Effect of Peroxide Treatment on Functional and Technological Properties of Fiber-Rich Powders Based on Spent Coffee Grounds

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***Abstract*—The preparation of coffee beverage, as well as the industrial production of soluble coffee, generates a considerable amount of residues, the spent coffee grounds, SCG. There are no current profitable applications for these residues and an adequate disposal must be carried out since they are heavily pollutant and can be used to adulterate roasted and ground coffee. Thus, the objective of this work was to evaluate the effect of alkaline hydrogen peroxide treatment on the chemical composition and technological properties of spent coffee grounds in order to verify its potential as source of antioxidant dietary fibers. Evaluated parameters included moisture, fat, ash, soluble and insoluble dietary fiber contents, phenolics and in-vitro antioxidant potential, hydration properties and color. Total dietary fibre contents were shown to be high in comparison to commonly employed fibre sources, and increased with peroxide concentration. Phenolics content and the antioxidant activity of the treated spent coffee grounds decreased with increasing hydrogen peroxide concentration. Luminosity values increased significantly, and hydration properties showed a slight improvement with the increase in peroxide concentration. Overall, the alkaline hydrogen peroxide treatment produced an insoluble-fiber-rich material with physical and chemical characteristics that allow for its utilization as a source of antioxidant dietary fibers.**

***Index Terms*—spent coffee grounds, dietary fiber, antioxidant, agri-food residues**

I. Introduction

Coffee is one of the most popular and consumed beverages in the world. The preparation of coffee beverage and production of soluble coffee generate significant amounts of solid residues, corresponding to roasted and ground coffee depleted of water soluble substances. Such residues, named Spent Coffee Grounds (SCG), are extensively pollutant, given the high contents of organic substances that demand great quantities of oxygen to decompose [1]. Aside from the environmental issues, SCG present an additional disposal problem, because they can be used for adulteration of roasted and ground coffee [2]-[4].

As it is usually observed for agricultural and food wastes in general, the first attempts towards finding alternative uses for SCG have been concentrated on applications such as fertilizers, solid fuel, and supplements for animal feed [5]-[7]. Recent studies confirmed their potential as a fuel and fertilizer [8], [9] as well as for other applications, including use as an adsorbent [10] and as raw material for production of liquid biofuels such as ethanol and biodiesel [11].

The role of coffee consumption in preventing several diseases has been well established, in association with phenolics, indicating that it could be viewed as a functional beverage [12]-[14]. Phenolic compounds are mainly found in coffee beans as Chlorogenic Acids (CGA). Recent studies have shown that the amount of CGA is much higher in spent coffee grounds in comparison to the corresponding brew, indicating that although these water-soluble compounds are transferred to the beverage, a significant amount will remain on the solid matrix and thus contribute to the antioxidant activity of spent coffee grounds [15]. SCG are also rich in polysaccharides, polymers that can be used as dietary fiber and present immunostimulatory activity [16]. Thus, SCG have been the subject of recent studies in order to better evaluate its chemical composition, especially as a source of dietary fiber and phenolics [17]-[19].

The use of dietary fibers obtained from food and agricultural residues is often limited by undesirable sensory characteristics of the resulting food product, including gritty texture and poor appearance [20]. The physical properties of fibers can be substantially altered by treatment with alkaline hydrogen peroxide, which acts by solubilizing part of the lignin and reducing cellulose crystallinity through rupture of the hydrogen bonding between and into chains, producing a material with more open internal structure [20]. As a consequence, technological properties such as water retention capacity and swelling are improved, with a positive impact on the sensory characteristics of product [21], [22]. In view of the aforementioned, and the well established potential of SCG as a source of phenolics, the objective of this study was to evaluate the effect of alkaline hydrogen peroxide treatment on the chemical composition and technological properties of SCG.

Manuscript received January 5, 2016; revised March 18, 2016.

doi: 10.18178/ijfe.2.1.42-47

II. Materials and Methods

A. Materials

Spent coffee grounds obtained as solid residues after preparation of espresso coffee in a commercial establishment (Belo Horizonte, MG, Brazil) were employed in the tests. Samples were kept frozen (-12 ℃) until further use.

