Industrial Crops and Products 53 (2014) 350–357

Contents lists available at ScienceDirect

Industrial Crops and Products

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

Optimization of antioxidants extraction from coffee silverskin, a roasting by-product, having in view a sustainable process

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ARTICLE INFO

Article history:
Received 19 August 2013
Received in revised form
13 December 2013
Accepted 2 January 2014

Keywords:
Coffee silverskin
By-products valorization
Extracts
Bioactive compounds
Phenolics
Antioxidant activity

ABSTRACT

Coffee silverskin is a major roasting by-product that could be valued as a source of antioxidant compounds. The effect of the major variables (solvent polarity, temperature and extraction time) affecting the extraction yields of bioactive compounds and antioxidant activity of silverskin extracts was evaluated. The extracts composition varied significantly with the extraction conditions used. A factorial experimental design showed that the use of a hydroalcoholic solvent (50%:50%) at 40 °C for 60 min is a sustainable option to maximize the extraction yield of bioactive compounds and the antioxidant capacity of extracts. Using this set of conditions it was possible to obtain extracts containing total phenolics (302.5 ± 7.1 mg GAE/L), tannins (0.43 ± 0.06 mg TAE/L), and flavonoids (83.0 ± 1.4 mg ECE/L), exhibiting DPPH• scavenging activity (326.0 ± 5.7 mg TE/L) and ferric reducing antioxidant power (1791.9 ± 126.3 mg SFE/L). These conditions allowed, in comparison with other "more effective" for some individual parameters, a cost reduction, saving time and energy.

1. Introduction

In recent years, sustainability has become an essential aspect to be observed in any human activity. For example, sustainable food manufacturing and processing (which reduces raw materials waste and minimizes refuse) is already a mandatory component of the business model. Furthermore, it can represent a challenge and an opportunity to innovate, looking for better commodities and production technologies, improving the overall environmental performance of products throughout their life-cycle, and, in this way, to gain the confidence of the increasingly informed consumers.

In the specific case of coffee producers, several procedures have already been adopted to obtain a more sustainable product namely: organic farming practices, preserving native forests and biodiversity, bird safe and shade grown coffee, and fair-trade coffee. With this, producers aimed to obtain a certification that will provide them preferential market access and sell at a differential price. Nevertheless, the coffee industry (the second largest commodity in the world after oil industry) still generates large amounts of residues representing serious environmental problems (Mussatto et al., 2011) and, because of that, waste management is also an increasingly important issue. Studies show that coffee husks, a residue from coffee processing, are potential raw materials for bioethanol production (Gouvea et al., 2009). Other alternative is biodiesel production using oil extracted from defective coffee beans instead of roasting for consumption of beverage with depreciated quality (Oliveira et al., 2008b). Other potential examples of by-products valorization in the coffee industry are the use of defective coffee beans press cake (resultant from biodiesel production) or coffee husks to be used as adsorbents for the removal of dyes from aqueous media (Oliveira et al., 2008a). The production of low-cost adsorbents from coffee husks for the treatment of wastewater containing heavy metals has been under study as well (Oliveira et al., 2008c).

The aforementioned recycling alternatives are only interesting for coffee producing countries while in importing countries, where roast of green processed beans is performed, the wastes are mainly associated with roasting and consumption: coffee silverskin and spent grounds, respectively. The last example consists in the remains of ground roasted coffee after beverage preparation. Recently, a lot of attention is being given to their recycling, namely,

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http://dx.doi.org/10.1016/j.indcrop.2014.01.006

as a possible fertilizer after composting, a versatile barrier in reducing pesticide leaching through soil, a source of oil for biodiesel or biomass for bunker fuel substitution (Adi and Noor, 2009; Fenoll et al., 2011; Kondamudi et al., 2008).

Coffee silverskin is an integument that covers the raw coffee bean. This light pellicle constitutes a by-product of coffee roasting since it is detached during this procedure. Since usually it is not used to prepare coffee beverages, silverskin is separated from the beans by air flow and used, afterwards, as firelighters or dispatched for landfills.

The preparation of dietary supplements/nutraceuticals, food ingredients, and some pharmaceutical products is increasingly made from the extraction of bioactive compounds from natural products (Dai and Mumper, 2010). We believe that coffee silverskin might be an important source of several bioactive compounds. In fact, the scarce literature about this matrix suggests that it contains dietary fiber (Napolitano et al., 2007; Pourfarzad et al., 2013) and antioxidants (Borrelli et al., 2004; Murthy and Naidu, 2012), two recognized factors in the prevention of chronic diseases (Kris-Etherton et al., 2002). In the work presented herein, we aimed to optimize a sustainable method to obtain silverskin extracts with the highest possible amount of antioxidants. The effect of the main variables affecting the extraction yields and antioxidant activity of extracts were studied, namely, solvent polarity, temperature and time of extraction. Water and ethanol were selected because of their low toxicity. In order to modulate the solvent polarity, different ratio mixtures (25:75, 50:50 and 75:25) were used. An experimental design for analyzing the effects of time and temperature of extraction was performed for each solvent mixture. Extracts were compared regarding their total phenolics, flavonoids and tannins contents. Moreover, their antioxidant capacity was assessed by two complementary procedures, namely, the ferric reducing antioxidant power (FRAP) method and the 1,1-diphenyl-2-picrylhydrazyl radical (DPPH•) scavenging capacity assay.

