

New powder material obtained from spent coffee ground and whey protein; Thermal and morphological analysis

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HIGHLIGHTS

- Spray dry process was a suitable technique for recovering agroindustry-waste.
- A novel raw material with potential application in food industry was obtained.
- A coating effect of whey protein concentrate over spent coffee ground was observed.
- Physical, structural, and thermodynamic properties were evaluated.
- The content of spend coffee did not significantly affect the variables analyzed.

GRAPHICAL ABSTRACT



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ABSTRACT

Currently, the food industry generates large amounts of agroindustry waste. Specifically, the coffee and cheese industry produce spent coffee ground and whey respectively. Therefore, it is important to find applications for these residues allowing the food industry to be friendly with the environment. The main goal of this research was to evaluate some morphological and thermodynamic properties of a powder product obtained by spray drying, from the mixture of Spent Coffee Ground (SCG) with Whey Concentrated (WC). The methodology includes the mixture of different combinations of SCG (4, 10 & 16%) with WC; afterward, the mixture was spray dried using an inlet temperature of 170 °C. The powder characterization includes: Moisture, Water Activity (A_w), Glass Transition temperature (T_g). In addition, isothermal curves were obtained. As a result, the moisture content and A_w of the powder were 3.25 ± 0.18 and 0.252 ± 0.01 , while the bulk density was 0.495 ± 0.10 g/cm³, showing a significant correlation ($p < 0.05$) with the particle size (43.87 μ m). Such an effect was confirmed by a Scanning electron microscope. The T_g values were in between 59.72 and 84.58 °C. Moisture-sorption isotherms were determined (17–45 °C) and the moisture content at the monolayer was between 0.043 and 0.086 kg of water/g dry basis, and the isosteric heat was -27.65 kJ/mol, which decreased as a function of the increase in moisture content. In general, it can be concluded that it is possible to obtain a powder, from the residues of the coffee and cheese industry; which has physical, thermal and morphological properties with high potential for application in the dairy industry.

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1. Introduction

Currently, the food industry generates large amounts of waste and by-products, which have a direct effect on the environment, as they produce greenhouse gases, in addition, to contamination of water sources, among other problems [1]. The by-products in the food industry are produced during different stages of processing, such as: harvesting, post-harvesting, processing and consumption processes [2]. Therefore, it is important to seek new alternatives for processing and utilization of the by-products. Then, allowing the development of new processes, products, raw materials and applications in different industries and thus reducing the environmental impact [3]. On the other hand, the by-products of the food industry are composed of carbohydrates, proteins, lipids and beneficial compounds for health, such as antioxidants, dietary fiber, polyphenols, collagen, carotenoids, among others [1]. Making them susceptible to transformation in different types of products; in this sense, the literature reports the procurement of different products from the agro-industrial by-products such as dietary fiber [4], polyhydroxyalkanoates [5], nutraceutical compounds [6], enzymes [7], extraction of biomolecules [8], among others.

Among important by-products of the food industry, are whey and the spent coffee ground (SCG), which are by-products generated in the production of cheese and instant coffee, respectively. Approximately 6 million tons of SCG [9] and 185 million tons/year of whey [10] are generated worldwide. Currently, these by-product are discarded in soils and bodies of water, which generates a negative impact on the environment [10]. The most common problems generated by this waste are damage to the soil, decrease in crop yield, increase in pollution and reduction in aquatic life [11–13].

Within the potential applications reported in the literature for whey appears the nutritional enrichment of foods [14,15], as an encapsulating agent [16,17] and in the production of foaming bases [18]. In the case of SCG, it is reported that it has beneficial effects for health [19,20] and has been used for the extraction of antioxidant compounds such as phenolic acids [21], kahweol and cafestol [22]. Among other applications, the use of coffee grounds as fertilizer has been reported [23] and in the formulation of products intended for animal feed [24].

Dehydration is one of the most common methods for processing agroindustry waste [4]; since it decreases water activity present in the by-products, assuring the conservation of the residues since the reactions that can affect them are inhibited [25]. Among the most common processes in the dehydration of agroindustry waste is spray drying, which is a process where an aqueous solution can be transformed into solid powdered products [26]. Spray drying is widely used in the production of different types of food such as: whey, coffee, milk, tea, and soups [16]. In the case of the transformation of by-products, there are reports of the use of spray drying in the use of olive leaves [27], jabuticaba by-product [28] and whey [18].

The aim of the present research was to evaluate physical, morphological and thermodynamic properties of a powder product obtained by spray drying, from the mixture of whey concentrated (WC) with different percentages of dried spent coffee grounds (SCG), with sights to obtain a functional powder that can be applied in different types of food or cosmetic industries. Then, the development of this ingredient contributes to the food industry having more efficient processes; reducing by-products that are generated through food processing, providing a process with low environmental impact.