B. Preparation of Dietary Fiber Powder

SCG samples were defrosted and dried at 60±2 ℃. Samples were then ground (0.355mm<D<0.425mm) and stored at 25±2 ℃ in tightly sealed plastic containers for further analyses (SC). SC samples were treated with a dilute, alkaline solution of hydrogen peroxide [21]. In summary, the treatment consisted of contacting the sample with a solution of distilled water (1:5 w/v) containing hydrogen peroxide at 0, 5, 10, 15, 20 and 25% (v/v). Each suspension was adjusted to pH 11.5 with NaOH and stirred gently at room temperature for 18 h. The suspension was then adjusted to pH 6 with HCl, and the insoluble portion collected by filtration. The treated materials were washed with water until pH 7 and dried in a convective oven at 60 ℃ for 12h, and the resulting powders were termed SC0, SC5, SC10, SC15, SC20 and SC25 in regard to the H_2O_2 solution concentrations (see Fig. 1).

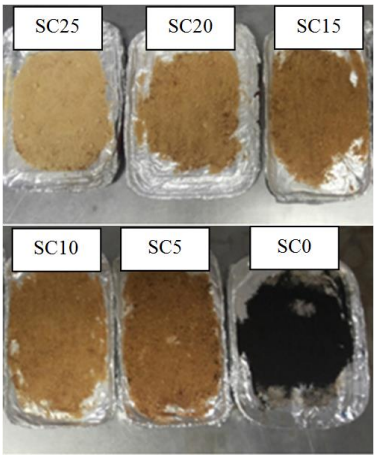

Figure 1. Resulting powders obtained after hydrogen peroxide treatment.

C. Chemical Characterization

Moisture, ash and fat contents were determined according to the reference methods described by AOAC [23]. Moisture content of the samples was determined by oven-drying at 105 ℃ until constant weight. Crude fat was determined by the Soxhlet method using ether extraction (AOAC method 4.5.05). Ash was quantified after burning at 550 ℃ for 20 h (AOAC method 942.05). The total Dietary Fiber Content (TDF) was evaluated by an enzymatic–gravimetric method. Briefly, the samples were submitted to enzymatic digestion with α-amylase (100 ℃ for 15 min), followed by pepsin and pancreatin, both for 60 min at 40 ℃. Subsequently, Insoluble Dietary Fiber (IDF) was filtered and washed with warm distilled water. The filtrate and washed water were combined, mixed with 95% ethanol at 60 ℃ and let stand for 60 min in order to precipitate the Soluble Dietary Fiber (SDF). The residues were weighed after overnight drying at 105 ℃ in a convective oven. Total Dietary Fiber (TDF) was calculated as the sum of IDF and SDF.

D. Technological Properties

SCG color was measured using a Hunter colorimeter model ColorFlex (Hunter Associates Laboratory, Reston, VA) with standard illumination D_{65} and colorimetric normal observer angle of 10 °. Measurements were based on the CIE $L^*a^*b^*$ three dimensional Cartesian (xyz) color space represented by: Luminosity (L^*), ranging from 0 (black) to 100 (white) – z axis; parameter a^*, representing the green–red color component – x axis; and parameter b^*, representing the blue–yellow component – y axis. However, given that visual color can be better represented and discussed in terms of polar coordinates, a^* and b^* values were converted to color saturation or chroma (c^*) and color tone or hue angle (h):