With this, we aim to contribute to coffee silverskin valorization and recycling, taking advantage of its potential health properties since its extracts could be henceforth applied in products such as antioxidant food supplements or cosmetic products.

2. Materials and methods

2.1. Reagents and standards

Gallic acid, tannic acid, epicatechin, trolox, sodium acetate, Folin–Ciocalteu's phenol reagent, DPPH• (2, 2-diphenyl-1-picrylhydrazyl), sodium nitrite, ferric chloride, aluminum chloride, TPTZ (2,4,6-tripyridyl-s-triazine) solution, and ferrous sulfate heptahydrate were all obtained from Sigma–Aldrich (St. Louis, USA). Sodium carbonate anhydrous, sodium hydroxide and absolute ethanol were purchased from Merck (Darmstadt, Germany). Ultrapure water was treated in a Milli-Q water purification system (Millipore, Bedford, MA, USA) and used to prepare all aqueous solutions.

2.2. Samples and sample preparation

Silverskin sample were gently provided by a national coffee roaster industry (Bicafé – Torrefação e Comércio de Café, Lda., Portugal). Sample was representative of the major by-product of this industry and resulted from the roast of a commercial coffee blend constituted by ~40% of arabica and ~60% of robusta coffee beans (*Coffea arabica* and *Coffea canephora* var. *robusta*, respectively). After reception, samples were ground (Grindomix GM 200, Retsch, Haan, Germany), homogenized and used to prepare extracts.

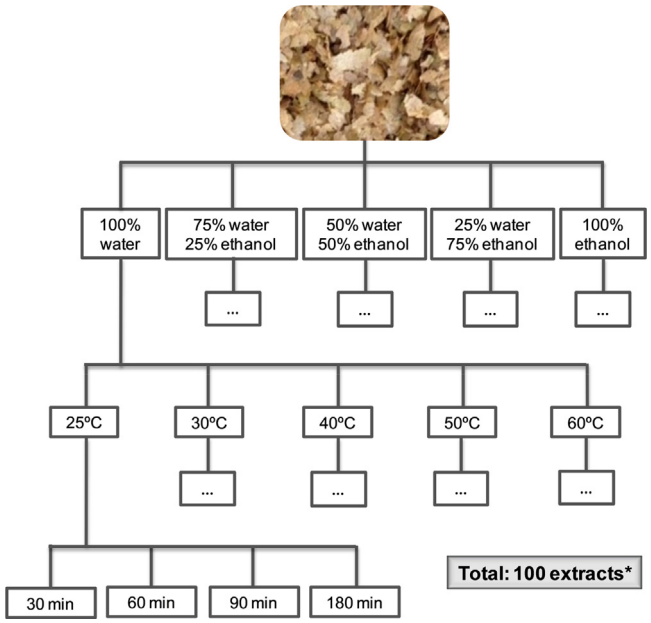

Fig. 1. Scheme of the silverskin extracts preparation. *Each extract was prepared in triplicate and each replicate was analyzed in triplicate.

2.3. Extracts preparation

In order to study the optimal extraction conditions, different procedures were tested by varying solvent polarity, temperature, and time of extraction. Fig. 1 outlines the different conditions used to prepare extracts.

Briefly, 1 g of ground sample and 50 mL of solvent/solvent mixture (100% water, 75% water/25% ethanol, 50% water/50% ethanol, 25% water/75% ethanol or 100% ethanol) were brought into contact at different temperatures (25, 30, 40, 50, and 60 °C), for periods of 30, 60, 90, and 180 min. Extractions were performed on a heating plate with constant stirring (600 rpm) and each set of conditions (solvent, time and temperature) was performed in triplicate. The final extracts were filtered and stored at −25 °C prior analysis.

2.4. Total phenolics content

Total phenolics contents of diluted extracts (1:10) were determined according to Alves et al. (2010). Briefly, 500 μL of each extract were mixed with 2.5 mL of the Folin–Ciocalteu reagent (1:10) and 2 mL of a sodium carbonate solution (7.5% m/v). The mixture was first incubated at 45 °C, during 15 min, followed by 30 min incubation at room temperature before absorbance readings at 765 nm were performed. Total phenolics content was calculated from a calibration curve prepared with gallic acid (10–100 mg/L; $r = 0.9997$) and expressed as mg of gallic acid equivalents (GAE)/L of extract.

2.5. Total flavonoids content

Total flavonoids contents were determined according to Barroso et al. (2011) with slight modifications. Aliquots of 1 mL of extract were mixed with 4 mL of distilled water and 300 μL of 25% sodium nitrite. After 5 min at room temperature, 300 μL of 10% $AlCl_3$ were added, and 1 min after 2 mL sodium hydroxide (1 M) and 2.4 mL of ultrapure water. The absorbance was recorded at 510 nm. Total flavonoids content was calculated through a calibration curve of