavadiya & Narasimhacharya, 2008) among other effects. Other important aspect of this cake is the high protein content, which is of very important nutritional value. The cake proteins can be extracted and purified, for the obtention of a protein isolate or the cake can be directly used as an ingredient. The aminoacid profile of sesame proteins shows that it is rich in methionine, tryptophan and cysteine, which are frequently limiting amino acids in vegetables. Due to this profile, sesame protein has been studied in combination with other vegetable proteins such as soy, corn, beans and peanuts, to increase their nutritional value (Beltrão et al., 2001; Hwang, 2005; Ramachandran et al., 2007).

Conventional extraction methods of compounds from vegetable matrixes involve mechanical disintegration and/or the use of strong organic solvents. The efficiency of these methods depends on the degree

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of permeabilization of the cell membrane. Recently, several techniques have been studied with the goal of reducing cost, energy consumption and solvent use. Among them is the use of pulsed electric fields (PEF) (Boussetta, Vorobiev, Le, Cordin-Falcimaigne, & Lanoisellé, 2012; Loginova, Lebovka, & Vorobiev, 2011; Vorobiev & Lebovka, 2006) and high voltage electrical discharges (HVED) (Boussetta et al., 2013; Boussetta & Vorobiev, 2014). There is an increasing interest from the industry in the application of PEF and HVED. Extraction assisted by PEF can enable a cold diffusion, based on the transformation or rupture of cell membranes when submitted to an external electric field, increasing the electrical conductivity and the permeability of intracellular material (Vorobiev & Lebovka, 2011; Zimmermann, Pilwat, & Riemann, 1974). HVED causes a more extensive damage to the product, affecting cell walls as well as membranes. This technology is based on the phenomenon of electrical breakdown in water, which induces physical (e.g. shock waves) and chemical (e.g. formation of O_3) processes. These phenomena affect the cell, enhancing the release of intracellular components (Boussetta, Soichi, Lanoisellé, & Vorobiev, 2014; Gros, Lanoiselle, & Vorobiev, 2003).

Most of the studies concerning PEF and HVED focus on the application of these technologies on fruits and vegetables for the extraction of polyphenols and sugar. Only a small number of works have been performed using oilseeds (Boussetta, Turk, et al., 2013; Boussetta et al., 2014; Grémy-Gros, Lanoisellé, & Vorobiev, 2008; Gros et al., 2003) and, to the best of our knowledge, no work has been performed using these technologies on sesame seed or cake. The goal of this study was to evaluate the effects of different variables in order to enhance extraction of polyphenols, lignans and proteins from sesame cake through the application of PEF and HVED. The effects of the treatment energy input and diffusion parameters (solvent and temperature) were investigated. Moreover, the mass diffusivity of the solutes was determined for comparison between treatments.

2. Materials and methods

2.1. Materials

Sesame (*S. indicum L.*) seed cake was kindly provided by the company Vital Âtman (São Paulo, Brazil). The seeds, used for oil production, were cultivated in Brazil and cropped in 2010. The cake was obtained after cold pressing of the seeds using a screw press and it has a cylinder shape with a diameter of approximately 1 cm. Before treatments the cylinders were cut in pieces of 1 cm length. The Folin–Ciocalteau and Bradford reagents and the standards (gallic acid (CAS 149-91-7), bovine serum albumin (BSA, CAS 9048-46-8), sesamin (CAS 607-80-7) and sesamol (CAS 533-31-3) were purchased from Sigma Aldrich. HPLC grade methanol from Vetec (Brazil) and Mili-Q water (Milipore, France) were used for the chromatographic analysis.

2.2. PEF and HVED treatments

The apparatus used for the application of the electrical treatments consists of a pulsed high voltage power supply (Tomsk Polytechnic University, Tomsk, Russia) and a treatment chamber of 1 L capacity. For PEF application the chamber was equipped with two parallel disc electrodes (11 cm of diameter), placed with a distance of 3 cm, and for HVED the chamber was set with one disc electrode at the bottom (3.5 cm of diameter) and one needle electrode at the top. The needle and the disk electrode were 5 mm distant. Each pulse or discharge performed by the equipment provides 40 kV energy input; therefore the electric field for PEF was 13.3 kV/cm. Pulse or discharge duration was 10 μs and frequency was 0.5 Hz (2 s between pulses or discharges), which is imposed by the generator. This setup is described in more detail by Boussetta et al. (2012).