2. Materials and methods

2.1. Sample

The SCG (grains obtained after the preparation of the coffee drink, 100% Arabica variety) was obtained from a cafeteria located at the University of Antioquia (Medellín, Colombia). Whey concentrate (WC) was obtained from a local dairy industry, Cooperativa Colanta

(Medellín, Colombia), which the following characteristics: protein content: 6.26 g/100 g, density: 1.32 g/cm³ and total solids: 39% (w/w).

2.2. Powder samples production

To produce the whey-spent coffee ground (WC-SCG) powder, different solutions of whey concentrate and spent coffee ground were processed in a pilot-scale spray-dryer (PSA 5 model, Vibrasec, Medellín, Colombia). Before the mixture, the SCG was dried and grinded to a final moisture of about 5% using a tray-type convection dryer [29]. The solutions were prepared by mixing concentrated WC with SCG (weight ratio of 96/4, 90/10, and 84/16% respectively). The mixing was conducted at 25 °C under constant agitation. Spray drying process was conducted with an inlet temperature of 170 °C and outlet temperature of 90 °C was applied [18]. The flow rate of the feed was 2.0 L/h and the pre-heated air had a flow rate of 540 L/h. The powder was immediately collected, vacuum-packed and put into the freezer at -20 ± 2 °C until further analysis.

2.3. Characterization of Whey-SCG powder

2.3.1. Physicochemical properties

The moisture content, water activity (A_w), apparent density, color and particle size were determined. Moisture content was determined according to the AOAC methodology [30]. Water activity (A_w) was obtained by the methodology described by Martinez-Saez et al. [9]. Apparent density was determined using the pycnometer method described by Caparino et al. [31]. Color of the samples was determined according to the CIELAB methodology, as described by Contreras-Calderón et al. [32]. The particle size distribution of the Whey-SCG powder was obtained from the velocity 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 μ m [18]. All analysis was run in triplicate ($n = 3$).

2.3.2. Oil holding capacity, formation and foam stability

The Oil holding capacity (OHC), was obtained by mixing 1 g of sample with 10 ml of Premier vegetable oil (L715Y2083, density: 0.91 g/mL) in vortex (Benchmark) for 30 min at 25 °C, then the mixture was centrifuged (Boeco C-28A) at 2500 rpm for 10 min. The supernatant (milliliters) was discarded and the results were expressed as g of oil/g of sample [4]. The formation capacity and foam stability were determined according to the methodology described by Osorio et al. [18]. To produce the foam, 4.00 g of the Whey-SCG powder was placed into a 250 mL beaker with 85 mL of distilled water at 80 ± 1 °C. The resulting solution was whipped with a mechanical blender (Hamilton Beach) for 2 min at a medium rate. After formation, the foam was added to a 100 mL graduated glass cylinder. The foaming capacity was obtained by measuring the height of the foam in the cylinder at 25 °C. The foam stability was evaluated according to the visual method Osorio et al. [18]. Finally, the collapse of the foam into the graduated glass cylinder was observed, and the volume of the liquid drained over time was recorded. Then, the time required to collapse all of the bubbles present in the foam was measured and considered to represent the stability time of the foam. Triplicate analyses of each sample were carried out ($n = 3$).

2.3.3. Scanning electron microscope SEM

Morphological analyses were conducted using a scanning electron microscope SEM-Quanta 200F Environmental Scanning Electron Microscope (FEI, Field Emission Instruments, Hillsboro, Oregon, USA) in the Centro de Microscopía Avanzada – CAM (Universidad de Antioquia, Medellín, Colombia). In addition, representative micrographs were taken at 40 $\times$, 100 $\times$, 300 $\times$ and 1000 $\times$ magnification in order to visualize the surface morphology. Triplicate analyses of each sample were carried out ($n = 3$).

2.3.4. Determination of thermal properties by differential scanning calorimeter (DSC)

Thermal properties of the Whey-SCG powder were measured using differential scanning calorimetry (DSC, TA Instruments, model Q100), under UHP nitrogen atmosphere conditions. DSC was determined following the method of Farrokhi et al. [33] with slight modifications. Then, the samples were subjected to thermal erasure, with a heating ramp of 20 °C/min from 25 °C to 100 °C, then cooling to −40 °C maintaining the temperature for 5 min, then proceeding with the analysis from −40 °C to 200 °C at a rate of 10 °C/min. The glass transition temperature (T_g), melting temperature (T_m) and melting enthalpy (ΔH_m) were measured, using a specialized software. Triplicate analyses of each sample were carried out ($n = 3$).

2.4. Zeta potential (ZP)

Zeta potential (ZP), was measured in a ZetaSizer Malvern Instruments (UK) system. This unit automatically calculates electrophoretic mobility (EM), and then ZP according to the moluchowski's equation:

$$ZP = 4\pi \frac{n}{D} EM \quad (1)$$

where n and D , are the viscosity in poise and the dielectric constant of the suspending liquid, respectively. Measurements were performed at the natural pH of the sample. Triplicate analyses of each sample were carried out ($n = 3$).

