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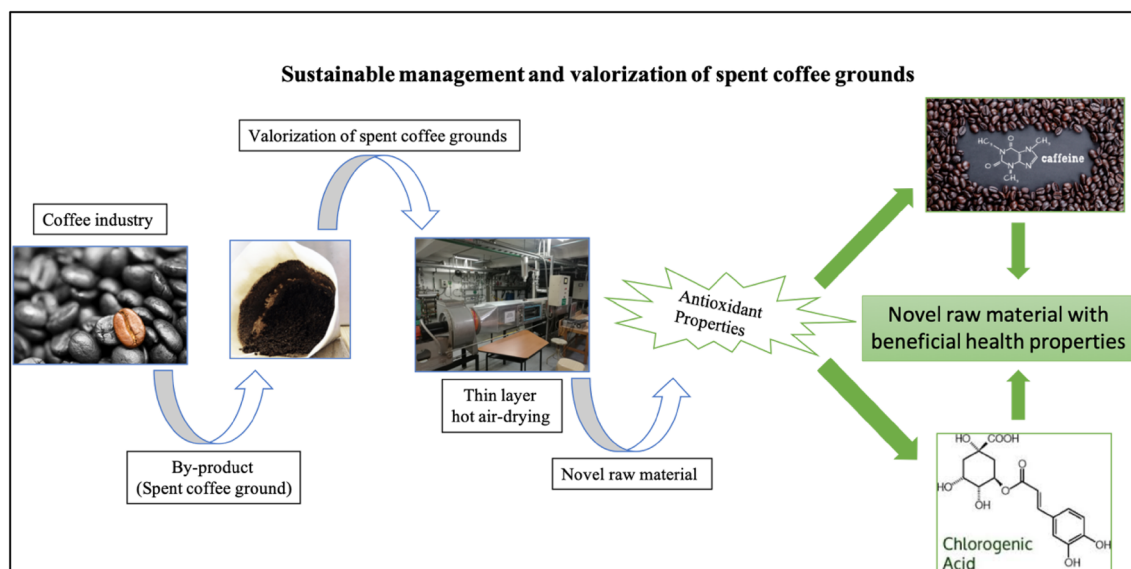
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Abstract

The spent coffee ground is a by-product of the coffee industry with high potential because of its beneficial properties for health, however, due to high water content, it is necessary to stabilize it without affecting bioactive capacities. The main objective of this work was to evaluate the effect of different convective drying conditions, on some technological and functional properties of the spent coffee ground and to determine the thermal degradation kinetics of chlorogenic acids. The methodology included the optimization of the drying process conditions of spent coffee ground: temperature (40–60 °C), air flow (1.0–2.0 m/s) and cake thickness (0.01–0.02 m). Effective diffusion coefficient, moisture, water activity, water and oil holding capacity, antioxidant capacity, caffeine, and seven chlorogenic acids were determined. The results showed that the best drying conditions were 60 °C, 2.0 m/s, 1.28 cm which allows retaining 84.24% of total polyphenols and 66.00% of the antioxidant capacity. Chlorogenic acids showed thermal degradation kinetics of first order under the optimal process conditions. In general, it is concluded that the convective drying process is a valid technique for processing of coffee grounds, as it allows the preservation of antioxidant compounds with a potentially beneficial effect on health, providing a stabilized low moisture content that can be used in food applications.

Graphic Abstract



Keywords Spent coffee ground · Effective diffusion coefficient · Bioactive properties · Technological properties · Chlorogenic acids

Extended author information available on the last page of the article

Statement of Novelty

Spent coffee ground is an industrial by-product produced after the extraction of the soluble solids from coffee beans. Spent coffee ground has a natural antioxidants compounds (phenolic and chlorogenic acids). Chlorogenic acids are important for their beneficial health properties. This study proposes for the first time to optimize the drying conditions, that allows retain the antioxidants compounds of spent coffee ground and can be used as an ingredient food, pharmaceutical or cosmetic industry. In the best of our knowledge, this is the first-time spent coffee ground are successfully processed by using a thin layer hot air-drying process method.

Introduction

The spent coffee ground (SCG), is the by-product obtained after the industrially or domestically extraction of the soluble solids present in roasted and ground coffee beans, formed by fine particles with a high moisture content [1, 2]. On average for each kilogram of instant coffee produced, 2 kg of SCG is generated; currently worldwide it is estimated that the annual production of SCG is approximately 6 million tons [3]. The SCG being 45–50% of the total by-products produced in the coffee industry, the remaining percentage is distributed among the pulp, cherry peels and parchment skin [4–6]. Due to the poor disposition that many of the companies give to the waste of coffee, this is considered as a highly polluting agro-industrial waste, which can generate changes in the biological demand of oxygen in the water sources, due to the presence of compounds such as caffeine, polyphenols, and tannins [7], increasing environmental pollution and even generation of carbon dioxide when burned [8].

Given the above, it is important to search alternatives for waste processing from the coffee industry; several authors report applications for coffee by-products, such as fertilizer [9], animal feed [10] and heavy metal removal [11]. It is also reported that SCG contain: cellulose, hemicellulose, protein, fatty acids, lignin, polyphenols, sugars, caffeine, and dietary fiber [12–16], as well as having anti-inflammatory and anti-allergenic properties [17, 18]. Different studies report applications of SCG to the extraction of active compounds such as phenolic acids [19], glycerin [20], kahweol and cafestol [21]. However, it is necessary to search for other processing alternatives that take full advantage of the spent coffee ground.

