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Research Article

Physicochemical Characterization of Spent Coffee Ground (*Coffea arabica* L) and its Antioxidant Evaluation

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Abstract: Spent Coffee Ground (SCG) represents an opportunity to reuse as a new raw material due to its properties and the amount of material available. Taking in account this, the aim of this study was to characterize its physicochemical properties and antioxidant activity. Bromatological properties like ash, cellulose, fat, Elemental analysis (C, H, N, Calcium, Iron, Sodium, Phosphorus and potassium) and moisture were evaluated. As results of investigations it was found values Ash 1.71%, residual moisture 7.42%, fat 14.7%, cellulose 30.58%. Hemicellulose 20.2%, lignin 17.91% and protein 10.34%. In other hand, minerals analyzed for major elements showed that potassium was the most abundant mineral with a value of 0.442%. While Calcium, phosphorus, sodium and Magnesium had values of 0.184, 0.145, 0.024 and 0.148%, respectively. Minor elements Copper (ppm) 12.8, Iron (ppm) 7.50 and Manganese (ppm) 50.98. Emulsifying Activity values using four different oils 60.42% (Corn Oil), 62.30% (Soya Oil), 63.44% (Isopropylmiristate) and 62.30% (Mineral Oil). Relating to antioxidant activity exhaustive coffee waste (*Coffee arabica* L) had promising result with values of IC50 of 14.38 and 8.10 for DPPH and ABTS respectively. SCG showed characteristics of a natural antioxidant that could be employed in food industry as possible substituents of synthetic antioxidants.

Keywords: Agroindustrial waste, antioxidant, bromatological properties, coffee, physicochemical properties

INTRODUCTION

Coffee is one of the world's most popular beverages and its commercial importance has grown steadily in during the last 150 years (Daglia *et al*., 2000). Coffee is a key product in any economy i.e., Colombia produced in 2016 around 853.8 tons of coffee (FedeCafe, 2017). In the process of transformation coffee to get soluble coffee production and beverages a solid residue is obtained, this product is Spent Coffee Ground (SCG).

SCG is the most abundant coffee by-products generated in coffee beverage preparation (Murthy and Naidu, 2012). About 2 kg of wet SCG are obtained for each kg of instant coffee preparation, with an annual generation of 6 million tons worldwide (Mussatto *et al*., 2011a; Tokimoto *et al*., 2005). Considering this huge amount of coffee residue produced all over the world, so reutilization of this material is a relevant subject (Mussatto *et al*., 2011a). To reuse SCG is necessary to know chemical and physicochemical properties.

According to investigations, SCG contains large amounts of organic compounds like fatty acids, lignin, cellulose, hemicellulose and other polysaccharides. Extraction of these compounds can open a source of value-added products. Thus, SCG has been investigated for biodiesel production (Caetano *et al*., 2012), as source of sugars (Mussatto *et al*., 2011a), animal feeding (Givens and Barber, 1986), production of ethanol (Machado, 2009) and as a source antioxidants (Esquivel and Jimenez, 2012).

In this study, physicochemical characterization of an SCG from a renowned coffeehouse chain was investigated. Its characterization includes Elemental composition (C, N, Major elements and Minor Elements), bromatological properties (Ash, Fat, Moisture and Volatiles, Protein, Emulsifying activity), Fourier Transform-Infrared (FTIR) spectroscopy and Antioxidant Activity (DPPH, ABTS). We expect from data to find a potential use of constituents of this waste material.

MATERIALS AND METHODS

Sample collection: Sample was collected during 7 days in a recognize brand and store coffee, then was homogenized and dried until values <10%.

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Moisture and volatile matter: Moisture content was determined by gravimetric method as described in AOAC, 1990 925.10 at 105°C until constant weight. Results were expressed as %.

Ashes: The amount of ashes was quantified by gravimetric method as described in AOAC (1990) 923.03 based in completed incineration of organic Matter at 550°C. Results were expressed as %.

