Separation and Purification Technology 122 (2014) 506–514

Contents lists available at ScienceDirect

Separation and Purification Technology

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

Optimization of phenolics extraction from sesame seed cake

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ARTICLE INFO

Article history:
Received 7 June 2013
Received in revised form 19 November 2013
Accepted 24 November 2013
Available online 11 December 2013

Keywords:
Sesame
Lignan glucosides
Extraction
Optimization

ABSTRACT

Sesame seed cake is considered to be a by-product of the oil industry, being commonly used as cattle feed in several producing countries. However, this residue can be recovered and value added. The main phenolics found in sesame seed are lignans, which are important bioactive compounds of this seed. The main lignans in defatted sesame were identified as sesaminol triglucoside, sesamolinol diglucoside and sesaminol diglucoside. The objective of this study was to optimize the extraction of total phenolics and lignans from sesame seed cake. A second-order polynomial model was set up to predict the responses using response surface methodology (RSM). The independent variables analyzed were temperature (25–90 °C), solid/solvent ratio (0.03–0.11 g/mL) and ethanol concentration (0–95%). Response variables were the concentrations of total phenolics (TP), sesamin (SES), sesaminol triglucoside (ST) and antioxidant activity (ABTS). The extracted amount of TP ranged between 129.7 and 355.3 mg GAE/100 g, SES from 3.2 to 25.7 mg SES/100 g, ST from 208.1 to 537.5 mg SE/100 g and the ABTS of the extracts was between 8460 and 24311 μM TE. Among the analyzed compounds, ST is presented in the highest quantity in sesame cake. Solid to liquid ratio and ethanol concentration were the most important factors affecting extraction, whereas temperature showed reduced influence. The model obtained to describe the effect of these factors on the extraction of ST was satisfactorily validated.

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1. Introduction

Sesame (*Sesamum indicum* L.) is an oilseed that belongs to the pedaliaceae family. The sesame seed is an important crop around the world due to its high oil content, being the ninth most cultivated oilseed [1,2]. World production of sesame in 2011 was 4.092 million ton, with a harvested area of 6.628 million ha. The main producing countries of this seed are China, India and Myanmar [3]. Sesame seed cake is the product obtained after oil is removed from sesame, usually by cold pressing. The cake obtained from sesame is composed by 35.6% protein, 7.6% crude fiber and 11.8% ash and has 83.2% of dry matter [4]. The production of sesame cake is estimated to be very high, considering that 70% of these seeds are cultivated for oil production [1].

Sesame seed cake is considered to be a by-product of the oil industry, being commonly used as cattle feed in several producing countries. However, this residue can be recovered and value added. The main phenolics found in sesame seed are lignans, which are physiologically active and are important functional compound of this seed [5–10]. Studies involving sesame lignans have been done for a long time. The main oil soluble lignans in sesame are sesamin and sesamolin, while sesamol and sesaminol are found in the seed [1,11–13]. More recently, the presence of high amounts of lignan glucosides were found in sesame cake; these glucosides can be considered as hydrophilic antioxidants. Among them, are sesaminol, pinoresinol and sesamolinol glucosides [14,15]. The main lignans in defatted sesame were identified as sesaminol triglucoside, sesamolinol diglucoside and sesaminol diglucoside. These compounds should be considered when breeding new sesame seeds or for any potential incorporation of the seeds into functional foods [16,17]. The amount of lignans in sesame seeds has been reported to be of 0.63%, which makes them a rich source of lignans [18]. Sesame seeds and flaxseeds are considered to be the richest sources of lignans of all plant foods [19,20]. Sesame lignans have demonstrated several health benefits in disease treatment and prevention, being considered bioactive compounds [21]. Studies performed with sesame seed cake or defatted sesame flour show that this product can reduce susceptibility to oxidative stress [14], prevent obesity and hyperglycemia [22], reduce cholesterol levels [23], among other effects [24,25].

The extraction and purification of antioxidants from natural sources is of high interest, especially from residues. These bioactive substances are used in functional foods, food additives, nutraceuticals, pharmaceuticals and cosmetic industries [26]. The extraction yield is dependent on several parameters; solvent and extraction method [27], extraction time and temperature [27–30] and solid to solvent ratio [31], among others. For this reason, an appropriate

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http://dx.doi.org/10.1016/j.seppur.2013.11.036

choice of these variables may significantly enhance extraction efficiency. It is important to consider that, the role of each factor in the extraction process is not always obvious. Each natural product has a different composition and structure and this will interact with the chemical characteristics of the solvent in a diverse way [32]. This variability reinforces the importance of researching the extraction process for different food matrixes.

To the best of our knowledge, very little research has been published on the evaluation of the influence of different parameters on lignans extraction from oilseeds [17,33–35] and most of the referenced works are aimed to extract flaxseed lignans. The objective of this study was to optimize the extraction of total phenolics and lignans from sesame seed cake. A second-order polynomial model was set up to predict the responses using response surface methodology (RSM). The effects and interactions among extraction temperature, solid/liquid ratio and ethanol concentration were investigated by the response surface analysis.

