LWT - Food Science and Technology 129 (2020) 109523

Contents lists available at ScienceDirect

LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

Rheological, texture, structural, and functional properties of Greek-style yogurt fortified with cheese whey-spent coffee ground powder

Juan Osorio-Arias[a,b], Angela Pérez-Martínez[a], Oscar Vega-Castro[a], Sergio I. Martínez-Monteagudo[c,*]

[a] BIOALI Research Group, Faculty of Pharmaceutical and Food Science, Universidad de Antioquia, Medellin, Colombia
[b] Aoxlab Research Group, Aoxlab S.A.S., Development and Innovation Department, Medellin, Colombia
[c] Dairy and Food Science Department, South Dakota State University, South Dakota, Brookings, SD, USA

ARTICLE INFO

Keywords:
Spent coffee ground
Rheological properties
Texture
Microstructure
Fortification

ABSTRACT

The present study evaluated the feasibility of the fortification of Greek-style yogurt with a newly developed ingredient consisting of cheese whey-spent coffee ground (CW-SCG) powder. The yogurts were analyzed using a battery of tests, including whey production, water holding capacity, firmness, rheological properties, protein content, available lysine, and antioxidant activity. The milk base was fortified to 15% dry matter with different skim milk powder to CW-SCG ratios (100/0, 75/25, 25/75, and 0/100 wt/wt.). Fortification of the yogurt with CW-SCG decreased the water holding capacity by 13–25%, depending on the level of fortification. Similarly, the addition of CW-SCG significantly decreased the hardness of the yogurts from 76.57 ± 1.18 to 30.27 ± 1.73 N s. Additionally, fortified yogurts with CW-SCG yielded a product with less shear-thinning behavior as compared to control. The microstructure of the fortified yogurts revealed the incorporation of spent coffee ground particles within the protein network. Polyphenol-protein associations may explain the texture and rheological behavior of the yogurts. The fortification of the yogurt up to 25% of CW-SCG yielded comparable properties than the control.

1. Introduction

Yogurt is a semi-solid food, produced by lowering the pH of milk proteins to their isoelectric point by the fermentation of lactose using lactic acid bacteria, such as *Lactobacillus delbrueckii* and *Streptococcus thermophilus* (Tamime & Robinson, 2007). The global market of yogurt has increased from about 77 billion U.S. dollars in 2016 to over 86 billion U.S. dollars in 2019, and it is projected to exceed 100 billion by 2023 (Prasannan, 2017). Likewise, the production of Greek-style yogurt is forecasted to be on the rise due to its robust adaption in a variety of drinks, frozen, and bakery products. Greek-style is characterized by high solid content (mostly proteins) that provides appealing nutritional and organoleptic properties. A key processing step is the fortification of the milk base since it influences the final texture, rheology, morphology, functional, and nutritional properties (Karam, Gaiani, Hosri, Burgain, & Scher, 2013).

Fortification of the yogurt base with whey proteins is one of the most common approaches to manufacture high-quality Greek-style yogurt, resulting in reduced syneresis and increased firmness (Mahomud, Katsuno, & Nishizu, 2017; Sodini, Remeuf, Haddad, & Corrieu, 2004; Tamime & Robinson, 2007). Over the last few years, isolates and concentrates of milk proteins have been used for the fortification of the milk base (Karam et al., 2013). In summary, the existing literature suggested the fortification by creative combinations of different solids offers new strategies for the manufacture of Greek-style yogurt. For instance, Brückner-Gühmann, Benthin, and Drusch (2019) fortified the milk base with oat protein concentrate. These authors reported that the addition of oat protein concentrate lowered the rate of syneresis compared with the control, milk base fortified with skim milk powder. Similarly, the fortification of the milk base with chickpea flour (up to 2%) as a source protein showed no significant difference in the sensory scores compared with the control yogurt (Chen, Singh, Bhargava, & Ramanathan, 2018). Hashim, Khalil, and Afifi (2009) evaluated the addition of date fiber on the texture profile and sensory properties of yogurt. It was found that the addition of date fiber up to 3% yielded firmer texture and similar overall acceptance when compared with the control. Ozturkoglu-Budak, Akal, and Yetisemiyen (2016) fortified the milk base with dried nuts (walnut, hazelnut, almond, or pistachio), and found that higher protein content and lower syneresis was obtained than the control yogurt. The fortification of milk

* Corresponding author. Dairy and Food Science Department, South Dakota State University, Brookings, SD, 57007, USA.
E-mail address: Sergio.MartinezMonteagudo@sdstate.edu (S.I. Martínez-Monteagudo).

https://doi.org/10.1016/j.lwt.2020.109523
Received 12 February 2020; Received in revised form 28 April 2020; Accepted 29 April 2020
Available online 15 May 2020
0023-6438/

base with different solids should consider the effect of such solids on a number of quality tests, including fermentation, texture, whey production, sensory, and others. Factors such as particle size and chemical composition play a crucial role in the development of texture and overall quality. The present research aimed at evaluating the feasibility of the fortification of a type-set yogurt with a newly developed ingredient consisting of cheese whey-spent coffee (CW-SCG) ground powder (Osorio-Arias et al., 2020). The feasibility was evaluated in terms of syneresis, water holding capacity, firmness, hardness, rheological properties, protein content, available lysine, and antioxidant activity. The fortification of a type-set yogurt with a newly developed ingredient offers opportunities for the utilization of agricultural by-products. The majority of Latin America dairy industry does not have the technology to recover the whey proteins from cheese whey, and the creation of value is of great relevance. The combination of SCG and CW also provides important nutritional compounds such as protein, carbohydrates, fiber, and antioxidant compounds. Additionally, the current research helps to reduce the carbon footprint and therefore the impact on the environment generated by this kind of by-products of the food industry.

2. Materials and methods

2.1. Chemicals

Acetonitrile (≥99.8%), methanol (≥99.8%), hydrochloric acid (37%), and glacial acetic acid (≥99.8%) were purchased from Panreac Quimica S.L.U. (Barcelona, Spain). Reagent Folin-Ciocalteu (2.0 N, Merck Group, Darmstadt, Germany), gallic acid (≥97%, Sigma-Aldrich, Oakville-Ontario, Canada), standard N-e−2,4-DNP-L-lysine (≥98.9%, Sigma-Aldrich), and 1-fluoro-2,4-dinitrobenzene (FDNB) solution in 3% ethanol (v/v) were purchased from a commercial supplier.