Fat: Total fat content was quantified by soxhlet extraction with petroleum ether as described in AOAC (1990) 945.16. Results were expressed as %.

Total protein: Content of total protein was determinated by Kjeldahl acid digestion method as described in (AOAC, 1990) 955.04. A conversion factor of 6.25 was used to calculate proteins content. Results were expressed as %.

Elemental analysis: The elemental analysis C of exhaustive coffee sample were analyzed using a Leco CS844 Content of minerals (Major and Minor elements) was quantified by Atomic Absorption Spectrometry (AAS) as described in AOAC (1990) 965.09. Results were expressed as % for major elements and ppm for minor elements.

Cellulose, hemicellulose and lignin: Previous cellulose, hemicellulose and lignin determination, the extractives from SCG were removed in a Soxhlet extraction system (Tecator, HT2, Netherlands) using ultrapure water and absolute ethanol as solvents in two sequential stages (Sluiter *et al*., 2008). The extractive free SCG samples were dried at 60°C to constant weight to be stored. To determine the cellulose, hemicellulose and lignin (ash-free) contents, the raw material was submitted to a two-steps sequential acid hydrolysis (Sluiter *et al*., 2010). Sugars in the resulting solution were determined by high performance liquid chromatography (Mussatto *et al*., 2011b) and were used to calculate the cellulose (as glucose) and hemicellulose (as arabinose, mannose, galactose and xylose) contents (Mussatto and Roberto 2006). The lignin (ash-free) content was also calculated as described by Mussatto and Roberto (2006).

Emulsifying capacity: To determine the ability of the starches to hold a stable emulsion, 1 g of each starch was mixed with 25 mL of distilled water, stirred for 15 min with a magnetic system. This solution was added with 25 mL of an oily substance used in the cosmetic industry and stirred/homogenized for 3 min. Afterward the product was centrifuged at 1300 rpm for 5 min. The Emulsifying Capacity (EC) was expressed in terms of percentage, considering the Volume of the Layer which still remaining Emulsified (VLE) to the Total Liquid Volume (TLV):

$$\%EC = VLE/\,TLV * 100$$

Antioxidant activity evaluation: Two methodologies were used to determine the antioxidant activity of each EO: DPPH and ABTS.+

Radical method DPPH•: Free radical scavenging activity DPPH• was determined using the method described in literature (Silva *et al*., 2014) with some modifications 75 µL of sample were added to 150 µL of a methanol solution of DPPH• (100 ppm) and they were incubated at room temperature for 30 min, after the disappearance of the DPPH• radical was determined spectrophotometrically at 405 Nm in microplate reader Multiskan Ex (Thermoscientific). Ascorbic acid (25 µg/mL) was used as a positive control:

$$\%Inhibition = (Ao - Af)/\,Ao * 100$$

where,

A_0 and A_f : The absorbance values of blank (DPPH solution in alcohol) and the sample (DPPH solution plus alcohol-dissolved), respectively

Radical method ABTS•+: The free radical scavenging activity ABTS was determined using the method described by Re *et al*. (1999), with some modifications. The ABTS• radical was formed following the reaction of 3.5 mM ABTS with 1.25 mM of potassium persulfate (final concentration). The samples were incubated at 2-8°C and in darkness for 16-24 h. Once the ABTS radical was formed, it was diluted with ethanol until having an absorbance of 0.7±0.05 at 734 nm. To a volume of 190 µL of the ABTS radical dilution was added 10 µL of the EO sample and incubated at room temperature for 5 min; after this time, the disappearance of the ABTS radical at 734 nm was determined spectrophotometrically in the microplate reader Multiskan Ex (Thermoscientific). Ascorbic acid (4 µg/mL) was used as a positive control for the uptake of ABTS • radicals.

Statistical analysis: All analyzes were performed by quintupled and the results are the mean values. The Standard Deviation (SD) and the evaluation coefficient (SD) were also calculated.