One of the alternatives for the processing and stabilization of agro-industrial by-products is drying, which aims

to reduce the water present in the waste, through the use of hot air, by significantly reducing water activity (A_w) [22, 23]. Drying is a complex phenomenon that involves processes of heat and mass, which are a function of the heat applied to vaporize the water or the volatile constituents present in the by-products to be processed [22, 24]. Several authors have reported the use of convective drying as a method of processing agroindustry by-products of avocado [25], fresh lime [26], mango (shell and seeds) [27], passion fruit [28] and spent coffee ground [7].

The objective of the present work was to evaluate different convective drying conditions that preserve the technological and functional properties of the spent coffee ground, establishing the kinetics of drying and degradation of some compounds of interest such as chlorogenic acids. Finally, this work is a processing alternative to agro-industrial waste generated in the coffee industry, with the aim of recovering a material with technological and functional properties suitable for its potential application in the development of products in the food and pharmaceutical industry; besides contributing to the reduction of environmental damage caused by waste.

Materials and Methods

Materials

The spent coffee ground (grains obtained after the preparation of the drink, 100% Arabica variety) was obtained from a cafeteria located at the University of Antioquia in the city of Medellín, Colombia. The standards of caffeine, chlorogenic acids, reagent Folin–Ciocalteu (2.0 N) (Darmstadt, Germany); Gallic acid, 2,2'-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (ABTS) Sigma-Aldrich Chemical® (Oakville-Ontario, Canada); Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) MP Biomedicals (Illkirch-Graffenstaden, France), magnesium oxide and solvents such as methanol and acetone Merck® (Germany).

Characterization of Spent Coffee Ground

The moisture, lipid content, ash, protein and total nitrogen of SCG were determined according to the methodology AOAC [29] and the carbohydrates were determined by difference [30]. The water activity (A_w) according to Martinez-Saez [3], all results were expressed in grams per gram of dry solids.

Hot Air-Drying Process of the Spent Coffee Ground

The drying process was carried out in a tray-type convection dryer, which consists of a fan coupled to an electrical

resistance for air heating, which passes through a tunnel of 3 m in length. The weight change over time was measured with a balance Vector® S-type scale, 5AX797, with an error of ± 0.1 g. The data collection was done by a data acquisition system, every 5 s. The drying process was done until achieving constant weight in the samples. The drying temperature was set at 40, 50 and 60 ± 1 °C, and the air velocity rate was set at 1.0, 1.5 and 2.0 ± 0.1 m/s and the thickness of the spent coffee ground cake was of 0.01, 0.015 and 0.02 m.

A dimensionless moisture ratio MR_t was calculated from the drying curves as shown in Eq. 1:

$$MR_t = \frac{X_t - X_e}{X_0 - X_e} \quad (1)$$

where X_t is the moisture content at any time t (g water/g dry basis), X_e is the moisture content at the equilibrium (g water/g dry basis) and X_0 is the initial moisture content (g water/g dry basis). Values of X_e are considered relatively small compared to X_t or X_0 [28].

The determination of the effective diffusion coefficients (D_{eff}) was performed using the solution for an infinite slab of second Fick's law (Eq. 2) [31]. The solution of Fick's law was used for one-dimensional transport, with the assumptions that moisture migrates only by diffusion, that negligible shrinkage occurs and that the diffusion coefficients and temperature are constant [32], Eq. 2.

$$MR_t = \frac{8}{\pi^2} \sum_{i=1}^{\infty} \frac{1}{(2i-1)^2} e^{\left(\frac{-(2i-1)^2 \pi^2 D_{eff} t}{4L^2} \right)}. \quad (2)$$

However, for long drying times ($MR_t < 0.6$), only the first terms of Eq. 2 is relevant for the evaluation of MR said equation, it is possible to simplify it in Eq. 3.

$$MR_t = \frac{8}{\pi^2} e^{\left(\frac{-D_{eff} \times \pi^2 \times t}{4L^2} \right)} \quad (3)$$

where D_{eff} is the effective moisture diffusion coefficient (m^2/s), t is the drying time (s) and L is the half-thickness of the slice (m).

Also, an anomalous diffusion model based on fractional calculus approach was applied in which for long drying times ($MR_t < 0.6$), the diffusion equation is written as follows:

$$MR_t = \frac{8}{\pi^2} e^{\left(-D_{eff} \left(\frac{\pi}{2L} \right)^2 t^\alpha \right)} \quad (4)$$

Diffusion coefficient D_{eff} has the dimension m^2/s^α . The value of α can distinguish some domains of anomalous transport: sub-diffusion if $0 < \alpha < 1$ or super-diffusion if

$\alpha > 1$. The threshold between both anomalous transport domains is normal Brownian diffusion [33].

Determination of Antioxidant Capacity and Total Polyphenols

Sample Preparation

For both, the wet and dry samples of the spent coffee ground, the extraction of the antioxidant compounds was based on the methodology proposed by Contreras-Calderón [34]. Basically, 500 mg of spent coffee ground was used to perform the aqueous extraction using a mixture of ethanol: water (50:50) and Acetone: water (60:40) at 25 °C in a mechanical stirrer, the solutions were centrifuged, and the supernatant was recoopered. The aqueous and dehydrated extracts were stored at -18 °C in the dark, extraction and measurements were determined in triplicate.