2.2. Manufacturing procedure

2.2.1. Cheese whey-Spent coffee ground powder

The cheese whey-spent coffee ground powder was manufactured according to the methodology reported by Osorio-Arias et al. (2020). Concentrated cheese whey (~39% total solids) was obtained from a local cheese factory (Cooperativa Colanta, San Pedro de los Milagros, Colombia). A predetermined portion (10%) of the spent coffee ground (SCG) was mixed with the concentrated cheese whey. Then, the solution was processed in a pilot-scale spray-dryer (PSA 5 model, Vibrasec, Medellín, Colombia), following the conditions reported elsewhere (Osorio et al., 2020). The CW-SCG was mainly composed of carbohydrates (78.65 ± 0.16%), protein (10.69 ± 0.08%), ashes (4.91 ± 0.01%), and fat (0.73 ± 0.21%).

2.2.2. Non-fat Greek-style yogurt

Skim milk obtained from the Davis Dairy Plant at South Dakota State University (Brookings, SD) was fortified to 15% dry matter with different skim milk powder to CW-SCG ratios (100/0, 75/25, 25/75, and 0/100 wt/wt). The resulting samples were designed as x-CW-SCG with x refers to the weight percent of CW-SCG. Then, the fortified base was heated at 90°C for 10 min and subsequently cooled down to the fermentation temperature (43°C), followed by the addition of the starter culture (0.03% wt/wt, *Lactobacillus delbrueckii* subsp. *bulgaricus* and *Streptococcus thermophilus,* YC-X11 Yo-Flex, Chr. Hansen, Denmark). The inoculated base was then mixed thoroughly for 5 min to acclimate the starter culture. Afterward, the yogurt base was transferred into different 500 mL plastic containers. All containers were incubated at 43 °C in a water bath until the pH reached 4.6. The time needed to achieve a pH of 4.6 varied from 230 to 300 min.

2.3. Analytical determinations

2.3.1. Total polyphenol content and antioxidant capacity

For each treatment, an aliquot of 1.0 g was clarified with 3 mL of Carrez-I (15% potassium ferrocyanide) and 3 mL of Carrez-II (40% trichloroacetic acid). Then, the clarified samples were dissolved in 20 mL of ethanol:water (50:50) and 15 mL of $G' = a{\cdot}\omega^b$ acetone:water (60:40) for 60 min at room temperature. The solution was then centrifuged, and the supernatant was recovered for the determination of total polyphenol content (TPC) and antioxidant capacity. For the total polyphenols, the Folin-Ciocalteu test was used following the guidelines reported elsewhere (Osorio-Arias et al., 2020). The antioxidant capacity was determined using the ABTS-assay (2,2′-Azino-bis-3-ethylbenzthiazolin-6-sulfonic acid), following the methodology described by Contreras-Calderón et al. (2016).

2.3.2. Total nitrogen content and available lysine

The total protein content was determined using the Kjeldahl analysis (AOAC 920.105), while the available lysine was determined following the methodology reported by Contreras-Calderón et al. (2016).

2.4. Physical determinations

2.4.1. Whey production and water holding capacity

The whey production (WP) of the yogurts was measured gravimetrically, following the methodology reported elsewhere (Lee & Lucey, 2004). The expelled whey from the yogurt after 24 h of storage at 4°C was carefully removed and weighed. The water holding capacity (WHC) of the yogurt was measured gravimetrically following the methodology described by Mahomud et al. (2017). Samples of 25 g were centrifuged (2500 rpm) for 20 min at 4°C. The expelled liquid was removed and weighed.

2.4.2. Textural properties of the yogurt

A TA.XT plus texture analyzer (Texture Technologies Corp. and Stable Micro Systems, Ltd. Hamilton, MA) with a TA-4 load cells (38 mm diameter) was used to measure the firmness, hardness, and adhesiveness of the yogurts. Each yogurt was contained in a plastic cup (120 mm high and 70 mm diameter). The yogurt samples were penetrated vertically to a depth of 20 mm at a speed of 1 mm s^{-1} and retracted to its original position at the same speed. The maximum force required to break the gel was used as a measure of the firmness of the yogurt. The positive and negative area under the curve (N.s) were related to hardness and adhesiveness, respectively (Lesme et al., 2019).

2.4.3. Rheological properties

An MCR 92 rheometer (Anton Paar, GmbH, Ostfildern, Germany) equipped with a plate and plate geometry (PP25/S, diameter 25 mm) was used to characterize the rheological behavior of the produced yogurts. Three types of tests were performed: i) strain sweep from 0.01 to 100% at a constant frequency of 1 rad s^{-1}; ii) frequency sweep from 0.1 to 100 rad s^{-1} at a constant strain of 0.5%; and iii) flow behavior from 0.1 to 100 s^{-1}. Samples were tested at 4°C. The mechanical spectra of the formulated yogurts were obtained by plotting storage module (G′) and loss module (G″) as a function of frequency (ω) in a double logarithmic plot. The relationship between G′ and ω was characterized using Equation (1):

$$G' = a{\cdot}\omega^b \tag{1}$$

where a and b are regression coefficients obtained through non-linear regression analysis performed using the Solver option in Excel Microsoft. The flow behavior was characterized by the Herschel-Buckley model (Equation (2)) was fitted to the upward flow curve.

$$\sigma = \sigma_0 + \kappa{*}\dot{\gamma}^{\eta} \tag{2}$$

where σ_0 represents yield stress (Pa), κ is consistency index ($Pa.s^n$), $\dot{\gamma}$ is shear rate (s^{-1}), and η is a dimensionless number known as flow behavior index (n = 1 for Newtonian fluids, n < 1 for pseudoplastic, and n > 1 for dilatant).

2.4.4. Scanning electron microscope

The microstructure of the produced yogurts was determined using scanning electron microscope (model S–3400N Hitachi, Ltd. Tokyo, Japan) at the Electrical Engineering and Computer Science Department at South Dakota State University.