2.5. Moisture-sorption isotherms and thermodynamic properties

To obtain the moisture-sorption isotherms, the Dynamic Dewpoint Isotherm (DDI) method was used, using an AquaLab Vapor Sorption Analyzer (VSA), under three temperature conditions (17, 26 and 45 °C). The experimental data obtained in the moisture-sorption isotherms were adjusted to the mathematical model Guggenheim-Anderson-de Boer (GAB), equation (2) [34]. The adjustment of the experimental data was evaluated by the determination of the R^2 coefficient and the statistical parameters quadratic mean error (RMSE) and reduced Chi-square (χ^2), the previous according to Largo-Avila et al. [35].

$$X_w = \frac{X_o C K a_w}{(1 - k a_w)(1 + (C - 1)K a_w)} \quad (2)$$

For thermodynamic properties, the net isosteric heat of sorption (q_{st}) or differential heat of adsorption (ΔH) was obtained by addition of total heat of sorption (Q_{st}) from the vaporization enthalpy of water (λ). The Clausius-Clapeyron equation (Equation (3)), was applied to the temperature-vapor pressure data of the product.

$$q_{st} = \Delta H = RT^2 \left[\frac{\partial \ln P}{\partial T} \right]_{X_w} = Q_{st} + \lambda \quad (3)$$

where P is the pressure of water vapor in equilibrium with the material at an absolute temperature, X_w is the equilibrium moisture content in kg/kg dry solid, R is the universal gas constant, Q_{st} is the total isosteric heat of sorption and λ is the heat to vaporization of pure water. The heat of vaporization of pure water (λ) can be calculated by:

$$\lambda = R[6887 - 5.31 \cdot T_K] \quad (4)$$

The Gibbs free energy (ΔG) and entropy (ΔS) were calculated for the adsorption process by applying equations (5) and (6), respectively.

$$\Delta G = RT \ln \frac{P}{P_0} = RT \ln a_w \quad (5)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (6)$$

2.6. Statistical analysis

To analyze the data, a completely randomized design was made, taking as independent variable the percentage of dry spent coffee ground with three levels (4, 10 and 16%) and as dependent variables: moisture content, water activity (A_w), apparent density, color, particle size, oil holding capacity, Zeta potential, and formation capacity and foam stability. Each treatment was done by triplicate. To determine if there were significant differences, an ANOVA variance analysis was performed with a level of significance of 95% and to determine the difference between the treatments, a multiple range test was performed by means of a Fisher LSD test. The results are expressed as mean $\pm$ standard deviation (SD). The Shapiro-Wilk test was applied to determine the normality. All statistical analyzes were performed using STATGRAPHICS Centurion XVI software, (Statistical Graphics Corporation, Version 16.0.07, Rockville, USA).

3. Results

3.1. Physicochemical properties

In Table 1, the values of response variables: moisture content, A_w , density, color, particle size, oil retention capacity, foam formation, foam stability, and Zeta potential can be observed, corresponding to the different treatments of the Whey-SCG samples obtained after the spray drying process. In addition, in Fig. 1, the different LSD analyzes can be observed for the variables analyzed.

In general, the moisture content of the samples ranged between 2.746 and 3.746 g/100 g; while the A_w had an average value of 0.252, with a range of 0.204 and 0.319. Regarding the statistical analysis, it was found that the SCG percentage did not present a significant incidence ($p > 0.05$), on the moisture content and A_w (Fig. 1a and b). These moisture values and A_w achieve a microbiologically stable powder, because the availability of free water is lower, preventing the growth of microorganisms and also chemical reactions [36]. On the other hand, the moisture content and A_w , Table 1, are in close agreement with the values reported by different authors for food matrices subjected to spray drying processes. Felix da Silva et al. [37] reported values of 3.6–4.2% in powdered cheese samples obtained by spray drying. Nishanthi et al. [38] reported moisture values between 3.9 and 5.7% in different types of serum. Bazaria and Kumar [39] report values between 2.81 and 4.37% for samples of beet juice using encapsulating agent concentrated whey. Furthermore, Osorio et al. [18] reported moisture contents between 1.69 and 2.14 g/100 g, for samples of a foaming base obtained from whey.