RESULTS AND DISCUSSION

Discussion of results:

Total protein: As seen in Table 1, SCG showed a significant protein percent of 10.03%. In literature this result was similar according to range reported about 6.7-9.9% (Lago *et al*., 2001), lower values than reported in other investigations 11.20% (Martinez-Saez *et al*., 2017) and up to 16.9% for SCG (Cruz *et al*., 2012). In some occasions, the protein content is maybe overestimated

Table 1: Physicochemical characterization of SCG

Analysis	Results
Moisture and volatile matter (%)	7.43±0.073
Ashes (%)	1.71±0.051
Total protein (%)	10.03±0.277
Fat (%)	14.70±0.557

Sample was analyzed as received; Results are expressed as mean (n = 5)

Table 2: Elemental analysis of SCG

Analysis	Results
Carbon (%)	47.90±0.150
Nitrogen (%)	2.01±0.074
Carbon/nitrogen ratio	23.83

Table 3: Elemental constituents of SCG

Major elements	Results
Calcium (%)	0.184±0.003
Magnesium (%)	0.148±0.002
Phosphorus (%)	0.145±0.003
Potassium (%)	0.422±0.018
Sodium (%)	0.024±0.001
Minor elements	
Copper (ppm)	12.80±0.187
Iron (ppm)	77.50±0.716
Manganese (ppm)	50.98±0.192

due to the presence of other nitrogen-containing compounds like caffeine, amino acids and free amines (Campos-Vega *et al*., 2015; Delgado *et al*., 2008). Total nitrogen value was about 2.01%. However, mean value accepted for SCG is 13.6% (Mussatto *et al*., 2011a). It is important to establish that content of protein in SCG is higher than roasted bean coffee due concentration of the non-extracted nitrogen compounds during preparation of the beverage (Arya and Rao, 2007).

Ash and elemental composition: The content of ashes in SCG was 1.71% was higher compared with the ranged of values for SCG (0.19-1.6%) (Pujol *et al*., 2013; Caetano *et al*., 2012; Lago *et al*., 2001; Mussatto *et al*., 2011b; Campos-Vega *et al*., 2015). These variations could be related to the quality of soil where plant is set.

Table 2 shows elemental analysis, where values for C and N were 47.9 and 2.01% respectively. These results are lower than reported 57.16% for C and higher 1.18% for N (Pujol *et al*., 2013). C/N ratio found was 23.83, this results opens an opportunity to use SCG as soil remediation. Because is similar to soil needs (20:1) (Elbl *et al*., 2014) Compost available carbon increases microbial activity, resulting in increased capacity for mineral nitrogen retention (additionally supplied from compost and another mineral fertilizer). N is captured in soil organic matter (Diaz *et al*., 2011).

Minerals: Table 3 chows the amount of minerals in SCG. Major elements (Ca, Mg, P and K) are bigger values with a trend K>Ca>P>Mg>Na having same tendency in literature K>P>Mg (Mussatto *et al*., 2011a) and Ca>Na>Fe (Pujol *et al*., 2013). Now these tendencies are not a rule to follow because variations of metal content depending of soil and variety of coffee. (Hombunaka and Rowell, 2002; Laviola *et al*., 2007).

Cellulose, hemicellulose and lignin: Sugars are presents in SCG in different polymeric chain structures Cellulose (Glucose) and Hemicellulose (Arabinose, Galactose, Mannose and Xylose). These two sugars summed are most abundant component with an average value of 50.80%. Cellulose is more abundant 30.58% than hemicellulose 20.2%. Literature reports similar value of 51.50% (Ballesteros *et al*., 2014) and higher values range between 59.20 and 62.64% (Pujol *et al*., 2013). High values of sugar gives an opportunity to be extracted form SCG and used to production of fermented products.