2.5. Experiments design and statistical analysis

Each experimental data point was performed in triplicates. Data were expressed as mean ± standard deviation of three replicates. Comparison of mean values was performed with one-way analysis of variance using Tukey's post hoc ($p < 0.05$). All figures and statistical analyses were made using Sigmaplot software V11 for Windows (SPSS Inc., Chicago, IL, USA).

3. Results and discussions

3.1. Effect of CW-SCG on the acidification profile

Fig. 1 shows the effect of the fortification with different levels of CW-SCG on the acidification of Greek-style yogurt. In general, the acidification curves were sigmoidal in shape, resembling a typical acidification curve for Greek-style yogurt (Tamime & Robinson, 2007). The addition of CW-SCG up to 75% did not significantly change the acidification curve when comparing with the control sample, reaching the target pH 4.7 after 270–300 min. The exception to this generalization was observed in those samples fortified with 100-CW-SCG, where the time needed to achieve the target pH 4.7 was significantly shortened, 225 min. Similarly, do Espírito Santo, Perego, Converti, and Oliveira (2012) reported an overall acceleration of the fermentation by the addition of passion fruit peel powder due to the presence of phenolic compounds. The fortification of the yogurt base with 100-CW-SCG powder contained 23.11 ± 2.01 mg of GAEs per 100 g^{-1} (Table 1), which may lower the buffering capacity of the yogurt base. Chlorogenic acids accounted for about 95% of the total phenolic compounds in CW-SCG powder (Osorio-Arias et al., 2020), and they may serve as chelating agents, inducing changes in the acid-base equilibrium. Upon dissolving in the yogurt base, chlorogenic acids may ionize and their degree of

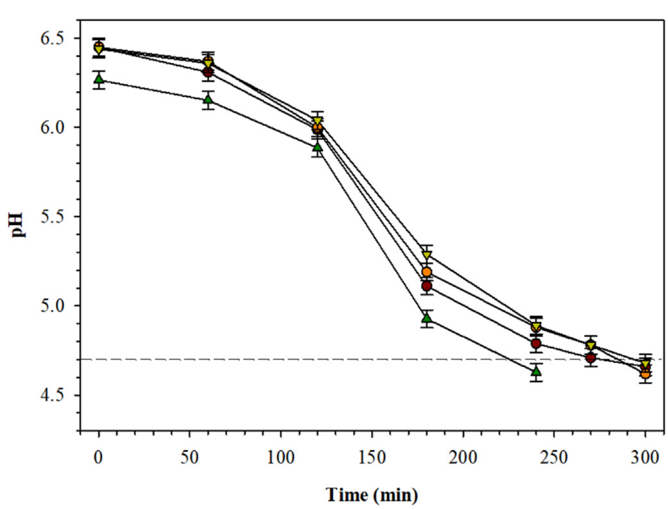

Fig. 1. Acidification profile for the Greek-style yogurts fortified with cheese whey-spend coffee ground powder. The discontinuous line indicates a pH value of 4.7. 0-, 25-, 75-, and 100-CW-SCG represent the percentage of cheese whey-spend coffee ground powder. Error bars represent standard deviation of three experiments. ● 0-CW-SCG; ●25-CW-SCG; ▼75-CW-SCG; and ▲100-CW-SCG.

dissociation depend on the pH. Šeruga and Tomac (2014) studied the oxidation/reduction behavior of six isomers of chlorogenic acids, and they found that chlorogenic acids are found as neutral and anion molecules in solutions of pH range 3–7, having a maximum dissociation at pH 7. Thus, the addition of CW-SCG shifts the buffering capacity towards high pH, which may explain the overall acceleration of the fermentation.

3.2. Effect of CW-SCG on the composition

The effect of adding CW-SCG on the total protein content, available lysine, total polyphenols content (TPC), and antioxidant capacity is shown in Table 1. The differences in the protein content and available lysine (Table 1) are not surprising since the protein content of skim-milk powder is higher than that of CW-SCG, 34, and 10%, respectively. On the other hand, the total content of polyphenols was only detected (23.11 ± 2.01 mg of GAEs per 100 g^{-1}) in those yogurts formulated with 100-CW-SCG. The total phenolic content for 0-, 25-, and 75-CW-SCG was below the detection limit. Phenolic compounds and milk proteins can form complexes (Dönmez, Mogol, & Gökmen, 2017), which in turn inhibits the oxidation/reduction reaction of the Folin-Ciocalteu method (Agbor, Vinson, & Donnelly, 2014).

The antioxidant capacity increased proportionally to the added CW-SCG powder, yielding values of 68.41 ± 2.78, 80.71 ± 2.54, 100.51 ± 3.44, and 120.21 ± 3.18 μmol TE per 100 g for 0-, 25-, 75-, and 100-CW-SCG, respectively. In the case of the control (0-CW-SCG), similar values of antioxidant activity (76.9–131.8 μmol TE 100 g^{-1}) has been reported by Şanlıdere Aloğlu and Öner (2011), who attributed the antioxidant capacity to the release of free amino acids and the formation of peptides. During the manufacture of yogurt, the base yogurt is heated at such conditions that extensively denaturized the fraction of whey proteins. As a result, the proteins unfolded and exposed their thiol groups, which may serve as hydrogen donors (Virtanen, Pihlanto, Akkanen, & Korhonen, 2007). Additionally, the formation of free amino acids due to proteolysis during the fermentation cannot be ignored as a source of antioxidants, as previously reported elsewhere (Mann, Athira, Sharma, & Bajaj, 2017). Overall, the antioxidant activity of free amino acids and peptides greatly depends on the composition and chemical structure, that determine the ability to donate protons (Chen, Muramoto, Yamauchi, Fujimoto, & Nokihara, 1998; Mann et al., 2017).

3.3. Whey production and water holding capacity

Table 2 shows the whey production of the formulated yogurts with different percentages of CW-SCG powder. The highest value of whey production was obtained in those samples fortified with 100-CW-SCG (6.33 ± 0.35%), followed by 75- and 25-CW-SCG (5.17 ± 0.99 and 3.01 ± 0.81%, respectively). Whey production, also known as wheying-off measures the spontaneous expulsion of whey from the network, and it is related to unstable network (Lee & Lucey, 2004). On the other hand, the water holding capacity decreased from about 73 to 55–63% with the addition of CW-SCG powder. Table 2 also shows the water holding capacity of the yogurts formulated with CW-SCG powder. The highest values for water holding capacity was 73.82 ± 0.85 and 63.07 ± 0.84%, followed by 59.49 ± 1.34 and 55.35 ± 1.27% for 0-, 100-, 25-, and 75-CW-SCG, respectively. The values of water holding capacity obtained for 0-CW-SCG can be explain by the higher protein content (Table 1) of the yogurt base. The fortification of the yogurt base with whey protein induces cross-linkage of the gel network, immobilizing the water and preventing it from expulsion (Bong & Moraru, 2014; Mahomud et al., 2017; Sodini, Montella, & Tong, 2005). The fortification with CW-SCG may result in an open gel structure due to the lower protein content, resulting in more whey production and syneresis (González-Martí;nez et al., 2002).