On the other hand, in Table 1, it can be seen that the data of the apparent density of the powder ranged between 0.434 and 0.563 g/cm³ and its average value was 0.495 g/cm³. Of these values, it could be said that the product can present good behavior at the time of packaging and stability in storage. This stability is due to the fact that products with high densities have a lower volume of air inside the matrix, which delay oxidation processes [40]. Goula and Adamopoulos [41] reported values between 0.199 and 0.365 g/cm³ in samples of tomato pulp processed by spray dry. Oliveira et al. [36], reported values of apparent density between 0.154 and 0.202 g/cm³, for a powder obtained from whey with grape skin. These results are lower than those found in the current investigation, this phenomenon is due to the fact that the drying temperatures were higher, which in turn generates a lower density. However, the values are in agreement with Bhandari et al. [42] who reported that the apparent density of some food product such as milk, chocolate and flour ranged between 0.520 and 0.590 g/cm³. Finally, in Fig. 1C, it can be observed that the apparent density was not significantly influenced ($p > 0.05$) by different percentages of SCG.

With respect to the particle size, the values obtained ranged between 35.20 and 53.30 μ m, these values are in accordance with that reported in

Table 1

Results of the experimental design of the spray drying of the different mixtures of whey concentrate and spent coffee ground.

No	Solution % SCG	Moisture g/100 g of sample	A _w	Density g/cm ³	Color L*	Particle Size microns	OHC g oil/g sample	Foam Formation mL	Foam Stability min	Zeta Potential mV
1	10	3.405 ± 0.33	0.233 ± 0.01	0.481 ± 0.10	58.97 ± 3.75	39.8 ± 0.04	1.06 ± 0.10	6.00 ± 0.10	5.24 ± 0.10	-29.0 ± 5.28
2	16	3.223 ± 0.15	0.256 ± 0.03	0.447 ± 0.10	57.54 ± 1.93	53.2 ± 0.24	0.94 ± 0.10	4.50 ± 0.80	2.75 ± 0.26	-25.8 ± 4.62
3	16	2.771 ± 0.10	0.204 ± 0.06	0.440 ± 0.10	55.57 ± 1.93	53.3 ± 0.20	1.90 ± 0.10	4.50 ± 0.50	2.54 ± 0.45	-26.0 ± 4.51
4	10	3.000 ± 0.11	0.235 ± 0.01	0.434 ± 0.10	54.04 ± 3.75	43.2 ± 1.40	2.12 ± 0.10	4.26 ± 0.25	2.92 ± 0.34	-26.4 ± 4.31
5	4	3.238 ± 0.38	0.223 ± 0.01	0.477 ± 0.10	59.92 ± 5.84	42.2 ± 0.19	1.78 ± 0.10	3.55 ± 0.45	2.22 ± 0.15	-25.1 ± 4.21
6	4	3.746 ± 0.18	0.284 ± 0.01	0.533 ± 0.10	71.30 ± 5.84	40.2 ± 1.83	1.72 ± 0.10	3.00 ± 0.50	1.40 ± 0.06	-22.2 ± 3.75
7	10	3.419 ± 0.06	0.213 ± 0.01	0.527 ± 0.10	61.94 ± 3.75	35.2 ± 1.57	2.14 ± 0.10	4.66 ± 0.28	4.34 ± 0.07	-16.7 ± 4.28
8	4	2.746 ± 0.40	0.306 ± 0.01	0.563 ± 0.10	67.76 ± 5.84	45.4 ± 0.55	2.12 ± 0.10	5.50 ± 0.26	5.12 ± 0.10	-20.5 ± 4.26
9	16	3.711 ± 0.32	0.319 ± 0.01	0.560 ± 0.10	59.43 ± 1.93	42.4 ± 1.27	2.03 ± 0.10	4.70 ± 0.28	4.30 ± 0.11	-22.8 ± 5.30

OHC: Oil holding capacity.

different food matrices subjected to spray drying process, such as skimmed milk [43], foaming bases [18], instant coffee [44]. Although, the particle size was not influenced by the percentage of SCG ($p > 0.05$) (Fig. 1E), this presented a high and significant correlation ($p < 0.05$, $r = 0.80$) with the bulk density (according to the analysis of Pearson correlation), this phenomenon is due to small particles of material occupying spaces between large particles, increasing the apparent density of the system [36]. In addition, the particle size had an average value of 43.87 μm . This value allows the product to have good handling and reconstitution properties; however, some authors report that particle sizes below 50 μm , compromise the stability of the product, because it has a greater surface area exposed to environmental conditions, allowing the degradation and oxidation of sensitive compounds [45–47]. This effect is counteracted with high-density values, because as mentioned above, high-density values decrease the contact of the product with the environment, delaying degradation reactions.

As for color parameters, in Table 1 it is observed that the L parameter ranged between 54.04 and 71.30 and on average it had a value of 60.71. Likewise, it was determined that the SCG percentage factor did not present a significant incidence ($p > 0.05$) on the color (Fig. 1D). However, when the percentage of SCG in the mixture increases, the luminosity of the powder decreases; which is expected due to the high coloration of SCG, which is due to the presence of compounds like melanoidins. Which are compounds of high molecular weight, having effects on color and antioxidant activity [48].