On the hand, content of lignin is significant with a value of 17.92% for this SCG. This value is similar to previous reported 17.20% (Roberto *et al*., 2003) and 18.93% (Mesa *et al*., 2011). But is a lower value compared to 23.90% (Ballesteros *et al*., 2014) and 19.40% (Meneses *et al*., 2013). Lignin is a macromolecule composed of a great variety of functional groups including phenolic hydroxyl, aliphatic hydroxyl, methoxyl, carbonyl and sulfonates and its structure varies from a raw material to another (Stewart, 2008). Chlorogenic, caffeic and coumaric acids are the most relevant lignin components in coffee and such compounds play an important role in health due to their antioxidant properties (Maydata, 2002).

Fat: SCG have been reported to contain 10-15% in fat (Jenkins *et al*., 2014), 9.3-16.2% (Cruz *et al*., 2012) and sometimes higher average of 20% (Lago *et al*., 2001; Martinez-Saez *et al*., 2017). In this case, we reported a mean value of 14.7%±0.557 and it is according to literature. This amount of fat is considerable taking in account that it is a residue produced a lot and could be extract to some applications. Variations of content are related to the method extraction and variety of coffee.

FTIR analysis spectra: FTIR shows all possible chemical groups' presents in SCG (Fig. 1). This spectrum has absorption bands typical of lignocellulosic materials. Two strong bands between 3000-3600/cm are observed, 3329 and 3014 /cm, could be related to modes of vibration of -OH and -NH functional groups respectively (Kante *et al*., 2012; Ballesteros *et al*., 2013).

Another 2 peaks at 2926 and 2858 /cm can be attributed to symmetric and asymmetric stretching of C-H bond. These bands confirm presence of methyl and methylene groups (Ballesteros *et al*., 2014). In previous report of FTIR analysis of coffee beans these peaks are associated to the presence of caffeine (Craig *et al*., 2012). However, because caffeine is soluble in hot water it is unlikely that there is an appreciable amount in SCG sample. But in caffeine beverage, like coffee or tea, where large amounts are expected could be used these

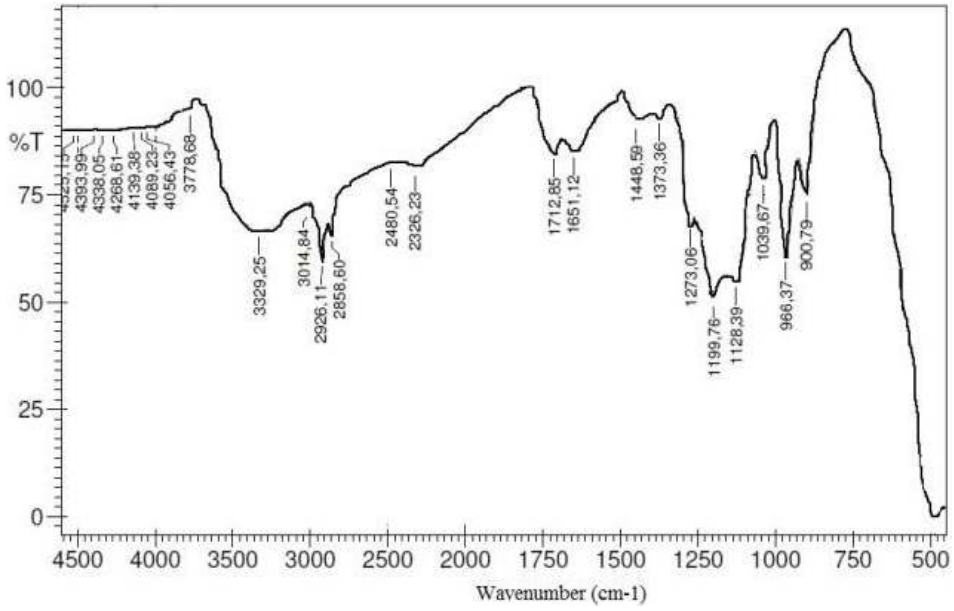

Fig. 1: FTIR spectra of SCG

bands to quantitative analysis of caffeine (Paradkar and Irudayaraj, 2002). Literature reported these bands could be associated to presence of lipids (Cremer and Kaletunc, 2003).

