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Influence of extraction process on antioxidant capacity of spent coffee

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ABSTRACT

Spent coffee that is produced in tons by restaurants and cafeterias, and consumers at domestic levels, could be a good opportunity to have an important source of natural antioxidants. The main aim of this work was to study the influence of several process factors on the antioxidant capacity extraction from spent coffee. Total phenolic compounds, radical scavenging activity (ABTS and DPPH) and browned compounds (Abs 420 nm) of spent coffee extracts obtained with continuous (Soxhlet 1 h and 3 h) and discontinuous methods (solid–liquid extraction and filter coffeemaker), several solvents (water, ethanol, methanol and their mixtures), successive extractions, and water with different pHs (4.5, 7.0 and 9.5) were carried out. Spent coffee extracts with the highest antioxidant capacity were obtained after one extraction with neutral water (pH 7.0) in a filter coffeemaker (24 g spent coffee per 400 mL water). Furthermore, spent coffee defatting and extract lyophilization allowed us to obtain spent coffee extracts powder with high antioxidant capacity that can be used as an ingredient or additive in food industry with potential preservation and functional properties.

1. Introduction

The preparation of a good cup of coffee requires several technological steps from the coffee fruit. During coffee processing and roasting, some residues are generated. Coffee pulp, and cherry and parchment husks are produced in plantations to obtain green coffee beans. In the roasting process, the teguments of green coffee beans, named silverskin, are removed. And during the manufacture of soluble and concentrated coffee, wastes are produced in the coffee industry. These coffee residues are frequently used by the industry as animal feed and fertilizer, but during the last few years other more friendly environment uses, such as biofuel production have been proposed (Saenger, Hartge, Werther, Ogada, & Siagi, 2001; Silva, Nebra, Machado, & Sanchez, 1998). Furthermore, during the last few years an increasing number of studies have shown the presence of phytochemicals related with health benefits in coffee residues (Borrelli, Esposito, Napolitano, Ritieni, & Fogliano, 2004; Ramalakshmi, Jagan Mohan Rao, Takano-Ishikawa, & Goto, 2009). In the case of silverskin, some authors have demonstrated the presence of high amounts of dietary fiber as well as antioxidant activity (Borrelli et al., 2004; Napolitano, Fogliano, Tafuri, & Ritieni, 2007). Also in the residues obtained during processing of soluble coffee, antioxidant properties, which may be attributed to phenolic and nonphenolic compounds, have been found (Murthy & Madhava Naidu, in press; Ramalakshmi et al., 2009; Yen, Wang, Chang, & Duh, 2005). Moreover, technological factors play an important role in antioxidant extraction during the brewing process (Andueza, Vila, de Peña, & Cid, 2007; Pérez-Martínez, Caemmerer, de Peña, Cid, & Kroh, 2010) and, consequently, may have an influence on the presence of remaining antioxidant compounds in spent coffee. For this reason, the results of the antioxidant activity reported for coffee residues from the soluble coffee industry cannot be directly extrapolated to the spent coffee obtained with coffeemakers.

Spent coffee that is produced in tons by restaurants and cafeterias, and consumers at domestic levels, could be a good opportunity to have an important source of natural antioxidants, also from an economical point of view. However, studies about the health related phytochemicals, such as antioxidants of spent coffee obtained during the brewing process have not been found. Previously it would be necessary to develop an easy, efficient, safe and cheap method to obtain spent coffee antioxidant extracts. In previous cited studies, the extraction of antioxidants from industry soluble coffee residues has been made for hours using continuous (Soxhlet) and discontinuous methods, with different solvents, such as water, ethanol, methanol, n-hexane, isopropanol and their mixtures at different proportions. Moreover, the antioxidant activity has also been measured by different methods. For all these reasons, the main aim of this work was to study the influence of several process factors on the antioxidant capacity extraction from spent coffee in order to establish the most efficient procedure, i.e. for obtaining spent coffee extracts with the highest antioxidant capacity. Consequently, the antioxidant capacity of spent coffee extracts obtained with continuous and discontinuous methods, several solvents, successive extractions, and

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solvents with different pHs were compared and the most efficient extraction conditions have been selected. Moreover, the influence of spent coffee defatting and extract lyophilization on the antioxidant capacity have also been studied in order to obtain a new product that can be used as a natural antioxidant or ingredient with potential preservation or functional properties.