ABTS-Assay (2,2'-Azino-bis-3-ethylbenzthiazolin-6-sulfonic acid) The ABTS-assay was developed based on the methodology proposed by Contreras-Calderón [35]. For this, 100 μ L of aqueous extract, were diluted (1/50) with distilled water and mixed with ABTS radical, after 30 min the absorbance was measured at 730 nm (Varian Cary 50 UV-Vis Spectrophotometer). Aqueous solutions of Trolox (concentrations between 0 and 200 μ M) were used for the calibration, the results were expressed as micro-moles equivalent of Trolox per gram of dry material (μ molTEs/ $g_{d.b.}$). taking into account the value of the maximum concentration of the inhibitory mean (IC50) [36]. The measurements were made in triplicate.

Total Polyphenols Content (TPC) The total polyphenols content was determined using the test Folin-Ciocalteu according to Contreras-Calderón [34]. For this, 100 μ L of aqueous extract was properly diluted in distilled water. The amount of 1600 μ L of diluted extract was mixed with Folin-Ciocalteu reagent (100 μ L) and 300 μ L sodium carbonate (20 wt%), after 60 min in the dark, the absorbance was measured at 725 nm against the target (Varian Cary 50 UV-Vis Spectrophotometer). Gallic acid solutions (concentrations between 0 and 1000 ppm) were used for the calibration. The results were expressed as milligrams of gallic acid equivalents per gram of dry material (mg of GAEs/ $g_{d.b.}$). The measurements were made in triplicate.

Water and Oil Holding Capacity

For the water holding capacity (WHC), 0.1 g of dry samples of the spent coffee ground was taken and mixed with 10 mL of distilled and agitated water before starting an incubation for 24 h. The mixture was then centrifuged at 2500 rpm for

30 min, finally, the volume of the supernatant was determined, and the results were expressed as mL of water/g of sample. The oil holding capacity (OHC) was obtained by mixing 1.0 g of dry samples of the spent coffee ground with 10 mL of vegetable oil for 30 min at 25 °C, then the mixture was centrifuged at 2500 rpm for 10 min, finally the milliliters of the supernatant was determined and the results were expressed as ml of oil/g sample [28]. The measurements were made in triplicate.

Determination of Chlorogenic Acids (ACGs) and Caffeine

For the determination of ACGs and caffeine, an Agilent model 1260 equipment coupled to a diode array detector with a capacity to work in the range of 254–325 nm and using the column was made by means of High-Resolution Liquid Chromatography in reverse phase Zorb Eclipse XDB C18 (4.6 × 150 mm, 3.5 μm). The content of chlorogenic acids was determined according to Ludwig, Gloess [37, 38] and presents similarity to the Standard DIN 10767 /1994: Analysis of coffee and coffee products-Determination of chlorogenic acids content in roasted coffee and extracted coffee—HPLC method [39]. Basically, extraction with a mixture of methanol and acid aqueous solution was performed to 500 mg of spent coffee ground and 25 μL was used to inject in the system. In this chromatographic system were separated: 5-caffeoylquinic acid (5-CQA), 3-caffeoylquinic acid (3-CQA), 4-caffeoylquinic acid (4-CQA), caffeic acid (CA), 3,4-dicaffeoylquinic acid (3,4 di-CQA); 4,5-dicaffeoylquinic acid (4,5 di-CQA), 3,5-dicaffeoylquinic acid (3,5 di-CQA). The total content of chlorogenic acids was calculated by means of individual quantification by means of the external standard method and reported as the sum of each one expressed as the dry base percentage of chlorogenic acids. The measurements were made in triplicate.

In order to determine the effect of temperature on the antioxidant compounds present in SCG, kinetics of degradation was evaluated, adjusting the concentrations to a first-order reaction (Eq. 5), for the temperatures of 70, 60 and 50 °C with 2.0 m/s, 1.28 cm for air velocity and thickness cake of spent coffee ground respectively.

$$\ln \left(\frac{C}{C_0} \right) = -kt \quad (5)$$

where C_0 is the initial concentration of the parameter studied (ABTS, PTC, ACGs), C is the concentration of this in time t and k is the reaction constant.

For determination of the caffeine content in the coffee bean, the methodology described by Vignoli [40] with

some modifications, which contemplated the use of reverse phase chromatography and isocratic mode with 30% ethanol. Basically, extraction with a mixture of distilled water and magnesium oxide was performed to 500 mg of spent coffee ground and 50 μL was used to inject in the system. The caffeine concentration was determined by the external standard method at 273 nm and was reported as the percentage on a dry basis. The measurements were made in triplicate.

Particle Size Distribution of Spent Coffee Ground

To determinate the particle size distribution of dry spent coffee ground, 5.0 g of dry samples were used. The particle size distribution was obtained from the velocity of the distribution of particles suspended in a dispersion medium using a particle size analyzer Microtract S3500 (Microtract Inc, Montgomeryville, PA, USA). This equipment has a tri-laser technology with a measurement capability from 0.0024 to 2800 μm [41]. The analysis was performed in triplicate.