2. Materials and methods

2.1. Plant material

Sesame (*S. indicum* L.) seed cake was kindly provided by the company *Vital Âtman* (São Paulo, Brazil). The seeds for cake production were cultivated in Brazil, cropped in 2010, and the cake was obtained after cold pressing of the seeds. The sample was ground to powder using a stainless steel blender and passed through a 2.36 mm sieve. Prior to the extraction, a defatting process was carried out using a fat extraction apparatus (*Tecnal*, model TE-044, Brazil). The cake was immersed in hexane for 1 h, then suspended and submitted to solvent recirculation for 2 h, followed by air-drying. After the complete oil removal, the moisture content of the cake was determined according to the gravimetric method [36]. This defatted cake was used for extractions, thus results are presented as mg per 100 g of defatted cake.

2.2. Chemicals

Ethanol 95% and the Folin–Ciocalteau reagent were obtained from (Vetec São Paulo, Brazil). HPLC grade methanol was used for the chromatographic analysis Vetec (São Paulo, Brazil). Gallic acid was purchased from Dinâmica (Diadema, Brazil) and ABTS, as well as sesamin (CAS 607-80-7), sesamol (CAS 533-31-3) and Trolox (CAS 53188-07-1) standards, were purchased from Sigma Aldrich (St. Louis, USA).

2.3. Extraction procedure

The extraction process was performed in a Pyrex glass vessel (7.5 cm external diameter and 10.0 cm height) equipped with a water jacket. Using a water bath (*Lauda*, modelo TYP T, Alemanha), hot water was passed through the jacket for the duration of the experiments in the desired temperature. During extraction, the glass cell was placed over a magnetic stirrer (Fisatom, model 706A, Brazil) and attached to a condenser, which was connected to a water bath (Lauda, model RM 12, Germany) maintained at 5 °C. According to the experimental design, a certain volume of solvent containing a pre-determined amount of ethanol was added to the glass cell and, after, the sesame seed cake (approximately 1.0 g) was inserted. The mixture was agitated and extractions were carried out for 40 min. To set this time, previous experiments were performed with the defatted cake. Contact time was evaluated up to 2 h for total phenolics and the extraction yield did not significantly increase after 40 min (data not shown). The test was performed at 58 °C, using 0.11 g/mL and ethanol 48% as a solvent. Subsequent to the extraction, samples were centrifuged (Cientec, model CT 5000R, Brazil) for 15 min, at 4757*g* and 25 °C. The supernatant was collected and the volume of extract was adjusted according to the initial ratio. Finally, extracts were cooled until analysis. Preceding the HPLC analysis, the extracts were filtered through a 0.45 μm membrane filter.

2.4. Experimental design and statistical data analysis

A central composite design (CCD) and response surface methodology (RSM) were used to optimize experimental conditions for the extraction of total phenolics, lignans and antioxidants from sesame seed cake and to analyze the influence of three independent variables. The independent variables chosen were temperature (X_1), solid to solvent ratio (X_2) and ethanol concentration (X_3); the levels in which the variables were analyzed are shown in Table 1.

To determine the influence of the selected parameters on the response variables, a 2^3 full factorial design with 6 axial and 5 central points was used. The complete set of experiments is presented in Table 2. The order in which the experiments were performed was randomized to minimize error due to extraneous factors.

Multiple linear regression analysis was performed using software Statistica® 7 (*Statsoft Inc.*, Tulsa, Estados Unidos). Experimental data were fitted to the second-order polynomial model presented in Eq. (1), and regression coefficients (β's) were obtained.

$$Y = \beta_0 + \sum_{i=1}^{3}\beta_i X_i + \sum_{i=1}^{3}\beta_{ii} X_{ii}^2 + \sum_{i=1}^{2}\sum_{j=i+1}^{3}\beta_{ij} X_i X_j \quad (1)$$

where Y represents the dependent variables (estimated responses) and β's represent the equation coefficients. An analysis of variance (ANOVA) was performed for each response and the p-values, at a probability of 0.05, indicated whether the terms were significant or not. Terms that were not significant were removed from the final models. The significance of the regression was also evaluated using ANOVA. To verify the adequacy of the models, the experimental data were compared to the values predicted by the regression models and the average error was determined using Eq. 2:

$$E(\%) = \frac{100}{n}\sum_{i=1}^{n}\frac{|y_{\exp} - y_{pred}|}{y_{\exp}} \quad (2)$$

where E is the average error, n is the number of experimental data points, y_{exp} is the experimental value and y_{pred} is the value predicted by the model.

2.5. Determination of the optimum conditions and validation of the model

The values of the independent variables that maximize the extraction yield for each response variable were determined through Matlab® 5.3 software using the *fmincon* function that uses Sequential Quadratic Programming (SQP) method and the experimental models obtained in this work. The verification of the validity and adequacy of the predictive extraction model was

Table 1
Coded and uncoded levels of the independent variables.

Independent variables	Code units	Code levels				
		−1.68	−1	0	1	1.68
Temperature (°C)	X_1	25	38	58	77	90
Solid to solvent ratio (g/mL)	X_2	0.03	0.05	0.07	0.09	0.11
Ethanol concentration (%)	X_3	0	19.2	47.5	75.8	95

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