$$c^* = \left[a^{*2} + b^{*2} \right]^{1/2} \quad (1)$$

$$h = \tan^{-1} \left[b^* / a^* \right] \quad (2)$$

Swelling Capacity (SWC), Water Retention Capacity (WRC) and Oil Retention Capacity (ORC) were evaluated according the methodologies described in the literature [24], [25] with slight modifications. For SWC determination, each sample (1g) was hydrated in distilled water (70mL) in a calibrated cylinder at room temperature. Suspensions were stirred in a shaker (150rpm) for 120 min. After equilibration for 18 h, the bed volume was recorded and SWC was calculated as volume (mL) per dry mass (g) of sample. For WRC/ORC determination, distilled water/soybean oil (25mL) were added to dry samples (1g), stirred (150rpm, 20 min) and centrifuged (3500rpm, 10 min). The supernatants were discarded and each shaker tube was weighed, with WRC and ORC calculated as g water or g oil per g of dry sample, respectively. Evaluation of the Water Solubility Index (WSI) was based on the procedure described by Yang *et al.* [25], with slight modifications. The supernatant from WRC determination was dehydrated (105 ℃, 15h). WSI was then evaluated as the ratio between the weight of this dried sample and the weight of the original sample.

E. Phenolics and in Vitro Antioxidant Capacity

The preparation of extracts for evaluation of phenolic compounds and antioxidant capacity was based on the following procedures [26]: The prepared fiber rich powders (1g) were placed in test tubes and sequentially extracted with 40mL of 50% (v/v) methanol and 40mL of 70% (v/v) acetone. The supernatants were combined and brought to a final volume of 100mL with distilled water.

The total amount of extractable phenolics (TEP) was determined according to the Folin–Ciocalteau method as described by Singleton *et al.* [27] with slight modifications. In summary, 1mL of the sample extract was added to 1mL of Folin–Ciocalteu reagent followed by addition of 2mL of sodium carbonate solution and

2mL of water. The mixture was stirred and allowed to stand for 120 min. The absorbance was read at 765nm using a UV/Vis spectrophotometer (Micronal AJX 1900, São Paulo, Brazil). The results were expressed as mg of Gallic Acid Equivalents (GAE) per g of dry matter based on a calibration curve of gallic acid at concentrations ranging from 10 to 40 μg/mL.

Non-Extractable Phenolics (NEPA) were evaluated according to the procedure described by Zurita *et al.* [28] employing the solid residues obtained from the preparation of extracts for evaluation of phenolic compounds. In vitro antioxidant capacity was evaluated according to the DPPH assay. In summary, 0.1mL of different dilutions (1:5, 1:10, 1:15, 1:20 and 1:25 v/v) of the prepared extracts in methanol were transferred to test tubes. 3.9mL of methanolic solution of DPPH (0.6mM) was added to each tube and homogenized. The tubes were incubated in the dark at room temperature for 20min. A control sample (0.1mL water/methanol/acetone) was prepared as previously described. Changes in the absorbance of the samples were measured at 515nm until absorbance readings became stable. IC_{50} represents the amount of dry extract required to decrease 50% of the initial DPPH concentration, and was determined by linear regression. Results were expressed in grams of dry sample per grams of DPPH [29].

F. Statistical Analysis

All determinations were done in triplicates and data expressed as mean values ± standard deviations. Data were statistically analyzed using one-way ANOVA and Tuckey tests, with 95% confidence ($p<0.05$).

III. Results and Discussion

A. Chemical Characterization

Table I shows the chemical composition of the obtained powders. Moisture levels are slightly higher for the samples that were treated with peroxide. Nonetheless, for all samples moisture levels are below the recommended upper limit of 9g/100g for fiber rich powders [30]. Both fat and ash contents show a slight decrease with water treatment (SCs vs. SC0) and then increase with peroxide concentration. The initial decrease is more significant in terms of ash content, due to the solubilization of these substances in water. The later increase is attributed to the fact that both fat and ash contents are expressed on dry basis of the specific sample and such values decrease with an increase of peroxide concentration, due to the solubilization of lignin and other substances. This effect was not as significant in ash content, due to the extraction of minerals during processing, so the decrease in dry mass (denominator) is partially compensated by the decrease in mineral content (numerator).