Design of Experiments and Statistical Analysis

To establish the drying conditions that maximize the bioactive, functional and technological properties of stabilized spent coffee ground, the response surface methodology was applied through a Box–Behnken experimental design; with temperature as independent variable (40, 50, 60 °C), air velocity (1.0, 1.5, 2.0 m/s) and the thickness of the spent coffee ground cake of 0.010, 0.015 and 0.020 m (Table 2). The responses that were measured included: D_{eff} , moisture content, A_w , WHC and OHC, antioxidant capacity, total polyphenols content, caffeine content and particle size. The responses variables were investigated by using a response surface methodology (RSM) and fitting the experimental values to the following second order equation, this is shown in Eq. 4 (Table 2).

$$Y = b_0 + \sum_{i=1}^2 b_i X_i + \sum_{i=1}^2 b_{ii} X_i^2 + \sum_{i < j=1}^2 b_{ij} X_i X_j \quad (6)$$

where Y is the response variable, b_0 is a constant, b_i are model coefficients linked to a linear effect, b_{ii} are the coefficients related to the quadratic effect, b_{ij} are the constants for the interaction effect, and X_i and X_j are the variables. The data and RMS were analyzed and performed using STAT GRAPHICS Centurion XVI software (Statistical Graphics Corporation, Version 16.0.07, Rockville, USA).

Table 1 Proximal characterization of the wet and dehydrated under optimal conditions of spent coffee ground

Parameter	Wet SCG	Dry SCG (optimal conditions)*
Moisture	60.07 ± 0.17	5.50 ± 0.07
Ash	1.87 ± 0.02	1.93 ± 0.01
Fat	12.4 ± 0.03	12.77 ± 0.04
Total Nitrogen	2.05	2.05
Protein (Nx6,25)	12.75 ± 0.08	12.84 ± 0.12
Carbohydrate content	72.8 ± 0.10	72.45 ± 0.10
Calories Kcal/100 g	181.48	431.07

*Units: g/100 g dry solid

Results

Characterization of Spent Coffee Ground

The results obtained for the proximal characterization of the spent coffee ground for wet and dehydrated under optimal drying conditions are showed in Table 1.

In general, the proximal characteristics of wet SCG had values of moisture, ash, fat, protein, and carbohydrates of 60, 1.87, 12.4, 12.75, and 72.8 g/100 g dry solid, respectively. The previous values are in accordance with that reported by Cruz et al. [42] who obtained values on dry basis for moisture, ash, fat, and protein of 60, 1.7, 11.5, and 14 g/100 g of SCG, respectively; likewise, [6] report values of 1.1 g/100 g of SCG for ashes, 15.1 g/100 g of SCG for total fat, 11.5 g/100 g of SCG for total protein, while for the carbohydrate content reported values of 68.4 g/100 g of SCG. Finally, [16] reported a content of ashes 1.6 wt% and total protein of 13.6 wt%.

The differences between the values reported respect to other presented by different authors, could be explained from the processing conditions in terms of the degree of roasting and grinding of the coffee beans, as well as the form of extraction or preparation of the beverage, since there are factors that have a direct effect on the properties and characteristics of spent coffee ground generated, as well as the variety of the type of coffee used [43].

Effect of Dehydration Conditions on Effective Diffusion and Functional Properties of Spent Coffee Ground

Table 2 summarizes the results of the drying process on the technological and functional properties of spent coffee ground.

According to the data shown in Table 2, the drying time required to get an MR = 0.1 varied from 130 to 310 min, while the D_{eff} based on second Fick's law model ranged from

0.99 to $4.53 \times 10^{-12} \text{ m}^2/\text{s}$ with values of R^2 ranged between 0.30 to 0.94, for run 12 and 14, respectively. Based on the values of R^2 observed it is possible to assume that the diffusion process is anomalous. Then, the anomalous model using fractional calculus was applied, obtaining that the α -value was 1.68, which is higher than 1 which implies that the diffusion process can be considered as super-diffusive [44]. The results also presented in Table 2 showed that the D_{eff} ranged between 2.44 to $12.43 \times 10^{-15} \text{ m}^2/\text{s}^{1.68}$ for runs 12 and 16, respectively. The values of R^2 were notoriously higher with values ranged between 0.8356 to 0.9948 attained in run 12 and 15, respectively. In general, the values of D_{eff} reported were lower than the values reported for food matrices, which varies between 10^{-11} y $10^{-8} \text{ m}^2/\text{s}$ [45], being comparable with values reported for other matrices such as: cassava, squash, spent coffee ground, passion fruit, banana [7, 28, 46, 47]. The temperature and air flow factors did not have a significant difference ($p > 0.05$) over the diffusion values, which is in accordance with what was reported by Duarte [28].

Figure 1 shows the drying curves obtained using the operating conditions that allowed to get the dehydrated product in 130 min (run 11) and 310 min (run 2), and the respective fitting of Fick's second law and anomalous model with $\alpha = 1.68$, where is observed better fitting for anomalous model.

Moisture content was between 4.38 and 26.9 g/100 $g_{d.b.}$ A_w between 0.1818 and 0.9863, WHC varied between 0 and 0.10-mL water/g SCG, while the OHC was between 1.0 y 2.833 mL oil/g SCG and antioxidant capacity ranged between 22.56 y 319.08 $\mu\text{molTEs}/g_{d.b.}$

$$\text{Moisture content} = 83.96 + 0.74125T - 10.93A - 78.62L - 0.017425T^2 - 0.0345TA + 0.4525TL - 4.25A^2 + 9.39AL + 13.97L^2 \quad (7)$$

$$Aw = -1.03971 + 0.123769T + 1.2465A - 2.17683L - 0.00134762T^2 - 0.024775TA + 0.022837L - 0.23655A^2 + 0.2531AL + 0.2783L^2 \quad (8)$$

$$\text{Caffeine} = -20.9675 - 0.005T + 18.695A + 15.14L + 0.0023T^2 + 0.0065TA - 0.1625TL - 4.86A^2 - 2.65AL - 0.82L^2 \quad (9)$$

$$D_{\text{eff Fick's law}} = 2.40375 - 0.0205T - 3.215A + 1.905L - 0.0001875T^2 + 0.024TA + 0.018TL + 0.365A^2 + 0.36AL - 0.415L^2 \quad (10)$$

$$D_{\text{eff anomalous}} = 68.0012 - 0.77425T - 34.6725A - 29.6275L + 0.00581T^2 + 0.137TA + 0.062TL + 6.175A^2L + 5.25AL + 7.615L^2 \quad (11)$$