3.2. Oil holding capacity, formation and foam stability

The oil retention capacity of the samples varied between 0.94 and 2.14 g of oil/g of sample. The percentage factor of SCG has not had significant importance ($p > 0.05$) on the capacity of oil retention. On average, mixtures of 4, 10 and 16% of SCG providing oil retention capacities of 1.87, 1.77 and 1.62 g of oil/g of sample respectively, which indicates that the greater the amount of oil is retained the lower the SCG content. This behavior was not as expected, since the SCG is a by-product that contains polysaccharides such as cellulose (12%), hemicellulose (39%) and lignin (23%) [49], which supposedly have the property of increase oil retention [50], therefore, it was expected that the greater the contents of SCG would allow a greater oil retention. However, it is well known that the application of thermal processes in solutions rich in globular proteins, as is the case of the present investigation, causes the degradation of them, generating their splitting; allowing a greater exposure to hydrophobic active sites [51]. According to the above, with a low percentage of SCG, the action of the whey as an encapsulating agent is lower, leaving active hydrophobic sites that can increase the oil retention capacity.

In general, the average value found for the oil retention capacity was 1.75 g oil/g of sample, and it is higher than that reported for other food matrices such as fiber concentrates of fruit residues (0.60–1.81 g of oil/g of sample) [52], orange waste extracts (0.86–1.27 g of oil/g of sample) [53]. According to the values found in the present research, the powder

could be used for the stabilization of products with high-fat content and emulsions [50] or in the development of foods that help the digestive transit [4].

On the other hand, the capacity of foam formation and stability of different samples of powder ranged between 3.0 and 6.0 mL and 1.4–5.24 min respectively, where, the SCG percentage factor did not present significant incidence ($p > 0.05$) on foam formation and stability parameters. However, 10% percentage of SCG was the better combination in terms of formation properties and foam stability, with average values of 4.98 mL and 4.20 min respectively. The values of foam formation and stability were at 4.52 mL and 3.42 min. These values are in close agreement with those found in a previous study [18].

3.3. Scanning electron microscope SEM

Fig. 2 shows the images of the dehydrated spent coffee ground and the powders obtained from the Whey-SCG mixtures (4, 10 and 16% of SCG). In Fig. 2a and b, the structure of the dehydrated SCG can be seen, these samples had a porous structure with flat leaf shapes, which is similar to that reported by Ballesteros et al. [49].

In the case of samples of Whey-SCG powder (Fig. 2 C-H), are depicted a large number of spherical particles agglomerates of different diameters and smooth surface. This phenomenon is due to the effect of the inlet temperature and the type of atomizer used; because the use of low temperatures (140 °C), generate particles with wrinkled surface; on the contrary, high process temperatures (180 °C), produces smooth and agglomerated surfaces [46].

Likewise, the micrographs confirm the correlation between the density and the particle size previously found, since it shows the space occupied by small particles between the free spaces that exist between the large particles, which increases the apparent density of the product [54]. To the foregoing, the particles of larger size and asymmetric shape of the micrographs D, F and H (Fig. 2), correspond to SCG particles, said particles present smooth and plane surface; which contrasts with the images obtained from the SCG samples (Fig. 2A and B). This phenomenon is due to the ability of the whey to form a film around other compounds during drying, fulfilling a protective role over them [55]; in the case of the present research the coffee particles and therefore their antioxidant compounds. Similar results have been reported by different authors for size, shape, agglomeration and encapsulation [17,54, and 55].

3.4. Thermal properties by differential scanning calorimeter (DSC)

In Table 2 are depicted the results for the glass transition temperature (T_g), melting temperature (T_m) and melting enthalpy (ΔH_m), for different samples of Whey-SCG powder by DSC analyzes.

Table 2 shows the glass transition temperature (T_g) (endothermic phase) 59.72, 84.37 and 84.58 °C; with an enthalpy change of 30.45, 33.95 and 24.28 J/g for the samples containing 4, 10 and 16% of SCG respectively. These values are higher than the reported for different

