Journal of Food Engineering 199 (2017) 1–8

Contents lists available at ScienceDirect

Journal of Food Engineering

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

Optimization of autohydrolysis conditions to extract antioxidant phenolic compounds from spent coffee grounds

Lina F. Ballesteros [a], Mónica J. Ramirez [b], Carlos E. Orrego [b], José A. Teixeira [a], Solange I. Mussatto [c, *]

[a] Centre of Biological Engineering, University of Minho, Campus Gualtar, 4710-057, Braga, Portugal
[b] Instituto de Biotecnología y Agroindustria, Universidad Nacional de Colombia sede Manizales, Km 7 via al Magdalena, Campus La Nubia, Manizales, Colombia
[c] Novo Nordisk Foundation Center for Biosustainability, Technical University of Denmark, Kemitorvet, Building 220, 2800, Kongens Lyngby, Denmark

ARTICLE INFO

Article history:
Received 21 June 2016
Received in revised form
6 November 2016
Accepted 19 November 2016
Available online 21 November 2016

Keywords:
Autohydrolysis
Spent coffee grounds
Phenolic compounds
Antioxidant activity
Extraction yield

ABSTRACT

Autohydrolysis, which is an eco-friendly technology that employs only water as extraction solvent, was used to extract antioxidant phenolic compounds from spent coffee grounds (SCG). Experimental assays were carried out using different temperatures (160–200 °C), liquid/solid ratios (5–15 ml/g SCG) and extraction times (10–50 min) in order to determine the conditions that maximize the extraction results. The optimum conditions to produce extracts with high content of phenolic compounds (40.36 mg GAE/g SCG) and high antioxidant activity (FRAP = 69.50 mg Fe(II)/g SCG, DPPH = 28.15 mg TE/g SCG, ABTS = 31.46 mg TE/g SCG, and TAA = 66.21 mg α-TOC/g SCG) consisted in using 15 ml water/g SCG, at 200 °C during 50 min. Apart from being a green technology, autohydrolysis under optimized conditions was demonstrated to be an efficient method to extract antioxidant phenolic compounds from SCG.

1. Introduction

Agro-industrial residues often contain high added-value substances that can be extracted by designing a proper bioprocess and exploited in the food, chemical, cosmetic and pharmaceutical industries. Spent coffee grounds (SCG), for instance, is the main waste of the coffee industry, obtained during the processing of roasted coffee powder with hot water or steam to prepare instant coffee. This residue is generated in large quantities around the world (approx. 6,000,000 tons/year) (Mussatto et al., 2011a) and, even though some researches have revealed functional potential of different compounds found in SCG such as polysaccharides, proteins, phenolic compounds, minerals, among others, this residue still has not been used as raw material in industrial processes.

Nowadays, phenolic compounds (PC) have attracted great interest due to their enormous benefits for human health. Some researches have shown that the potential of PC is related to their antioxidant activity, protecting against chronic-degenerative diseases such as cancer, cardiovascular diseases, neurodegenerative diseases and diabetes mellitus (Jiménez et al., 2008; Martins et al., 2011; Mussatto, 2015; Prasad et al., 2011). However, their properties are not limited to the antioxidant activity, but they can also present antiallergenic, antimicrobial and/or anti-inflammatory effects (Farah and Donangelo, 2006; Martins et al., 2011; Mussatto, 2015). Additionally, PC improve the organoleptic properties of vegetable origin food, and can also be used as raw material in the development of functional food or as natural preservatives against food degradation (Ballesteros et al., 2014b; Rodríguez-Meizoso et al., 2010).

A great variety of techniques can be used for recovering antioxidant PC from agro-industrial residues and natural resources, including solid–liquid extraction using organic solvents, autohydrolysis, ultrasound-assisted extraction, microwave-assisted extraction, among others (Ballesteros et al., 2014b; Cortazar et al., 2005; Markom et al., 2007; Mussatto, 2015). Recently, SCG have been studied as a natural source of PC (Murthy and Naidu, 2012; Mussatto et al., 2011b; Panusa et al., 2013; Zuorro and Lavecchia, 2012), and the ability of a conventional solid-liquid extraction method to recover PC from SCG using organic solvents such as ethanol (Panusa et al., 2013; Zuorro and Lavecchia, 2012), methanol

* Corresponding author.
E-mail addresses: smussatto@biosustain.dtu.dk, solangemussatto@hotmail.com (S.I. Mussatto).

http://dx.doi.org/10.1016/j.jfoodeng.2016.11.014
0260-8774/

(Mussatto et al., 2011b) and isopropanol (Murthy and Naidu, 2012) has also been demonstrated. However, there is a necessity of evaluating and identifying more eco-friendly methodologies that do not require the use of organic solvents and may enhance the extracts compatibility for the food industry and enable their use as added-value constituent for different applications. Among these techniques, autohydrolysis could be an interesting alternative for the recovery of antioxidant PC from SCG since it does not require organic solvents for the reaction, but only water, being able to generate a slightly acidic media due to the partial cleavage of acetyl groups existing in the material structure (Conde and Mussatto, 2016; Nabarlatz et al., 2007). Additionally, autohydrolysis process offers several advantages such as elimination of corrosive problems in the equipment due to mild pH of reaction media, reduction of operational costs since no further neutralization is needed, and mild operational conditions for selective degradation of the biomass (Carvalheiro et al., 2004; Conde and Mussatto, 2016).