2. Materials and methods

2.1. Chemicals and reagents

The methanol, ethanol and petroleum ether used were of analytical grade from Panreac (Barcelona, Spain). Folin–Ciocalteau reagent, sodium carbonate, sodium bicarbonate, lactic acid and sodium hydroxide were also obtained from Panreac (Barcelona, Spain). Gallic acid, Trolox (6-hydroxy-2,5,7,8-tetramethyl chroman-2-carboxylic acid), 2,2′-Azinobis (3-ethylbenzothiazonile-6-sulfonic acid) diammonium salt (ABTS) and 2,2-diphenyl-1-picrylhydrazyl (DPPH˙) were purchased from Sigma-Aldrich (Steinheim, Germany).

2.2. Spent coffee preparation

Guatemala Arabica roasted coffee was provided by a local factory. Coffee beans were ground for 20 s using a grinder (model Moulinex super junior "s"). Filter coffee brew was prepared from 24 g of ground roasted coffee for a volume of 400 mL of water, using a filter coffeemaker (model AVANTIS 70 Inox, Ufesa, Spain). Extraction took approximately 6 min at 90 °C. Ground roasted coffee after brewing, namely spent coffee, was dried to a constant weight for 2 h at 102 ± 3 °C in an oven (JP SELECTA, Barcelona, Spain).

Spent coffee was defatted with Petroleum Ether (1:11, w/v) at 60 °C for 3 h in a Soxhlet extraction system Extraction Unit B-811 Standard BÜCHI (Flawil, Switzerland).

2.3. Spent coffee extract preparation

Three procedures were used to prepare spent coffee extracts: solid–liquid extraction, filter coffeemaker and Soxhlet extractor. Spent coffee extracts were prepared from 24 g of spent coffee for a volume of 400 mL in all cases. For solid–liquid extraction, spent coffee was mixed with water at 80 °C for 10 min, cooled in an ice bath for 10 min and filtered through Whatman No. 1 filter paper. For extraction with filter coffeemaker, spent coffee was extracted with water in a filter coffeemaker (model AVANTIS 70 Inox, Ufesa, Spain) for approximately 6 min at 90 °C. For Soxhlet extraction, spent coffee was extracted with water at 100 °C, boiling for 15 min followed by reflux for 45 min (1 h of total extraction time) and for 165 min (3 h) in a SOXTEST SX-6 MP, Raypa (Terrassa, Spain).

For the selection of solvent experiment, 400 mL of water, four different water:ethanol mixtures (80:20, 60:40, 40:60, and 20:80), pure ethanol, two different water:methanol mixtures (30:70, and 70:30), and pure methanol were used to extract 24 g of spent coffee by means of a filter coffeemaker (model AVANTIS 70 Inox, Ufesa, Spain) for approximately 6 min at 90 °C.

For successive extraction study, 24 g of spent coffee was extracted five times with 400 mL of water each time in a filter coffeemaker (model AVANTIS 70 Inox, Ufesa, Spain) for approximately 6 min at 90 °C.

For the extraction with aqueous solutions at different pH, 400 mL of an acid solution (pH 4.5), water (pH 7), and alkaline solution (pH 9.5) were used to extract 24 g of spent coffee by means of a filter coffeemaker (model AVANTIS 70 Inox, Ufesa, Spain) for approximately 6 min at 90 °C. Acid solution (pH 4.5) was prepared with 36.03 mg of lactic acid and 6.99 mg sodium hydroxide. Alkaline solution (pH 9.5) was prepared with 1.68 g sodium bicarbonate and 3.77 g of sodium carbonate.