Table 1
Compositional properties of the different yogurts added with CW-SCG powder.

Parameter	Amount of CW-SCG (%)			
	0-CW-SCG	25-CW-SCG	75-CW-SCG	100-CW-SCG
Moisture content (%)	84.71 ± 0.55[a]	84.55 ± 0.45[a]	84.78 ± 0.75[a]	84.77 ± 0.35[a]
Total protein content (%)	5.89 ± 0.39[a]	4.29 ± 0.45[b]	3.55 ± 0.71[c]	2.98 ± 0.44[d]
Available lysine (mg per 100 g)	78.55 ± 1.56[a]	28.89 ± 2.45[b]	23.61 ± 4.42[c]	20.03 ± 2.72[cd]
pH	4.66 ± 0.09[a]	4.62 ± 0.05[a]	4.60 ± 0.04 [a]	4.66 ± 0.04[a]
Total polyphenol content (mg per GAEs 100 g^{-1})	Not detected	Not detected	Not detected	23.11 ± 2.01
Antioxidant capacity (μmol TEs per 100 g)	68.41 ± 2.78[a]	80.71 ± 2.54[ab]	100.51 ± 3.44[c]	120.12 ± 3.18[d]

CW-SCG represents cheese whey-spent coffee ground powder.
Mean ± standard deviation (n = 3) within each row with different letters (a–d) are significantly different ($p < 0.05$).

3.4. Firmness, hardness, and adhesiveness

The texture of the formulated yogurts was evaluated through firmness, hardness, and adhesiveness (Table 2). The addition of CW-SCG significantly changes the texture of the Greek-style yogurts. The hardness of the yogurts decreased from 76.57 ± 1.18 to 30.27 ± 1.73 N s with the addition of CW-SCG powder. The yogurt formulated with 0-CW-SCG showed the highest hardness due to the higher content of whey protein. Wang, Bao, Hendricks, and Guo (2012) reported that the addition of whey protein resulted in a gel network less susceptible to rupture. In the case of firmness, a decreasing tendency was observed in the yogurts formulated with CW-SCG, from 4.69 ± 0.06 to 1.72 ± 0.11 N. The firmness in yogurt is strongly influenced by the formation of protein complexes between denatured whey proteins attached to the surface of casein micelle (Jørgensen et al., 2015; Mahomud et al., 2017). It appears that the addition of CW-SCG disrupted the protein network (whey proteins and caseins), impacting negatively the firmness of the yogurt. The adhesiveness of the formulated yogurts increased with the concentration of CW-SCG, yielding values of −12.32 ± 0.57, −8.52 ± 0.42, −5.04 ± 0.26, and −3.43 ± 1.18 N s for 0-, 100-, 25-, and 75-CW-SCG, respectively. The texture parameters (Table 2) suggest that the presence of CW-SCG may disrupt the formation of protein complexes, impacting the hardness, firmness, and adhesiveness. Such an idea is further explored by scanning electronic microscopy.

3.5. Flow behavior and viscosity

The flow curve (shear stress vs. shear rate) of the formulated yogurts containing different concentration of CW-SCG are shown in Fig. 2. The curves displayed a non-linear shear stress-shear rate relation, indicating a shear-thinning behavior for all samples. Similarly, Singh and Muthukumarappan (2008) reported shear thinning behavior for yogurt fortified with calcium. The fortification with CW-SCG showed less shear-thinning behavior as compared to the control yogurt (0-CW-SCG). This observation somewhat supports our previous statement that the addition of CW-SCG seems to disrupt the formation of the protein network made of denatured whey proteins attached to the surface of micelle. The flow behavior of the formulated yogurts was characterized using the Herschel-Buckley model (Equation (1)). Table 3 shows the consistency index and power-law index for the formulated yogurts with CW-SCG. The values of the power-law index from the yogurts with

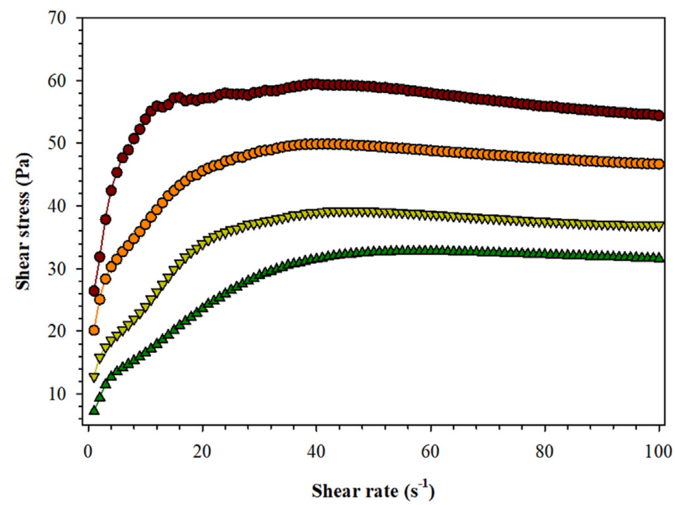

Fig. 2. Flow curve of the Greek-style yogurts fortified with cheese whey-spend coffee ground powder. 0-, 25-, 75-, and 100-CW-SCG represent the percentage of cheese whey-spend coffee ground powder. ●0-CW-SCG; ●25-CW-SCG; ▼75-CW-SCG; and ▲100-CW-SCG.

Table 3
Flow characteristics of the different yogurts added with CW-SCG powder.