The results for dietary fibre contents are also shown in Table I. Values are high in comparison to literature data. Average reported values for total dietary fiber content in SCG (without any treatment) are 45g/100g, comprised of 35g/100g insoluble and 8g/100g soluble fiber [17]. Total Dietary Fibre (TDF) contents are high in comparison to commonly employed fibre sources such as rice and wheat bran (~27-45g/100g) [31] and fibre rich powders based on banana flour (~42g/100g) [32] and chia seeds (~41g/100g) [33]. TDF values are slightly low in comparison to other coffee processing byproducts such as coffee silverskin (~62g/100g) [34] and coffee husks and pulp (~66g/100g) [35]. However, TDF values in SCG can increase up to 70 g/100 g after treatment with hydrogen peroxide over 20% concentration. Results confirm the potential of SCG as sources of dietary fibers.

TABLE I. Chemical Composition of the Produced Powders (g/100g)

Sample	Moisture	Fat	Ash
SC	5.92 ±0.76^{b}	11.07 ±0.21^{d}	3.58 ±0.29^{d}
SC0	6.00 ±0.28^{b}	9.23 ±0.12^{e}	1.93 ±0.30^{e}
SC5	7.97 ±0.11^{a}	16.43 ±0.08^{c}	4.32 ±0.39^{cd}
SC10	8.08 ±0.10^{a}	17.05 ±0.38^{c}	5.08 ±0.13^{c}
SC15	8.31 ±0.14^{a}	16.64 ±0.32^{c}	4.90 ±0.18^{c}
SC20	8.85 ±0.16^{a}	18.59 ±0.19^{b}	6.66 ±0.41^{b}
SC25	8.64 ±0.16^{a}	20.46 ±0.25^{a}	7.65 ±0.37^{a}
Sample	**TDF**	**IDF**	**SDF**
SC	50.62 ±0.72^{b}	48.35 ±0.55^{b}	2.27 ±0.16^{b}
SC0	50.73 ±0.94^{c}	48.96 ±0.92^{b}	1.77 ±0.15^{b}
SC5	48.58 ±0.31^{c}	45.17 ±0.24^{c}	3.40 ±0.11^{a}
SC10	47.14 ±0.42^{c}	43.68 ±0.29^{c}	3.46 ±0.14^{a}
SC15	46.97 ±0.15^{c}	43.46 ±0.28^{c}	3.50 ±0.36^{a}
SC20	70.04 ±0.81^{a}	69.11 ±0.87^{a}	0.91 ±0.05^{c}
SC25	68.33 ±0.85^{a}	67.75 ±0.81^{a}	0.57 ±0.15^{c}

Average value ± standard deviation. Different letters in the same column indicate that values are significantly different ($p>0.05$). TDF = total dietary fiber; IDF = insoluble dietary fiber; SDF=soluble dietary fiber.

The soluble and insoluble nature of dietary fibers affect both their technological functionality and physiological effects. IDF are usually associated to porosity, low density and ability to increase fecal bulk and decrease intestinal transit [31]. IDF corresponds to approximately 80 to 90% of the fibres present in SCG. IDF values are high in comparison to other residues such as mango (~39g/100g), lemon (~42g/100g), and orange (~52g/100g) peels [31]. SDF are also of interest in association to their capacity to increase viscosity and to reduce the glycemic response and plasma cholesterol as well as prebiotic action [31]. SDF values in spent coffee grounds are quite low in comparison to other agricultural residues such as carrot peel and pomegranate bagasse (~9.8-19.9g/100g) [24], [36].

B. Technological Properties

Results obtained for color parameters of the produced powders are displayed in Table II. All color parameters are significantly affected by peroxide treatment. Luminosity values increase with peroxide concentration, and this is an interesting characteristic if darkening of the food product is a concern. Color intensity also increased significantly with treatment, regardless of the peroxide concentration. Hue angle values are also very different for the treated samples, corresponding to a variation of color tone from reddish-yellow to yellow.