The above equations represent the mathematical model for Moisture content (Eq. 7), A_w (Eq. 8), caffeine (Eq. 9), Diffusion coefficient based on second Fick's law model (Eq. 10), and anomalous model Diffusion coefficient (Eq. 11); which have the following coefficients R^2 : 76.68, 87.38, 74.07, 80.21 and 67.98 respectively. On the other

Table 2 Results for the experimental design, convective drying of spent coffee ground

Run	Temperature (°C)	Flow air (m/s)	Thickness (m)	Drying time to MR=0.1 (min)	$D_{\text{eff}} \times 10^{-12}$ (m^2/s)	$D_{\text{eff}} \times 10^{-15}$ ($\text{m}^2/\text{s}^{1.68}$)	Moisture content (g/100 g db)	A_w	WHC	OHC	ABTS ($\mu\text{Mol ETs/gdb}$)	Particle size (μm)	Caffeine content (mg/gdb)
1	50	2	0.01	210	1.61	4.36	12.14 ± 0.33	0.7266 ± 0.01	0.067 ± 0.10	2.83 ± 0.10	108 ± 12.4	269.2 ± 0.20	4.29 ± 0.005
2	40	1.5	0.02	310	3.47	8.40	26.19 ± 0.10	0.9777 ± 0.01	0.033 ± 0.10	2.50 ± 0.10	22.56 ± 2.2	352.8 ± 0.19	6.59 ± 0.005
3	40	2	0.015	295	2.64	5.54	10.28 ± 0.15	0.7185 ± 0.01	0.067 ± 0.10	2.00 ± 0.10	128.62 ± 2.0	322.8 ± 0.21	3.34 ± 0.005
4	40	1	0.015	270	3.02	7.45	24.63 ± 0.18	0.9863 ± 0.01	0.067 ± 0.10	2.50 ± 0.10	319.08 ± 6.9	349.5 ± 0.22	3.48 ± 0.005
5	50	1	0.02	300	3.77	9.13	22.99 ± 0.3	0.9714 ± 0.01	0.067 ± 0.10	1.33 ± 0.10	68.23 ± 7.3	347.6 ± 0.13	3.88 ± 0.005
6	50	1.5	0.015	280	2.52	6.05	18.35 ± 0.23	0.9406 ± 0.01	0.033 ± 0.10	2.16 ± 0.10	80.22 ± 6.4	211.5 ± 0.17	4.19 ± 0.005
7	60	2	0.015	250	2.95	7.31	4.38 ± 0.21	0.1818 ± 0.01	0.067 ± 0.10	2.16 ± 0.10	108.49 ± 2.1	248.5 ± 0.20	4.3 ± 0.005
8	50	1.5	0.015	260	2.94	6.71	12.90 ± 0.13	0.8156 ± 0.01	0.100 ± 0.10	2.00 ± 0.10	106.03 ± 6.4	337.1 ± 0.23	5.52 ± 0.005
9	50	2	0.02	270	3.33	8.73	19.74 ± 0.19	0.9690 ± 0.01	0.033 ± 0.10	1.63 ± 0.10	158.47 ± 4.3	339.2 ± 0.16	3.56 ± 0.005
10	50	1.5	0.015	200	3.64	1.02	20.80 ± 0.10	0.9614 ± 0.01	0.067 ± 0.10	1.83 ± 0.10	154.53 ± 3.7	333.7 ± 0.11	5.26 ± 0.005
11	60	1.5	0.01	180	1.69	11.32	7.75 ± 0.20	0.4673 ± 0.01	0.000 ± 0.10	1.16 ± 0.10	122.31 ± 2.4	308 ± 0.21	4.77 ± 0.005
12	40	1.5	0.01	270	0.99	2.44	21.69 ± 0.33	0.9631 ± 0.01	0.033 ± 0.10	1.66 ± 0.10	138.59 ± 9.6	195.3 ± 0.13	4.4 ± 0.005
13	60	1	0.015	250	2.85	6.54	19.42 ± 0.31	0.9451 ± 0.01	0.067 ± 0.10	2.00 ± 0.10	43.09 ± 3.2	238.9 ± 0.16	4.31 ± 0.005
14	50	1	0.01	130	2.41	5.18	24.78 ± 0.25	0.9821 ± 0.01	0.067 ± 0.10	1.16 ± 0.10	118.7 ± 10.8	246.9 ± 0.18	1.96 ± 0.005
15	50	1.5	0.015	310	2.07	4.65	17.88 ± 0.28	0.8897 ± 0.01	0.100 ± 0.10	1.00 ± 0.10	60.89 ± 10.3	239.5 ± 0.13	4.4 ± 0.005
16	60	1.5	0.02	240	4.53	12.43	21.30 ± 0.14	0.9385 ± 0.01	0.067 ± 0.10	1.83 ± 0.10	194.65 ± 1.0	332.3 ± 0.20	3.71 ± 0.005