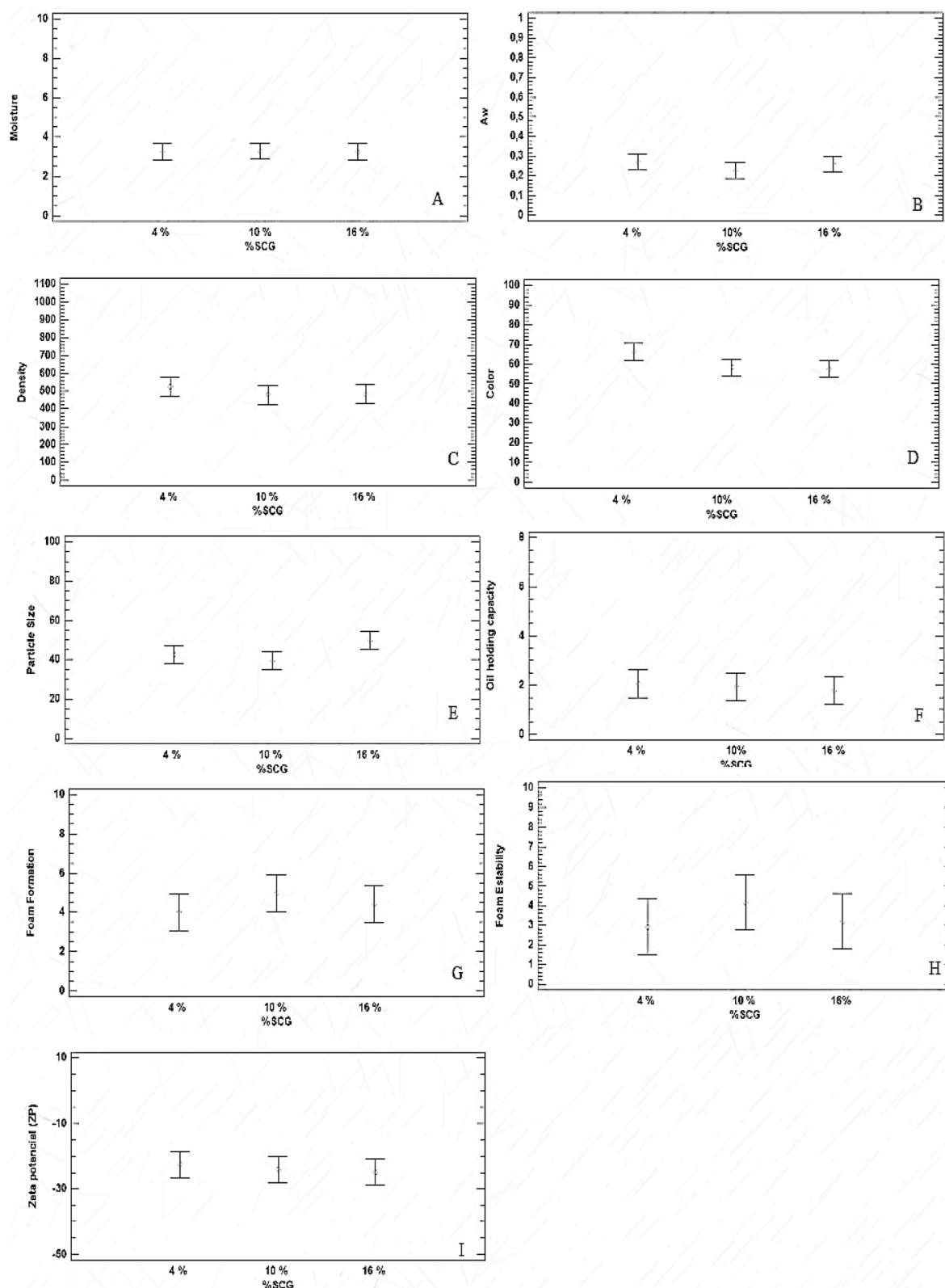


Fig. 1. Fisher LSD interval charts with 95% confidence, of the different variables analyzed, where: A: Moisture, B: Water activity (A_w). C: Density, D: Color, E: Particle size, F: Oil holding capacity, G: Foam formation, H: Foam stability and I: Zeta potential.

dietary matrices formulations from whey, such as infant formulas (57.5 °C) [58], lactose mixtures with whey isolate protein (WPI) (40–41 °C) [59], lactose/WPI systems (42.6–45.3 °C) [60]; as for the SCG samples (76.89 °C) [49]. The above results are due to the composition of

the mixture (WC-SCG) since both the whey and the SCG are systems with macromolecular components of high molecular weight such as proteins, in which, the transition occurs at high temperatures [60], causing the increase of T_g on the system [61]. These T_g values, allows the powder