Sample	n	*K* (Pa s^n)	R^2
0-CW-SCG	0.30 ± 0.03	12.43 ± 0.99	0.906
25-CW-SCG	0.59 ± 0.04	4.36 ± 0.67	0.988
75-CW-SCG	0.81 ± 0.01	1.78 ± 0.21	0.992
100-CW-SCG	0.71 ± 0.03	1.91 ± 0.27	0.986

CW-SCG represents cheese whey-spent coffee ground powder.
Mean ± standard deviation (n = 3); R^2 is the coefficient of determination.

Table 2
Effect of the addition of cheese whey-spent coffee ground (CW-SCG) powder on selected quality parameters.

Parameter	Amount of CW-SCG (%)			
	0-CW-SCG	25-CW-SCG	75-CW-SCG	100-CW-SCG
Whey production (%)	0.00 ± 0.00[a]	3.01 ± 0.81[b]	5.17 ± 0.99[c]	6.33 ± 0.35[c]
Water holding capacity (%)	73.82 ± 0.85[a]	59.49 ± 1.34[b]	55.35 ± 1.27[c]	63.07 ± 0.84[d]
Hardness (N s)	76.57 ± 1.18[a]	53.42 ± 0.75[b]	37.25 ± 1.39[c]	30.27 ± 1.73[d]
Firmness (N)	4.69 ± 0.06[a]	3.21 ± 0.04[b]	2.22 ± 0.06[c]	1.72 ± 0.11[d]
Adhesiveness (N s)	−12.32 ± 0.57[a]	−8.52 ± 0.42[b]	−5.04 ± 0.26[c]	−3.43 ± 1.18[d]

CW-SCG represents cheese whey-spent coffee ground powder.
Mean ± standard deviation (n = 3) within each row with different letters (a–d) are significantly different ($p < 0.05$).

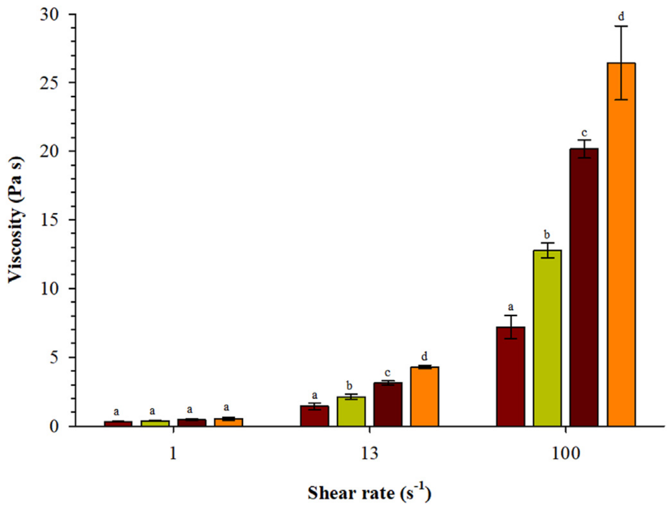

Fig. 3. Changes in viscosity of the Greek-style yogurts fortified with cheese whey-spend coffee ground powder. 0-, 25-, 75-, and 100-CW-SCG represent the percentage of cheese whey-spend coffee ground powder. 0-CW-SCG; 25-CW-SCG; 75-CW-SCG; and 100-CW-SCG. Mean ± standard deviation (n = 3) within treatments with different letters are (a–d) significantly different ($p < 0.05$) according to Tukey test.

added CW-SCG were significantly higher (0.59–0.81) than that obtained from the control (0.30 ± 0.03). Moreover, the values corresponding to the power-law index ($n < 1$) indicate a pseudoplastic shear thinning behavior for all samples. On the other hand, the consistency index decreased with the addition of CW-SCG, yielding values of 12.43 ± 0.99, 4.36 ± 0.67, and 1.78 ± 0.21 for 0-, 25-, and 75-CW-SCG, respectively. The exception to this generalization was observed in those samples added with 100-CW-SCG, where the consistency index slightly decreased, 1.91 ± 0.27. The flow behavior reported in Table 3 is comparable to that reported in the literature (Aportela-Palacios, Sosa-Morales, & Vélez-Ruiz, 2005; Singh & Muthukumarappan, 2008; Vercet, Oria, Marquina, Crelier, & Lopez-Buesa, 2002).

Fig. 3 shows the effect of adding CW-SCG on the viscosity of the Greek-style yogurt. The addition of CW-SCG significantly changes the viscosity of the yogurts. At the low shear spectrum (1 s^{-1}), the addition of 100-CW-SCG resulted in a reduction of up to 3.6-fold in the viscosity. Increasing the shear rates to 13 and 100 s^{-1} produced a less pronounced reduction in the viscosity, 2.99- and 1.72-fold, respectively.

3.6. Strain and frequency sweep

Fig. 4 shows the strain sweep (storage module, G′, vs. strain amplitude) for the Greek-style yogurts fortified with CW-SCG. For all samples, two distinct regions can be observed, linear and non-linear viscoelastic region. In the case of the linear region, the G′ remained constant, while in the non-linear region, the G′ decreased with increasing strain. The presence of the linear viscoelastic region indicates that the yogurts behaved like a viscoelastic solid. The length of the linear region has been related to product stability (Martínez-Monteagudo et al., 2017). The addition of CW-SCG produced yogurts having lower values of G′ when compared with the control yogurt. A possible explanation is that the incorporation of carbohydrates weakened the protein network. One of the causes of disruption of the protein network is due to the fact that polyphenols (chlorogenic acids), and fiber can bind to milk proteins through hydrophobic interactions that subsequently decrease hydrophobic regions within the protein surface (Yuksel, Avci, & Erdem, 2010). The strongest network according to the magnitude of G′ was obtained for the control sample. The appearance of the non-linear region indicates the yielding point, where