For the last experiment, aqueous extracts from undefatted and defatted spent coffee were lyophilized using a CRYODOS Telstar (Terrassa, Spain).

2.4. Total phenolic compounds

Total phenolic compounds were measured using the Folin–Ciocalteau reagent according to Singleton's method (Singleton & Rossi, 1965) and then calculated using Gallic Acid (GA) as standard. For every spent coffee extract, 3:10 dilutions with demineralized water were prepared. A volume of 500 μL of Folin–Ciocalteau reagent was added to a mixture of 100 μL of the extract sample and 7.9 mL of demineralized water. After a 2 min delay, 1.5 mL of a 7.5% sodium carbonate solution was added. Next, the sample was incubated in darkness at room temperature for 90 min. The absorbance of the sample was measured at 765 nm in a spectrophotometer Lambda 25 UV/VIS (Perkin Elmer Instruments, Madrid, Spain). Gallic Acid was used as reference, and the results were expressed as milligrams of GA per gram of spent coffee dry matter (mg GA/g dm).

2.5. Antioxidant capacity by ABTS assay

The ABTS antioxidant capacity was performed according to the method of Re et al. (1999). The radicals ABTS•$^{+}$ were generated by the addition of 0.36 mM potassium persulfate to a 0.9 mM ABTS solution prepared in phosphate buffered saline (PBS) (pH 7.4), and the ABTS•$^{+}$ solution was stored in darkness for 12 h. The ABTS•$^{+}$ solution was adjusted with PBS to an absorbance of 0.700 (±0.020) at 734 nm in a 3 mL capacity cuvette (1 cm length) at 25 °C (Lambda 25 UV–VIS spectrophotometer, Perkin-Elmer Instruments, Madrid, Spain). An aliquot of 100 μL of each spent coffee extract sample diluted with demineralized water (3:100) was added to 2 mL of ABTS•$^{+}$ solution. The absorbance was measured spectrophotometrically at 734 nm after exactly 18 min. Calibration was performed with Trolox solution (a water-soluble vitamin E analog), and the antioxidant capacity was expressed as micromoles of Trolox per gram of spent coffee dry matter (μmol Trolox/g dm).

2.6. Antioxidant capacity by DPPH assay

The antioxidant capacity was also measured using 2,2-diphenyl-1-picrylhydrazyl (DPPH•) decolorization assay (Brand-Williams, Cuvelier, & Berset, 1995). A 6.1×10^{-5} M. DPPH• methanolic solution was prepared immediately before use. The DPPH• solution was adjusted with methanol to an absorbance of 0.700 (±0.020) at 515 nm in a 3 mL capacity cuvette (1 cm length) at 25 °C (Lambda 25 UV–VIS spectrophotometer, Perkin-Elmer Instruments, Madrid, Spain). Spent coffee extracts were diluted 3:10 in demineralized water prior to analysis. Samples (50 μL) were added to 1.95 mL of the DPPH• solution. After mixing, the absorbance was measured at 515 nm after exactly 1 min and then every minute for 18 min. Calibration was performed with Trolox solution (a water-soluble vitamin E analog). The antioxidant capacity was expressed as micromoles of Trolox per gram of spent coffee dry matter (μmol Trolox/g dm).

2.7. Browned compounds (Abs 420 nm)

Fifty microliters of spent coffee extracts was diluted up to 2 mL with demineralized water. Browned compounds were measured by the absorbance of samples at 420 nm, after exactly 2 min in a 3 mL capacity cuvette (1 cm length) with a Lambda 25 UV–Vis spectrophotometer (Perkin-Elmer Instruments, Madrid, Spain) connected to a thermostatically controlled chamber (25 °C) and equipped with UV WinLab software (Perkin Elmer). This measurement was employed as a convenient index of the development of caramelization and Maillard reactions (MRs) (Meydav, Saguy, & Kopelman, 1977).