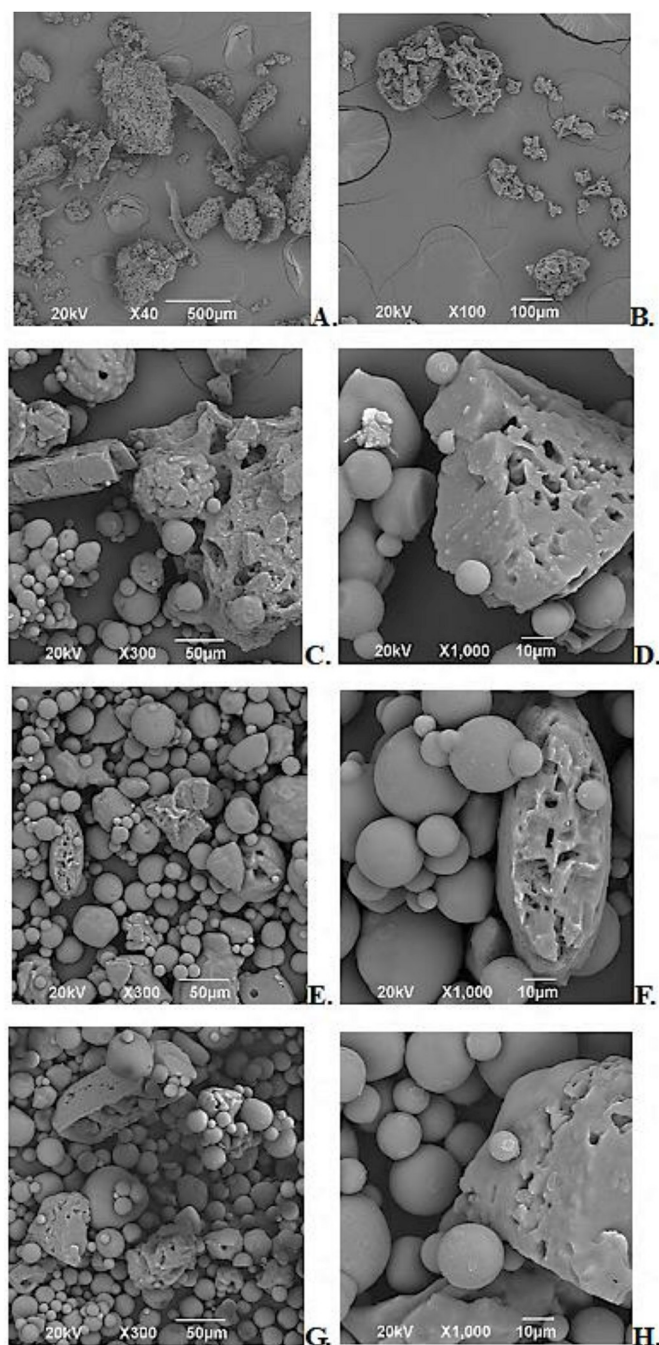


Fig. 2. Scanning electron microscope (SEM) images of powder by mixtures of whey-spent coffee ground (Whey-SCG). A–B: dry SCG; C–D: mixture 4% SCG; E–F: mixture 10% SCG; G–H: mixture 16%SCG. Magnification: $\times 40$, $\times 100$, $\times 300$ and $\times 1000$.

Table 2

Differential Scanning Calorimeter (DSC) of the spray drying samples for the different mixtures of whey concentrate and spent coffee grounds.

%SCG	T_g (°C)	T_f (°C)	Enthalpy (J/g)
4	59.72	149.43	30.45
10	84.37	147.34	33.95
16	84.58	148.49	24.28

conservation at room temperature, without chemical changes. Changes occur when storage temperatures exceed T_g [62].

3.5. Zeta potential (ZP)

The Zeta potential of the Whey-SCG powder samples varied between -29.00 and -16.70 mV, which suggests the affinity of the samples for substrates with a positive surface charge. However, when whey and SCG samples were analyzed separately, negative potentials were obtained, -26.5 and -26.8 respectively. This result reveals repulsive interactions, which does not favor electrostatic interactions when they are integrated separately into a formulation. The SCG percentage factor did not present significant incidence ($p > 0.05$) on the Zeta potential. Nevertheless, the mixtures of 4, 10 and 16% of SCG gave average values of -24.9 , -24.0 and -22.5 mV respectively. The results suggest that the Whey-SCG samples had sufficient electrostatic repulsion to avoid attractive interactions [63]. On the other hand, the interactions between the materials (WC-SCG), could be dominated by strong cohesion forces and supramolecular interactions (Vander walls interactions, hydrogen bridges, and interactions of London or induced dipoles) between the functional groups of both matrices.

3.6. Moisture-sorption isotherms and thermodynamic properties

Table 3 shows the parameters of Gab Model for monolayer moisture content (X_m) and the constants C and K at three different temperatures.

The moisture-sorption isotherms of spray-dried Whey-SCG powder, were determined at three different temperatures (17, 26 and 45 °C) (Fig. 3). The GAB model was selected to describe the moisture adsorption isotherms of the Whey-SCG powder. The GAB provides the value of monolayer moisture content of the material. Where this value is considered as the safe moisture content for dried foods during preservation [64]. The value of monolayer moisture content ranged between 0.043 y 0.086 kg of water/g dry basis (Table 3). The values of the monolayer moisture contents (X_m) decreased as the temperature increased. This behavior has been observed in sugar-rich foods [35]. Similar behavior has been reported by some authors [65]. On the other hand, moisture sorption isotherms could be classified as a type III isotherm [66]. In the zone ($0.3 < a_w < 0.70$) (Fig. 4), the course of isotherms reveals some inflections formed, most likely, as a result of moisture-induced phase transitions [67]. For A_w above 0.7 the equilibrium moisture content increased rapidly at all temperatures (Fig. 4). In this region excess of water is capable of acting as a solvent [68], which is indicative of water migrating into the bulk of the powder as it approaches a glass transition region, prior to the crystallization process, according with Kelly et al. [69]. The intersection of the isotherm was observed at the A_w of approximately 0.7–0.8. Similar results of the intersection of the isotherms, were previously reported for dried fruits and different sugar-rich products [67,70].