TABLE II. COLOR PARAMETERS

Sample	L (%)	c*	h (deg)
SC	17.98 ±0.43[a]	4.17±0.14[a]	29.06 ±2.7[a]
SC0	17.98 ±0.43[a]	4.17±0.14[a]	29.06 ±2.7[a]
SC5	45.91 ±0.19[b]	30.58 ±0.35[b]	65.64±0.26[b]
SC10	59.84 ±0.48[c]	36.88 ±0.16[b]	70.76 ±0.23[b]
SC15	60.48 ±0.41[c]	35.99 ±0.58[b]	71.11±0.38[b]
SC20	66.20 ±0.57[c]	38.61±0.03[b]	75.35 ±0.07[bc]
SC25	77.60 ±0.17[d]	30.58 ±0.19[b]	82.34 ±0.06[c]

Average value ± standard deviation. Different letters in the same column indicate that values are significantly different (p>0.05). L = luminosity; c* = color intensity; h = hue angle.

The results obtained regarding hydration properties of the produced powders are displayed in Table III. The Water Retention Capacity (WRC) represents the amount of water that remains bound to the hydrated fiber following the application of an external force (pressure or centrifugation), being affected by particle size, porosity, hydrophobicity, and measurement temperature. WRC values are low, but similar to those of other residues including pomegranate bagasse (4.5-4.9g/100g) and soybean hulls (3.5-4.0g/100g) [25], [31], [36]. WRC is also related to the soluble DF content, and low levels of SDF are usually associated to low WRC values, because cellulosic fibers present poorer WRC than soluble fibers.

TABLE III. TECHNOLOGICAL PROPERTIES OF THE PRODUCED POWDERS

Sample	WRC (g water/g)	ORC (g oil/g ms)	SWC (ml/g ms)	WSI (g/100g)
SC	2.43 ±0.04[d]	2.64 ±0.13[e]	4.97 ±0.00[b]	3.82 ±0.60[c]
SC0	2.67±0.10[d]	2.89 ±0.01[ed]	3.92 ±0.06[c]	7.62 ±1.25[bc]
SC5	3.00 ±0.11[c]	3.10 ±0.11[cd]	4.87 ±0.07[b]	4.03 ±2.04[c]
SC10	3.52 ±0.68[ab]	3.00 ±0.15[d]	4.97±0.01[b]	9.61 ±2.18[abc]
SC15	3.26 ±0.17[bc]	3.40 ±0.05[c]	4.91 ±0.08[b]	9.51 ±2.20[abc]
SC20	3.46 ±0.16[b]	4.58 ±0.07[a]	5.62 ±0.50[a]	11.41 ±1.60[ab]
SC25	3.78 ±0.05[a]	4.24 ±0.03[b]	5.52 ±0.46[a]	14.82 ±3.00[a]

Average value ± standard deviation. Different letters in the same column indicate that values are significantly different (p>0.05). WRC = water retention capacity; ORC = oil retention capacity; SWC=swelling capacity; WSI = water solubility index.

ORC is an important parameter if avoiding fat loss during processing is a concern. It is also beneficial in terms of flavor retention, and a relevant feature if the fiber is intended to be used as an emulsifier. The ORC values obtained in this study are high in comparison to that for tomato peel (~1.5g/g) and wheat bran (~1.6g/g) [31], [37], but low in comparison to that for blanched carrot peels (~27-34mL/g) [24]. This is attributed to carrot peels containing much higher amounts of SDF in comparison to our samples. WSI values are high in comparison to rice flour (~2.1g/100g). Peroxide treatment provided a significant improvement on technological parameters, as expected.

C. Phenolics and in Vitro Antioxidant Capacity

Results obtained for Total Extractable (TEP) and Non-Extractable (NEPA) phenolics are displayed in Table IV. These values represent an important indicator of antioxidant potential, and a preliminary screening method for any product intended as natural source of antioxidants in functional foods. From these results it can been seen that both TEP and NEPA decreased significantly with the peroxide treatment, indicating that both free and bound phenolics were removed with the treatment. Nonetheless, TEP values obtained in this study are higher or in the same range as those reported for chia seeds if up to 10% peroxide solution is employed [33]. It is important to point out that TEP values will vary significantly depending on the extraction procedure and that the employed method (Folin Cicauteau essay) is quite generic and thus a further investigation on the profile of phenolics of the produced powders is recommended.