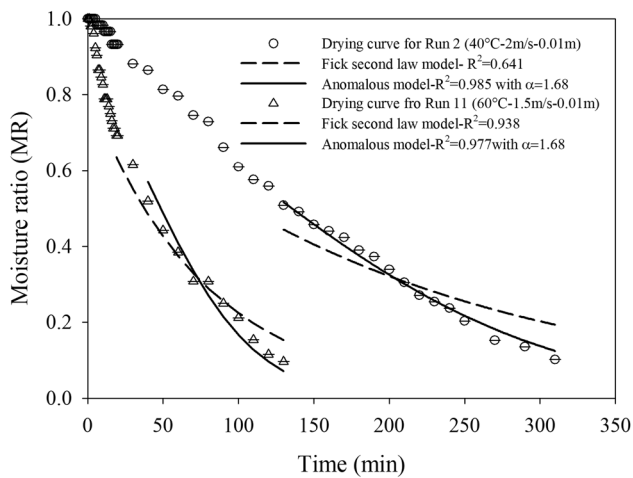


Fig. 1 Convective drying curve obtained for run 2 and 11 with their respective fitting to Fick's second law model and anomalous model

hand, the temperature (T) and air velocity (A) significantly influence the moisture content and A_w ($p < 0.05$), showing that higher values of temperature and air velocity decrease these parameters, this behavior is due to the higher temperature increases the enthalpy of the system, which increases the transfer of mass and energy of the same, accelerating the migration of water [48]. In the case of air velocity, similar results were reported by Duarte [28], who attribute this phenomenon to the fact that the increase of this factor generates an increase in the convection coefficient of the process, thus increasing the heat transfer process of the samples, which favor the quickly increment in the water temperature present in the cake.

In the case of thickness factor (L), it showed a significant impact ($p < 0.05$) on the A_w and the D_{eff} , which increased as thickness was lower. This phenomenon is due to the role that porosity played in the diffusion, since in a porous system, the presence of solid particles causes the diversion of the ways of diffusion of the species [49], this process is known as tortuosity, which is used to describe the resistance of a structure to the flow [50], since it is considered as the ratio between the length of a trajectory by means of a body and the linear distance between both points [51]. Therefore, during the dehydration process, a variation in the particle size of the material is generated, which decreases the distance of flow of water vapor through the material, thus decreasing the tortuosity of the system, and therefore increasing the diffusivity of the water. Similar results were found by Rojas and Augusto [52], who dehydrated pumpkin samples with two different types of cut and thickness.

Water and Oil Holding Capacity

Water holding capacity is an important technological property that is related to hydration capacity, mainly for those foods' rich in protein and fiber. The WHC obtained for the treated samples varied between 0 and 0.10 mL water/g SCG. On the other hand, the OHC obtained for the treated samples varied between 1.0 and 2.833 mL oil/g SCG. No significant effects ($p > 0.05$) of the studied factors were found with respect to the WHC and OHC. Higher values of OHC, indicate the affinity of the matrix and the potential use that can be provided in the formulation of emulsions, this effect can be explained from the physicochemical characterization of SCG, which has approximately 12.77 ± 0.04 g fat/100 g dry matter. The values obtained here are correlated for other by-products such as passion fruit and orange peel with oil retention between 2.98 and 5.15 g of oil/g of sample [28, 52].

Caffeine and Antioxidant Capacity

On average, the caffeine values obtained in the dehydrated samples were 4.5 mg of caffeine per gram of dry solid. This value is in the range of those reported by Panusa and Cruz [41, 53], who found values between 1.94 and 11.5 mg of caffeine per gram of dry solid. In general, none of the factors studied had a significant effect ($p > 0.05$) with respect to the caffeine content, this can be explained because the caffeine molecule is stable when subjected to thermal treatments [40].

The value of antioxidant capacity for wet SCG was $335.5 \mu\text{molTEs/g}_{d.b.}$, which is in accordance with literature reported [9, 42]. The variation of the antioxidant capacity is mainly due to the effect of the preparation of the beverage on the antioxidant content in waste, and the type of coffee used and the industrial treatments (roasting and grinding) [43]. The different drying processes allowed obtaining values of antioxidant capacity that ranged between 22.56 and $319.08 \mu\text{molTEs/g}_{d.b.}$, as shown in Table 2; with an average loss of 64% antioxidant capacity which is in accordance with [54–56]; who used convective drying as a technique for the dehydration of foods rich in antioxidant compounds. This effect can be explained by the structural changes that have antioxidant compounds, in the same way, physical changes in the material provide a greater exposure of these compounds to oxygen which increases its degradation.

With respect to the statistical analysis of the antioxidant capacity, none of the factors had a significant effect ($p > 0.05$), this possibly due to the great variability of the results, however, it was evidenced that the antioxidant

capacity decreased with the increase of the drying temperatures, being in accordance with Wojdylo [57], who report higher antioxidant capacity in samples of cherries dehydrated at 50 °C, than those processed at 60 and 70 °C. Although the temperature did not significantly affect the antioxidant capacity, and it can be evidenced that between 40 and 60 °C, it showed a concave parabolic behavior. This effect can be explained because the concentration of some phenolic compounds can increase when subjected to extreme stress conditions [58].

Optimization of the Drying Process of the Spent Coffee Ground

To optimize the drying process, the multiple response optimization methodologies were used, seeking to minimize moisture content, drying time, and A_w , in addition to maximizing the antioxidant capacity. The analyzes were made through the software STATGRAPHICS Centurion XVI software (Statistical Graphics Corporation, Version 16.07.07, Rockville, USA). Once the optimization of the process was done, the conditions thrown up were temperature of 60 °C, the air velocity of 2.0 m/s and cake thickness of 0.0128 m with desirability of 65%.