Fig. 4 shows the trend of the net isosteric heat of sorption (q_{st} or ΔH) and the thermodynamic properties of sorption for Whey-SCG powder using the GAB model and the Clausius-Clayperon intervals based on temperature and moisture content. Where the moisture sorption isotherms have been used to determine the net isosteric heat of sorption. The net isosteric heat of sorption is the amount of extra energy that is required to evaporate water from the solid material [65]. The results show a negative quantity, which illustrates the exothermic character of

Table 3

GAB model parameters calculated at different temperatures.

GAB parameters.	17 °C	26 °C	45 °C
X_m	0.0865	0.0618	0.0439
C	1.1774	2.3780	8.1218
K	0.7800	0.8247	0.8761

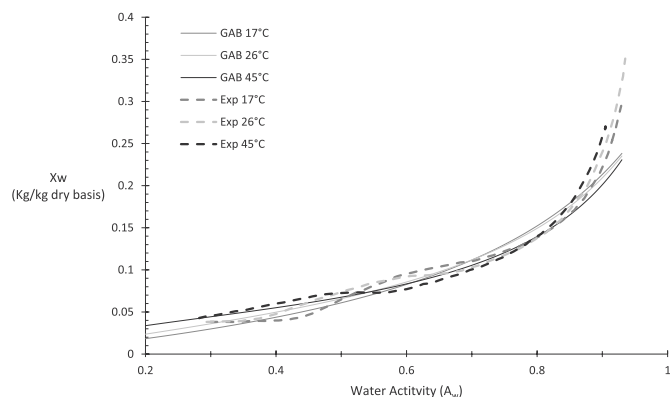


Fig. 3. Sorption isotherms of spray-dried Whey-SCG powders and the fitted GAB model at the three different temperatures. Whey-SCG powders kept at 17 °C, 26 °C, and 45 °C.

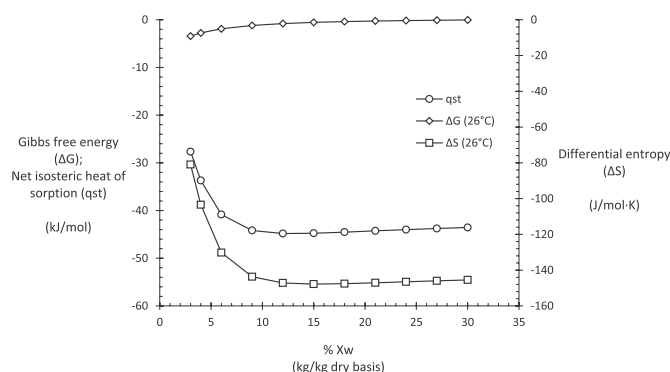


Fig. 4. Net isosteric heat of sorption and the thermodynamic properties of sorption of Whey-SCG powder at different moisture contents.

the process (adsorption). The net isosteric heat of sorption decreases as the moisture content increases from -27.65 kJ/mol for 3% dry basis to approximately -43.56 kJ/mol for moisture content of 30% dry basis. The decrease in the isosteric heat with the increase in adsorbed water content may be attributed to the fact that sorption initially occurs on the most active sites such as hydrophilic polar groups giving rise to the greatest interaction energy [65,71].

On the other hand, the thermodynamic properties of sorption for Whey-SCG powder (Fig. 4). The tendency showed by Gibbs free energy (ΔG) explains that water absorption was a spontaneous phenomenon, and the differential entropy (ΔS) is plotted as a function of moisture content where an increase in randomness is indicated by positive values for entropy change, a similar tendency was reported by Largo-Avila et al. [35].

4. Conclusions

By-products such as SCG and whey are a good alternative for the development of new raw materials with attractive technological properties for the application in the food industry. In addition, the whey had a protective effect on the SCG particles, which allows the preservation of high value compounds. Whey-SCG powder, presents good technological properties in terms of density, moisture, A_w , oil holding capacity, which favors the use of powder such raw material in the food industry. The content of SCG did not significantly affect the variables analyzed. According to the characteristics and technological properties found for Whey-SCG powder, the novel ingredient could be used as a raw material for the stabilization of products with high-fat content, emulsions, foods that require foam formation, some products like yogurt, ice cream, and

bakery products. As well as in the development of functional foods. Future studies should focus on the application of this product.

Conflicts of interest

The authors declare that have no conflict of interest.

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Appendix A. Supplementary data

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