TABLE IV. PHENOLIC CONTENT AND IN VITRO ANTIOXIDANT POTENTIAL OF THE PRODUCED POWDERS

Sample	TEP (mg GAE/100g)	NEPA (mg /100g)	IC_{50} (g/g DPPH)
SC	10.25 ±0.05[a]	2336.16 ±143.85[a]	604.92 ±50 54.14[c]
SC0	7.30 ±0.03[b]	2152.28 ±143.59[a]	746.77 ±30.31[c]
SC5	1.30 ±0.04[c]	886.64 ±109.23[b]	2026.91 ±774.84[b]
SC10	1.01 ±0.04[d]	501.21 ±41.37[c]	2207.98 ±46.41[b]
SC15	0.52 ±0.05[e]	486.85 ±95.39[c]	2295.97 ±201.25[b]
SC20	0.50 ±0.03[e]	256.55 ±33.51[c]	3220.48 ±69.23[b]
SC25	0.45 ±0.03[e]	249.78 ±28.24[c]	4607.05 ±51.55[a]

Average value ± standard deviation. Different letters in the same column indicate that values are significantly different (p > 0.05). TEP = total extractable phenolics; NEPA = non-extractable phenolics.

Although most studies that discuss the phenolic contents of food products have been focused on TEP data, a considerable amount of phenolic compounds are still present in the extraction residues [38], and are referred to as non-extractable polyphenols, consisting mostly of Non-Extractable Proanthocyanidins (NEPA). They are oligomers and polymers of flavan-3-ol and flavan-3,4-diols and have been shown to present health related properties associated to the prevention of chronic diseases and gastrointestinal disorders. Since food products are consumed as a whole, including both extractable and non-extractable compounds, significant amounts of NEPA are ingested daily and thus contribute to the reported health effects associated to polyphenols. However, literature data on such compounds are still scarce [38]. NEPA levels in the produced powders (Table IV) are quite high in comparison to the ones reported for apple and pear peels (80-180mg/ 100g) and similar to that for bananas (980mg/100g) [28], [39].

The DPPH assay measures the ability of the extract to donate hydrogen to the DPPH radical, resulting in bleaching of the DPPH solution. Thus, the greater the bleaching action, the higher the antioxidant activity, and the lower the IC_{50} value. Results of DDPH based radical scavenging activity (Table IV) indicate that the antioxidant activity also decreased with peroxide treatment. Nonetheless, values are still quite high in comparison to that for wheat bran (~25.729g/g). Although it is evident that the peroxide treatment decreases both the phenolics content and antioxidant potential of the produced powders, values are still high in comparison to commonly employed fiber sources, especially for lower values of peroxide concentration, confirming the potential of the produced powders as sources of dietary fibers.

IV. Conclusions

An alkaline hydrogen peroxide treatment was evaluated as a process to improve the chemical and technological properties of spent coffee grounds as a source of antioxidant dietary fibers. The treatment produced a material with contents of insoluble dietary fiber higher than those of other agri-food residues reported in the literature. As the hydrogen peroxide concentration was increased above 20%, the IDF content increased significantly, with adequate technological properties such water and oil retention capacity and color. Both the phenolics content and the antioxidant activity of the treated spent coffee grounds decreased with increasing hydrogen peroxide. However, the resulting material still presented values for these properties higher than those of other agri-food residues reported in the literature. Thus, the produced material is deemed a potential source of antioxidant dietary fibers.

Acknowledgment

The authors wish to acknowledge financial support from Brazilian Government Agencies CAPES, CNPq and FAPEMIG.

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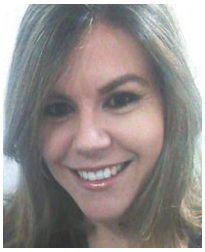

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