These processes conditions allowed to obtain a dehydrated spent coffee ground with a content of moisture, ash, fat, nitrogen, of 5.5 g/100gdb, 1.93 g/100gdb, 12.77 g/100gdb, and 2.05 g/100gdb, respectively. The parameters of ash, total fat, nitrogen, carbohydrates, and protein found in the samples of fresh and dehydrated SCG are similar, which indicates that the temperatures used in the study have no effect on the proximal characteristics. It can be concluded, the optimal conditions found, allow to have a product with antioxidant compounds and with values of percentage of humidity and water activity that allow its conservation, achieving a physical, biological and chemical stability [59].

Kinetics of Degradation of Polyphenols and ACGs

In order to understand the effect of temperature on the antioxidant compounds present in SCG, it was decided to evaluate its kinetics of degradation, adjusting the concentrations to a first-order reaction (Eq. 5), allowing to find the reaction constant (K) and the activation energy (E_a); depending on parameters such antioxidant capacity, total polyphenols content (TPC), and chlorogenic acids (ACGs) such as: 5-caffeoylquinic acid (5-CQA), 3-caffeoylquinic acid (3-CQA), 4-caffeoylquinic acid (4-CQA), caffeic acid (CA), 3,4-dicaffeoylquinquinic acid (3,4 di-CQA); 4,5-dicaffeoylquinquinic acid (4,5 di-CQA), 3,5-dicaffeoylquinquinic acid (3,5 di-CQA); which are

Table 3 Parameters of degradation kinetics of ABTS, total polyphenols and some chlorogenic acids, as a function of process temperatures

	Temperature (°C)	K (h ⁻¹)	R ²	E _A (kJ/mol)
ABTS	50	1.592	0.932	2.960
	60	1.639		
	70	1.657		
Total polyphenols	50	1.786	0.983	1.290
	60	1.803		
	70	1.814		
5CQA	50	1.302	0.814	2.830
	60	1.306		
	70	1.363		
3CQA	50	1.497	0.935	2.080
	60	1.508		
	70	1.542		
4CQA	50	1.391	0.843	2.500
	60	1.397		
	70	1.445		
3.4CQA	50	0.717	0.890	3.020
	60	0.769		
	70	0.783		
3.5CQA	50	0.697	0.998	3.570
	60	0.731		
	70	0.774		
4.5CQA	50	0.571	0.957	5.910
	60	0.609		
	70	0.698		

shown in Table 3. Chlorogenic acids are water-soluble molecules, formed by the esterification of quinic acid and one or more molecules of caffeic acid in carbons 3, 4 or 5; resulting in monocaffeoylquinic acids (3-CQA, 4-CQA, 5-CQA) and dicaffeoylquinic acids (3,4-diCQA, 3,5-diCQA, 4,5-diCQA) [13, 59].

In Table 3, the results for the reaction constants (K), activation energy (E_a) and R^2 , of antioxidant compounds present in SCG, subjected to a dehydration process with temperatures between 50 and 70 °C, are presented.

The reaction constant (k) and the regression coefficient R^2 are presented in Table 3. The first-order degradation kinetics for antioxidant capacity, total polyphenols, and chlorogenic acids was identified, where the rate of degradation increases as a function of temperature, under the conditions studied, the above in accordance with Aurelio [60], who report that the degradation of some antioxidant compounds such as polyphenols are adjusted to first and second order kinetics, in addition, that the degradation of the compounds increases with the increase in temperature.

On the other hand, the activation energy was: 2.960 kJ/mol; for ABTS, 1.290 kJ/mol; for total polyphenols and

Table 4 Concentrations of ABTS, total polyphenols and chlorogenic acids, as a function of process temperatures

Compound	Initial	70 °C	60 °C	50 °C
ABTS	277.13 ± 2.1	83.90 ± 1.8	105.48 ± 1.9	137.23 ± 2.0
TP	730.30 ± 0.2	309.48 ± 0.3	304.62 ± 0.1	374.86 ± 0.2
5 CQA	0.406 ± 0.1	0.318 ± 0.1	0.363 ± 0.1	0.450 ± 0.1
3 CQA	0.897 ± 0.1	0.713 ± 0.1	0.839 ± 0.1	0.998 ± 0.1
4 CQA	0.573 ± 0.1	0.448 ± 0.1	0.516 ± 0.1	0.630 ± 0.1
CA	0.005 ± 0.01	0.016 ± 0.01	0.023 ± 0.01	0.021 ± 0.01
3,4 di-CQA	0.079 ± 0.4	0.065 ± 0.4	0.072 ± 0.4	0.075 ± 0.4
3,5 di-CQA	0.075 ± 0.4	0.062 ± 0.4	0.074 ± 0.4	0.079 ± 0.4
4,5 di-CQA	0.061 ± 0.1	0.048 ± 0.1	0.057 ± 0.1	0.061 ± 0.1
Total ACGs	2.095 ± 0.1	1.669 ± 0.1	1.944 ± 0.1	2.314 ± 0.1

ABTS*: $\mu\text{Mol E Trolox/gdb}$

Total polyphenols content: mg E Gallic A/gdb

ACGs: mg ACGs/gdb

2.830, 2.080, 2.500, 3.020, 3.570, 5.910 kJ/mol ; for 5-CQA, 3-CQA, 4-CQA, 3,4-diCQA, 3,5-diCQA and 4,5-diCQA, respectively (Table 3). High values for the activation energy imply that the degradation of the polyphenolic compounds and chlorogenic acids are susceptible to the increase in temperature.