In a previous study, autohydrolysis under mild reaction conditions was demonstrated to be a technology with great potential to recover PC from SCG (Conde and Mussatto, 2016). However, the conditions that maximize the extraction of these compounds from SCG were not established yet, and it is well-know that the efficiency of this extraction process is affected by the variables used for reaction, such as the solvent/solid ratio, time, temperature, particle size of the solid matrix, among others. Thus, it is very important to optimize the extraction conditions in order to maximize the extraction efficiency. Optimizing the process conditions is also important because allows a more suitable and complete exploitation of the feedstock, saving time, manpower, and making the process less expensive, reliable, cleaner and attractive to be implemented at industrial scale. Taking these facts into account, the aim of the present study was to optimize the process conditions to extract antioxidant PC from SCG by using the eco-friendly technique of autohydrolysis. Extractions were performed using different temperatures, liquid/solid ratios and reaction times and the effects of these operational variables on the extraction results were verified. Finally, the conditions able to produce a phenolic rich extract with high antioxidant activity were determined.

2. Materials and methods

2.1. Raw material and chemicals

Spent coffee grounds (SCG), which were derived from mixtures of Arabica and Robusta coffee varieties imported from different countries including Brazil, Colombia, Timor and Angola, among others, were supplied by the Portuguese coffee industry NovaDelta-Comércio e Indústria de Cafés S.A. (Campo Maior, Portugal). The material was dried in an oven at 60 °C until 5% moisture content and stored for further extractions.

All the chemicals used were analytical grade, purchased from Sigma–Aldrich (Chemie GmbH, Steinheim, Germany), Panreac Química (Barcelona, Spain) and Fisher Scientific (Leicestershire, UK). Ultrapure water from a Milli-Q System (Millipore Inc., USA) was used.

2.2. Autohydrolysis process

Autohydrolysis assays were performed under different conditions of temperature (160–200 °C), liquid/solid ratio (5–15 ml water/g SCG) and extraction time (10–50 min), which were combined according to a 2^3 central composite design. For the reactions, ultrapure water and SCG were added into 160 ml cylindrical stainless steel reactors (Parr Instruments Company, Illinois, USA), which were duly closed and placed into an oil-bath with open heating circulator and temperature control (Julabo, Labortechnik GmbH, Seelbach, Germany). The reactors were maintained in the bath at the desired temperature, during the time required for each reaction. Then, they were removed and immediately cooled down in an ice-bath during 10 min to stop the reaction. The total content of each reactor was centrifuged (2500g, 20 min) and the supernatant (SCG extract) was filtered through 0.22 μm filters and stored at −20 °C until further analyses. The volume of extract recovered after each extraction was quantified and used to calculate the extraction yield, which was expressed as g recovered extract per 100 g SCG.

2.3. Analytical methodology

2.3.1. Phenolic compounds

The total content of phenolic compounds (PC) in SCG extracts was estimated by using the Folin-Ciocalteu reagent method adapted to a 96-well microplate, as previously described (Ballesteros et al., 2014a). The total content of PC was expressed as milligram of gallic acid equivalent per g of dry weight material (mg GAE/g SCG).

2.3.2. Antioxidant activity

The antioxidant activity of SCG extracts was determined by four different methods, namely the ferric reducing antioxidant power (FRAP) assay, DPPH radical scavenging activity assay, the radical cation decolorization (ABTS) assay, and total antioxidant activity (TAA), assay, as described by Ballesteros et al. (2015, 2014a). FRAP values were expressed as mg of ferrous equivalent per g of dry weight material (mg Fe (II)/g SCG). DPPH and ABTS data were plotted as a function of antioxidant concentration to obtain DPPH and ABTS inhibition concentration at 50% (IC_{50}). The IC_{50} values were expressed as mg of Trolox equivalent per g of dry weight material (mg TE/g SCG). TAA was expressed as milligrams of α-tocopherol equivalent per g of dry weight material (mg TOC/g SCG).

2.3.3. Flavonoids

The total content of flavonoids in SCG extracts was estimated by colorimetric assay as previously described (Ballesteros et al., 2014a), being the results expressed as milligram quercetin equivalent per dry weight material (mg QE/g SCG).

2.3.4. Determination of other compounds in SCG extracts

Chlorogenic acid, furfural and hydroxymethylfurfural were analyzed by high performance liquid chromatography (HPLC) (Mussatto et al., 2011b). The concentration of these components was determined from standard curves made with known concentrations of each compound. The response of the UV detector was recorded and integrated using the D-7000 HPLC System Manager software (Hitachi).

2.4. Statistical analysis

The influence of the variables temperature, liquid/solid ratio and extraction time on the recovery of antioxidant PC by autohydrolysis of SCG was investigated through a 2^3 central composite design. The real and coded values of the variables used in the experimental design are given in Table 1. Statistical significance of the variables was determined at 5% probability level ($p < 0.05$). The data obtained from the design were fitted to second order polynomial equations, and the models were simplified by elimination of statistically insignificant terms. Statistical significance of the regression coefficients was determined by Student's *t*-test, and the proportion of variance explained by the models were given by the multiple coefficient of determination, R^2. Statistical analysis of the data and the determination of the conditions able to maximize the extraction