Different energy inputs were evaluated to choose the most suitable pre-treatment for diffusion. The cake (35 g) and distilled water (350 g) were added to the treatment chamber and up to 700 pulses were applied. The corresponding treatment time varied from 1 to 7 ms and the holding time ranged from 4 to 28 min. The energy input (W) can be calculated using Eq. (1).

$$W(kJ/kg) = \frac{E_{pulse} \times n}{mass} \quad (1)$$

In Eq. (1) E_{pulse} is the energy input of one pulse, which, in the equipment used, was of 160 J, n is the total number of pulses applied and *mass* represents the total mass inside the cell, water and product. In this work, the treatment energy input varied from 42 to 291 kJ/kg. Samples (1 mL) were taken between series of 100 pulses or electrical discharges for total polyphenol and protein analysis and the electrical conductivity was measured using a conductivity meter (*Inolab*, Level 1 model, Germany). Moreover, after appliance of each train of 100 pulses (40 KJ/kg), the system was interrupted and the sample was left to cool for around 5 min.

To determine the treatment efficiency, the conductivity disintegration index (Z_C) was calculated using Eq. (2). In this equation σ is the electrical conductivity measured after each train of pulses, σ_i is the conductivity of the intact tissue at the same contact time and σ_d is the conductivity of the totally disintegrated tissue. For the conductivity of the intact cake, a control was prepared by keeping the sample without agitation for the total contact time and using the same solvent to solid ratio, 10 *w*/*w*. Electrical conductivity of the disintegrated cake was obtained by grinding the cake using a laboratory scale coffee grinder (*SEB*, France) and agitating the sample for 2 h in distilled water (10 *w*/*w*).

$$Z_C = (\sigma - \sigma_i)/(\sigma_d - \sigma_i) \quad (2)$$

Using Eq. (2) it was possible to observe how disintegration varies with different energy inputs. The best pre-treatment energy input was chosen for PEF and HVED based on the maximum disintegration index observed and, also, on the amount of polyphenols and proteins found after each treatment. Then, the selected energy input was applied before proceeding to the diffusion experiments. For the pre-treatment, the treatment chamber was filled with 35 g of sample and 300 g of distilled water at different temperatures (20, 40 and 60 °C). The grinding treatment was also evaluated for comparison with the electrical treatments, after 60 min of extraction in water at 20 °C (35 *w*/*w*).

2.3. Solid–liquid extraction

Diffusion kinetics were performed using different solvents and temperatures. After the PEF or HVED treatments, a supplementary amount (400 g) of water, or a combination of water and ethanol, was added to the mixture. The combination was chosen in order to have a final ethanol content of 10, 30 or 50%. The final liquid-to-solid ratio (*w*/*w*) was 20; this ratio was suitable to maintain a homogenous mixture for the extraction. The diffusion experiments were carried out in an Erlenmeyer vessel. The vessels were placed in an incubator shaker (*Infors Sarl*, Aerotron, France) and agitated for 1 h at 150 rpm. The shaker was set to the extraction temperature (20, 40 or 60 °C) and solvent was kept inside for 20 min before the diffusion experiments to reach the desired temperature. Samples (4 mL) were collected at different moments for total polyphenol, lignan and protein analysis. Prior to analysis the samples were centrifuged (*MiniSpin*, Eppendorf, Germany) at 12.100 ×*g* during 5 min. For control experiments, the same protocol was used but no pre-treatment was applied.

2.4. Analysis of the extracts

All samples were centrifuged (*Eppendorf*, MiniSpin plus, Germany) after collected and stored at −18 °C until analysis. Samples collected after HVED treatments were filtered through a 0.45 μm membrane