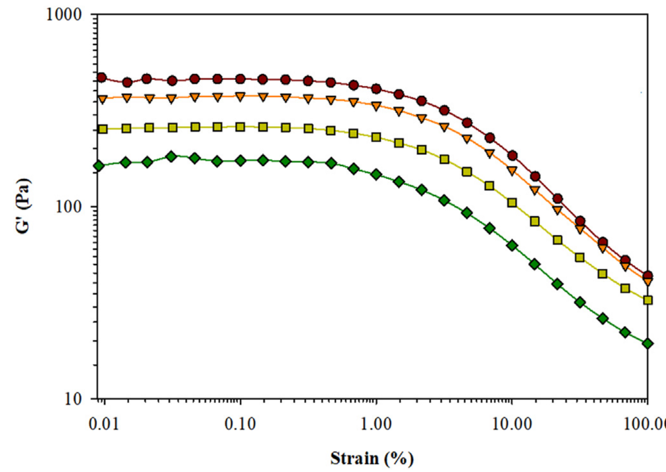

Fig. 4. Strain sweep dependency of storage module (G′) for the Greek-style yogurts fortified with cheese whey-spend coffee ground powder. 0-, 25-, 75-, and 100-CW-SCG represent the percentage of cheese whey-spend coffee ground powder. Frequency = 5 rad s^{-1}. ●0-CW-SCG; ▼ 25-CW-SCG; 75-CW-SCG; and ◆100-CW-SCG.

the breakdown of the network is taking place, changing from viscoelastic solid to viscoelastic liquid. Interestingly, the length of the linear segment was not impacted by the addition of the CW-SCG, which reflects the complexity of the formed network.

Fig. 5 shows the frequency sweep analysis, where the storage module (G′) and loss module (G″) are plotted against the frequency. For all samples, the G′ and G″ slightly increased with frequency, and the G′ values were always higher than G″ (G′ > G″), meaning that the elastic behavior dominates. Additionally, when G′ becomes greater than G″ indicates a clear tendency to form macromolecular networks. The analysis of log G′ vs. log ω can be used to infer possible structuring mechanisms for fluids (Ferry, 1980). A relation was found between G′ and frequency according to Equation (1), and their parameters are given in Table 4. The addition of CW-SCG resulted in values of the parameter *b* ranging from 0.90 to 1.10, which is the characteristics of an elastic gel (Hesarinejad, Koocheki, & Razavi, 2014). The lowest value of the parameter *b* was obtained for the control sample, followed

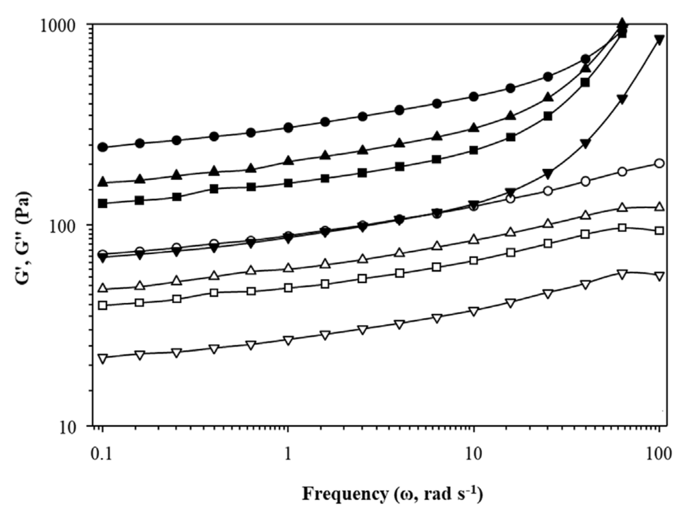

Fig. 5. Frequency sweep analysis for the Greek-style yogurts fortified with cheese whey-spend coffee ground powder. 0-, 25-, 75-, and 100-CW-SCG represent the percentage of cheese whey-spend coffee ground powder. Strain = 5%. Closed symbols represent storage module (G′), and open symbols represent loss module (G″). ●0-CW-SCG; ▲ 25-CW-SCG; ■75-CW-SCG; and ▼100-CW-SCG.

Table 4
Parameters of the relationship between storage module (G') and frequency (ω) for the different yogurts added with CW-SCG powder.

Sample	Parameters Equation (1)		R^2
	a	b	
0-CW-SCG	230.29	0.35	0.889
25-CW-SCG	27.29	0.91	0.921
75-CW-SCG	10.55	1.10	0.967
100-CW-SCG	10.82	0.92	0.937

CW-SCG represents cheese whey-spent coffee ground powder; R^2 is the coefficient of determination.

by the sample with added 25-CW-SG, 0.35 and 0.91, respectively. The parameter *b* is used to determine the strength and nature of liquid (Ferry, 1980). On the other hand, the simples with added 75- and 100-CW-SCG yielded values of the parameter *b* close to one (1.10 and 0.92, respectively), meaning that the yogurts have characteristics of a viscous gel. Contrary, the control sample showed characteristics of elastic gel, yielding *b* values of 0.35.

3.7. Microstructure

In an attempt to explain the influence of the addition of CW-SCG, the microstructure of the yogurts was evaluated through scanning electronic microscopy (Fig. 6). The control sample (0-CW-SCG) displayed a grainy network made of casein micelles aggregates

Linked in clusters, forming a three-dimensional network of irregular void spaces (Penna, Subbarao, & Barbosa-Cánovas, 2007). Such a network provides hardness and firmness to the yogurt (Jaya, 2009). On the other hand, the microstructure of the yogurts with added CW-SCG revealed the presence of SCG particles, judging by the appearance of flat-leaves particles (Ballesteros, Teixeira, & Mussatto, 2014). As the content of CW-SCG increases, the void spaces within the protein network were occupied by the SCG particles. This observation is rather evident when the yogurt was added with 100-CW-SCG. The microstructure of those yogurts added with 100-CW-SCG suggested that the SCG particles disrupt the protein network. Microstructurally, SCG is made of coffee particles, chlorogenic acids, and fiber. The addition of CW-SCG lowers the protein content, which directly impacts the formation of the protein network. Concomitantly, the relatively high content of chlorogenic acids in the CW-SCG (Table 1) may induce the formation of protein-polyphenol complexes (Dönmez et al., 2017). Polyphenols can bind to milk proteins through hydrophobic interactions that subsequently decreases the hydrophobic regions within the surface of the protein (Yuksel et al., 2010). It is reasonable to infer that the interaction between polyphenols and milk proteins yields a weaker network that impacts the hardness, firmness, and rheological behavior of the formulated yogurts.