Table 4 shows the results obtained for antioxidant capacity, total polyphenols content and each of the chlorogenic acids studied.

From Table 4, it can be seen that the antioxidant capacity and the initial total polyphenol concentration were 277.13 $\mu\text{molTEs/g}_{\text{d.b}}$ and 730.30 mg of $\text{GAEs/g}_{\text{d.b}}$, respectively. These values are in accordance with Murthy, Bravo and Campos-Vega [9, 13, 42], both parameters decreased between 50 and 70% for antioxidant capacity and 49 and 58% for total polyphenols content. Some authors have reported similar losses in antioxidant capacity and total polyphenols content in foods subjected to convective drying processes, such strawberries [55], avocado by-products [25] and cherries [56]. This phenomenon can be explained by the prolonged exposure of food to oxygen and high temperatures causing the deterioration of phenolic compounds [57].

Figure 2 shows the behavior of the antioxidant capacity and the total polyphenols content during the drying process; a rapid degradation of the antioxidant activity and phenolic compounds can be observed during the first 90 min of the process at each of the temperatures studied. This phenomenon is due to structural changes in the molecules and the high moisture content, which facilitates the degradation reactions, since some hydrophilic compounds can act as catalysts [25]. However, the second stage of degradation was presented at the end of each of the dried, said effects were reported by Méndez-Lagunas [55], who attribute this second

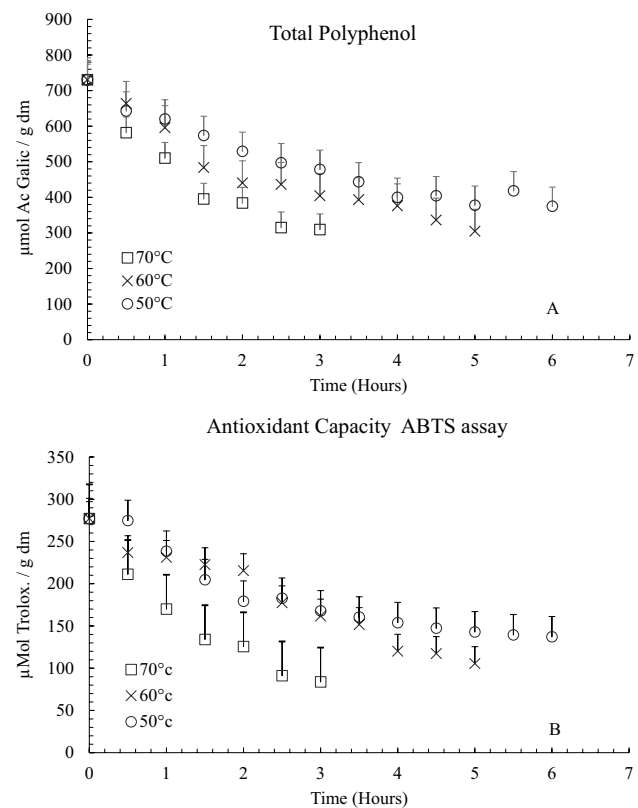


Fig. 2 Total polyphenol behavior (a) and antioxidant capacity (b) of SCG samples subjected to different process temperatures

stage of degradation to changes in the chemical structure of the antioxidant compounds.

The initial concentration of the ACGs was 2.095 mg ACGs/gdb , some studies have reported values between 4.78 and 13.24 mg ACGs/gDM [6, 41, 42]. On the other hand, the initial concentrations of 5-CQA, 3-CQA, 4-CQA, CA, 3,4-diCQA, 3,5-diCQA and 4,5-diCQA were 0.406, 0.897, 0.573, 0.005, 0.079, 0.075, 0.061 mg , respectively. In general terms, the temperatures of 70 and 60 °C decreased the concentration of the ACGs by 20 and 8% respectively; while the process carried out at 50 °C increased the concentration of the ACGs by 5%. In the specific case of caffeic acid, this increase 235, 392 and 352% for the temperatures of 70, 60 and 50 °C respectively.

These effects can be explained from the thermal instability of chlorogenic acids, which can undergo changes such as positional isomerization, epimerization, lactonization, hydrolysis and degradation too low molecular weight compounds [61]. It can be concluded that during the drying process at the established temperatures, the GCAs undergo structural changes due to positional isomerization and hydrolysis processes.

Conclusion

In general, it can be concluded that the hot-air drying was a suitable technique for processing waste generated in the coffee industry, since it allows obtaining a raw material with technological and functional properties with potential for application in the food industry and/or pharmaceutical industry. The optimization of the process yielded conditions of temperature, air velocity and cake thickness of 60 °C, 2.0 m/s, 0.012 m respectively, which allowed to retain 84.24% of total polyphenols and 66.00% of the initial antioxidant capacity of the initial sample. It was determined that the kinetics of degradation of chlorogenic acids is first order, due to hydrolysis reactions and positional isomerization. It can be said that the drying process is a good alternative for handling the residues of the coffee industry, since in addition to obtaining a raw material with beneficial health properties due to its antioxidant and chlorogenic acids content, in addition reducing environmental impact.

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Compliance with Ethical Standards

Conflict of interest The authors declare that have no conflict of interest.

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