The appearance of bands in 1712 and 1651 /cm are highly associated to chlorogenic acids and caffeine (Ribeiro *et al*., 2010). Also 1712 /cm have been related to stretching of C = O bond associated to presence of lipids and aliphatic esters (Lyman *et al*., 2003) or in triglycerides (Kemsley *et al*., 1995). Therefore, this band is assigned to lipids. The low intensity band at 1651 /cm is due to C = C vibration of lipids and fatty acids and C = C vibration of aromatic rings from lignin moieties, respectively (Wang and Lim, 2012). The band at 1651 /cm can be also ascribed to the carbonyl stretching from lignin moieties (Herbert, 1971). The band at 1448 /cm corresponds to C-H bending of CH_3 groups.

Finally, bands shown at 1128, 1039 and 966 /cm results from vibration of C-O and C-O-H bonds of sugars (Cellulose, Hemicellulose and Others sugars), respectively. It is important to establish that the FTIR technique can only be used to confirm the presence of functional groups in the sample given the significant amount of substances.

Emulsifying activity: Emulsifying Activity (EA) is the capacity that a compound has to form a homogenous dispersion of two immiscible liquids or emulsions (Sánchez-Zapata *et al*., 2009). EA values using four different oils, used in cosmetic products, were 60.42±0.531% (Corn Oil), 62.30±0.376% (Soya Oil), 63.44% (Isopropylmiristate) and 62.30±0.650% (Mineral Oil). EA values are higher than reported 54.72% (Ballesteros *et al*., 2014).

SCG is a material with excellent emulsifying activity and emulsion stability and present therefore great potential to be used as emulsifiers in different food products including beverages, dairy, baking, confectioneries, or in products for animal nutrition, which require long emulsion stability (Ballesteros *et al*., 2014).

Antioxidant activity: In order to evaluate antioxidant capacity extracts were produced by solid-liquid extraction using methanol using two methods. Results in antioxidant activity were 14.38±0.239 and 8.10±0.074 IC50 for DPPH and ABTS respectively. According to the current literature, antioxidant activity in food and biological systems uses different methods to evaluate it. However, as each method is based on a different reaction, it is strongly advisable determining the antioxidant potential of a sample by different methods in order to better interpret the results (Ballesteros *et al*., 2014).

Exhibition of antioxidant activity in SCG is related to different substances presence in sample. Antioxidant activity in SCG has been associated to Phenolic compounds (Balasundram *et al*., 2006), lipids (Jenkins *et al*., 2014) and terpenes (Silva *et al*., 2004). Some of these groups were detected in FTIR analysis described above. Antioxidant capacity can be used as an ingredient or additive in food industry with potential preservation and functional properties (Bravo *et al*., 2013) and These antioxidants have been associated with health benefits (Campos-Vega *et al*., 2013; Campos-Vega *et al*., 2009).

CONCLUSION

According to properties of SCG could open opportunities to reuse it for possible applications because

low cost and abundance in production of coffee products. SCG is an excellent material for different applications that could be used for production of bioethanol due high content of carbohydrates represented in Cellulose, Hemicellulose and bioremediation of soils because C/N ratio that would help to fix atmospheric nitrogen for plants.

In other side, values of fat indicate a possibility of extraction for possible uses. In this particular case, we extracted fat to make a chemical characterization and evaluation for its antioxidant activity and cosmetic use (Author´s paper in press).

Finally, values of EA indicate a great potential to reuse SCG as emulsifiers in different food products including beverages, dairy, baking, confectioneries, or in products for animal nutrition, which require long emulsion stability. Besides, stability of products improve taking in count the good antioxidant activity showed.

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CONFLICT OF INTEREST

Authors declare no interest conflict for this work.

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