4. Conclusions

The fortification of CW-SCG during the manufacture of Greek-style yogurt was evaluated in terms of acidification, chemical composition, texture, rheological behavior, and microstructure. The addition of CW-SCG up to 75% did not significantly change the acidification curve when compared with the control sample. The fortification up to 25% of CW-SCG resulted in yogurt of comparable properties than the control. In general, fortified yogurts with CW-SCG yielded a gel of weaker firmness and less shear-thinning behavior as compared to control. The addition of CW-SCG seems to disrupt the protein network and enhanced the interactions between polyphenols and proteins. Further scale-up will require to study the sensorial description of the product as well as the consumer acceptance.

CRediT authorship contribution statement

Juan Osorio-Arias: Methodology, Formal analysis, Writing - review & editing. **Angela Pérez-Martínez:** Formal analysis. **Oscar Vega-Castro:** Conceptualization, Writing - review & editing, Supervision. **Sergio I. Martínez-Monteagudo:** Conceptualization, Writing - review & editing, Supervision.

Declaration of competing interest

The authors whose names are listed immediately below certify that

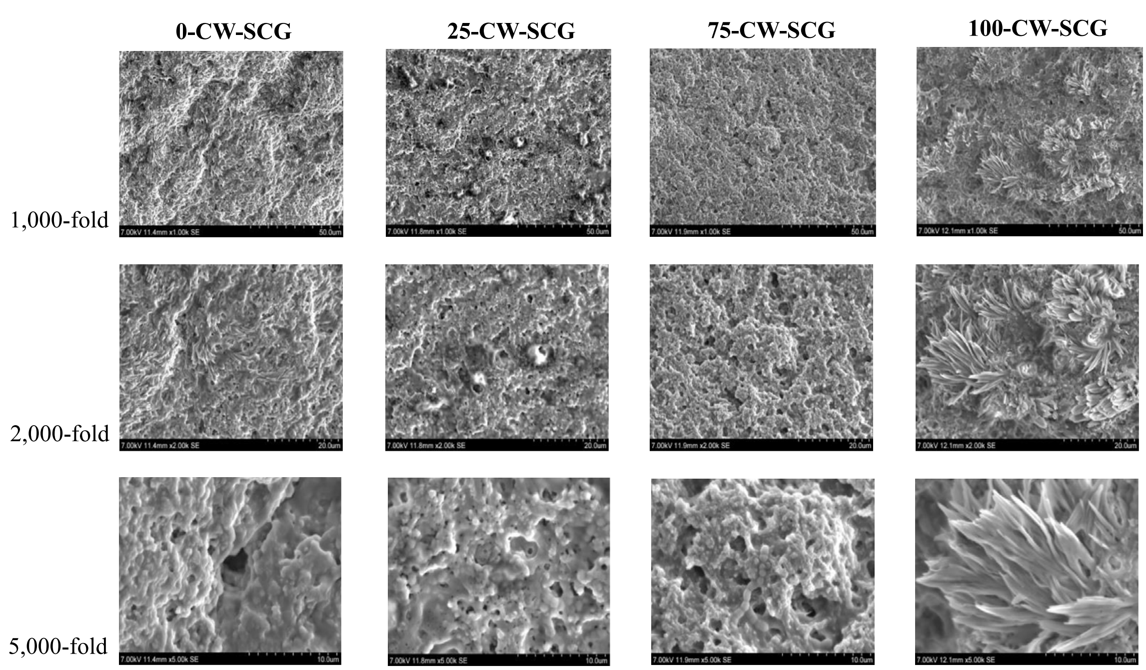

Fig. 6. Microstructure of for the Greek-style yogurts fortified with cheese whey-spend coffee ground powder. 0-, 25-, 75-, and 100-CW-SCG represent the percentage of cheese whey-spend coffee ground powder.

they have NO affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript.

- Juan C. Osorio-Arias
- Angela Perez-Martinez
- Oscar Vega-Castro
- Sergio I. Martínez-Monteagudo

Acknowledgments

Osorio-Arias thanks the financial support of fondo nacional de financiamiento para la ciencia, la tecnologia y la inovacion “Francisco Jose de Caldas” COLCIENCIAS (nr. 727-2015). Martinez-Monteagudo expresses his gratitude to USDA National Institute for Food and Agriculture (HATCH project SD00H607-16). Vega-Castro thanks Jhon Ortiz Duque for his technical contributions.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2020.109523.

References

Agbor, G. A., Vinson, J. A., & Donnelly, P. E. (2014). Folin-Ciocalteau reagent for polyphenolic assay. *International Journal of Food Science, Nutrition and Dietetics, 3*(8), 147–156.

Aportela-Palacios, A., Sosa-Morales, M. E., & Vélez-Ruiz, J. F. (2005). Rheological and physicochemical behavior of fortified yogurt, with fiber and calcium. *Journal of Texture Studies, 36*(3), 333–349.

Ballesteros, L. F., Teixeira, J. A., & Mussatto, S. I. (2014). Chemical, functional, and structural properties of spent coffee grounds and coffee silverskin. *Food and Bioprocess Technology, 7*(12), 3493–3503.

Bong, D. D., & Moraru, C. I. (2014). Use of micellar casein concentrate for Greek-style yogurt manufacturing: Effects on processing and product properties. *Journal of Dairy Science, 97*(3), 1259–1269.

Brückner-Gühmann, M., Benthin, A., & Drusch, S. (2019). Enrichment of yoghurt with oat protein fractions: Structure formation, textural properties and sensory evaluation. *Food Hydrocolloids, 86*, 146–153.

Chen, H.-M., Muramoto, K., Yamauchi, F., Fujimoto, K., & Nokihara, K. (1998). Antioxidative properties of histidine-containing peptides designed from peptide fragments found in the digests of a soybean protein. *Journal of Agricultural and Food Chemistry, 46*(1), 49–53.

Chen, X., Singh, M., Bhargava, K., & Ramanathan, R. (2018). Yogurt fortification with chickpea (*cicer arietinum*) flour: Physicochemical and sensory effects. *Journal of the American Oil Chemists Society, 95*, 1041–1048.

Contreras-Calderón, J., Mejía-Díaz, D., Martínez-Castaño, M., Bedoya-Ramírez, D., López-Rojas, N., Gómez-Narváez, F., et al. (2016). Evaluation of antioxidant capacity in coffees marketed in Colombia: Relationship with the extent of non-enzymatic browning. *Food Chemistry, 209*, 162–170.

Dönmez, Ö., Mogol, B. A., & Gökmen, V. (2017). Syneresis and rheological behaviors of set yogurt containing green tea and green coffee powders. *Journal of Dairy Science, 100*(2), 901–907.

do Espírito Santo, A. P., Perego, P., Converti, A., & Oliveira, M. N. (2012). Influence of milk type and addition of passion fruit peel powder on fermentation kinetics, texture profile and bacterial viability in probiotic yoghurts. *LWT – Food Science and Technology, 47*(2), 393–399.

Ferry, J. D. (1980). *Viscoelastic properties of polymers* (3rd ed.). New York: John Wiley & Sons.

González-Martínez, C., Becerra, M., Cháfer, M., Albors, A., Carot, J. M., & Chiralt, A. (2002). Influence of substituting milk powder for whey powder on yoghurt quality. *Trends in Food Science & Technology, 13*(9), 334–340.

Hashim, I. B., Khalil, A. H., & Afifi, H. S. (2009). Quality characteristics and consumer acceptance of yogurt fortified with date fiber. *Journal of Dairy Science, 92*(11), 5403–5407.

Hesarinejad, M. A., Koocheki, A., & Razavi, S. M. A. (2014). Dynamic rheological properties of Lepidium perfoliatum seed gum: Effect of concentration, temperature and heating/cooling rate. *Food Hydrocolloids, 35*, 583–589.

Jaya, S. (2009). Microstructure analysis of dried yogurt: Effect of different drying methods. *International Journal of Food Properties, 12*(3), 469–481.

Jørgensen, C. E., Abrahamsen, R. K., Rukke, E.-O., Johansen, A.-G., Schüller, R. B., & Skeie, S. B. (2015). Improving the structure and rheology of high protein, low fat yoghurt with undenatured whey proteins. *International Dairy Journal, 47*, 6–18.

Karam, M. C., Gaiani, C., Hosri, C., Burgain, J., & Scher, J. (2013). Effect of dairy powders fortification on yogurt textural and sensorial properties: A review. *Journal of Dairy Research, 80*(4), 400–409.

Lee, W. J., & Lucey, J. A. (2004). Structure and physical properties of yogurt gels: Effect of inoculation rate and incubation temperature. *Journal of Dairy Science, 87*(10), 3153–3164.

Lesme, H., Rannou, C., Loisel, C., Famelart, M.-H., Bouhallab, S., & Prost, C. (2019). Controlled whey protein aggregates to modulate the texture of fat-free set-type yoghurts. *International Dairy Journal, 92*, 28–36.

Mahomud, M. S., Katsuno, N., & Nishizu, T. (2017). Formation of soluble protein complexes and yoghurt properties influenced by the addition of whey protein concentrate. *Innovative Food Science & Emerging Technologies, 44*, 173–180.

Mann, B., Athira, S., Sharma, R., & Bajaj, R. (2017). Bioactive peptides in yogurt. In N. P. Shah (Ed.). *Yogurt in health and disease prevention*. Cambridge, MA: Academic Press LTD-Elsevier Science LTD.

Martínez-Monteagudo, S. I., Kamat, S., Patel, N., Konuklar, G., Rangavajla, N., & Balasubramaniam, V. M. (2017). Improvements in emulsion stability of dairy beverages treated by high pressure homogenization: A pilot-scale feasibility study. *Journal of Food Engineering, 193*, 42–52.

Osorio-Arias, J., Delgado-Arias, S., Duarte-Correa, Y., Largo-Ávila, E., Montaño, D., Simpson, R., et al. (2020). New powder material obtained from spent coffee ground and whey protein; Thermal and morphological analysis. *Materials Chemistry and Physics, 240*, 122171.

Ozturkoglu-Budak, S., Akal, C., & Yetisemiyen, A. (2016). Effect of dried nut fortification on functional, physicochemical, textural, and microbiological properties of yogurt. *Journal of Dairy Science, 99*(11), 8511–8523.

Penna, A. L. B., Subbarao, G., & Barbosa-Cánovas, G. V. (2007). High hydrostatic pressure processing on microstructure of probiotic low-fat yogurt. *Food Research International, 40*(4), 510–519.

Prasannan, A. (2017). *Yogurt market opportunities and forecasts*.

Şanlıdere Aloğlu, H., & Öner, Z. (2011). Determination of antioxidant activity of bioactive peptide fractions obtained from yogurt. *Journal of Dairy Science, 94*(11), 5305–5314.

Šeruga, M., & Tomac, V. (2014). Electrochemical behaviour of some chlorogenic acids and their characterization in coffee by square-wave voltammetry. *International Journal of Electrochemistry Science, 9*, 6134–6154.

Singh, G., & Muthukumarappan, K. (2008). Influence of calcium fortification on sensory, physical and rheological characteristics of fruit yogurt. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology, 41*(7), 1145–1152.

Sodini, I., Montella, J., & Tong, P. S. (2005). Physical properties of yogurt fortified with various commercial whey protein concentrates. *Journal of the Science of Food and Agriculture, 85*(5), 853–859.

Sodini, I., Remeuf, F., Haddad, S., & Corrieu, G. (2004). The relative effect of milk base, starter, and process on yogurt texture: A review. *Critical Reviews in Food Science and Nutrition, 44*(2), 113–137.

Tamime, A. Y., & Robinson, R. K. (2007). *Tamime and robinson's yoghurt: Science and technology*. Boca Raton, FL: Woodhead Publishing.

Vercet, A., Oria, R., Marquina, P., Crelier, S., & Lopez-Buesa, P. (2002). Rheological properties of yoghurt made with milk submitted to manothermosonication. *Journal of Agricultural and Food Chemistry, 50*(21), 6165–6171.

Virtanen, T., Pihlanto, A., Akkanen, S., & Korhonen, H. (2007). Development of antioxidant activity in milk whey during fermentation with lactic acid bacteria. *Journal of Applied Microbiology, 102*(1), 106–115.

Yuksel, Z., Avci, E., & Erdem, Y. K. (2010). Characterization of binding interactions between green tea flavanoids and milk proteins. *Food Chemistry, 121*(2), 450–456.

Wang, B., Bao, Y., Hendricks, G. M., & Guo, M. (2012). Consistency, microstructure and probiotic survivability of goats' milk yoghurt using polymerized whey protein as a co-thickening agent. *International Dairy Journal, 24*(2), 113–119.