

# Effects of spray drying conditions and the addition of surfactants on the foaming properties of a whey protein concentrate



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## ABSTRACT

Whey is the main waste by-product from dairy industry and at the same time is the major source of globular proteins. These proteins are concentrated mainly through spray drying, but high temperatures affect the foaming properties of globular protein. The addition of surfactants can have a protective role against thermal effects. The aim of this work was to optimize the spray-drying condition and surfactant concentration to obtain a whey protein concentrate (WPC) to be used in hot beverages according to the industry criteria for foaming stability. Three temperatures and three surfactant concentrations were applied, and the optimization was conducted using response surface analysis. Sensory analysis was applied to the WPC obtained under optimal conditions. The results showed that the foaming stability according to industrial criteria was attained when the spray drying was performed at 210 °C with surfactant concentration of 1.50 g/100 g. This resulted in foaming capacity of 3.80 mL, moisture content of 1.82 g/100 g and apparent density of 0.181 g/cm<sup>3</sup>. The sensory analysis suggested that aroma was related to dairy, cooked and whey and taste was related to sweet and dairy notes. In conclusion, temperature and surfactant concentration played an important role in the foaming capacity and stability of WPC.

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## 1. Introduction

Whey protein concentrate (WPC) is the main source of globular proteins, which are used in the food industry for their emulsifying and foaming properties (Bernard, Regnault, Gendreau, Charbonneau, & Relkin, 2011). Proteins have been recognized as foaming agents in the food industry, with applications in the baking, hot beverages, confectionery and other industries, especially when the gas phase is dispersed by beating or whipping. The most common commercial foaming ingredients are egg white proteins (EWP) and milk proteins, with WPC being the most effective (Nicorescu et al., 2011). Due to their surface properties, which involve a decrease in the interfacial tension, globular proteins alone or mixed with caseins have been shown to adsorb at the droplet surface and stabilize oil-in-water emulsions (Dickinson, 2001; Sourdet, Relkin, Fosseux & Aubry, 2002). Damodaran (1997) reported that the capacity of whey to form and stabilize foams is

due mainly to the relationship between monomeric and polymeric species. The monomeric form would contribute to the foaming capacity, and the polymeric forms would contribute to foam stabilization.

Several studies have been conducted to understand the effect of heat treatments on whey proteins either in solution or after incorporation into an emulsion. According to the literature, heating whey proteins in solution or in an emulsion at temperatures higher than 70 °C induces denaturation, resulting in the unfolding of the whey protein molecules and the exposure of their reactive sites (Galani & Apenten, 1999; Millqvist-Fureby, Eloffsson, & Bergenstahl, 2001; Relkin, 1996), which affect on foam and emulsion stabilization (Livney, Corredig, & Dalgleish, 2003; Millqvist-Fureby et al., 2001; Relkin, Bernard, Meylheuc, Vasseur, & Courtois, 2007; Sourdet, Relkin, Fosseux, & Aubry, 2002).

According to Master (1979) and Nath and Satpatthy (1998), spray drying transforms a liquid or pasty food into a powder through the disintegration of the liquid in atomized small particles of a high-pressure spray after contact with hot air.

Compared with other drying methods, this process is noted for its applicability in heat-sensitive products such as food due to the rapid evaporation of water, which reduces the temperature of the gesticulate and keeps the drying time very short, which reduces the

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thermal damage and ensures that good quality food is produced. All of these outcomes are due to the establishment of a large exchange surface between the liquid spray and the surrounding gas (Zhartha & Palacio, 2009). However, this process is often considered as a process that can affect negatively the protein properties due to high temperatures that commonly are applied. For this reason, the effect of surfactant a protection agent during thermal process has been extensively studied. Also in other research fields, like pharmaceutical area, surfactants are suitable candidates as protectors because they are mainly present on the particle surface, avoiding thermal damage of enzymes and other proteins (Alder, Unger, & Lee, 2000; Yoshii et al., 2008). Also, surfactants have been identified as agents that can control mass transfer during spray drying depending on their characteristics. For example, insoluble surfactant monolayer can form a solid film on the particle surface which acts like diffusional barrier. In the case of soluble surfactants forming a Gibbs monolayer, such barrier effect is usually not presents (Frey & King, 1986).

Fang, Rogers, Salomulya, and Dong Chen (2012) reported that lactoglobulin provided more stability than casein (a milk protein) when both were dried at different temperatures in a spray dryer. Oldfield, Taylor, and Singh (2005) reported that spray-drying temperatures ranging from 160 to 200 °C for the inlet and 89–101 °C for the outlet did not significantly affect the denaturation of  $\beta$ -lactoglobulin. Pisecký (2005) observed that the production of WPC powder is an attractive alternative for processing whey because the product has a high value on the market. It is used mainly as a component of baby food and for protein fortification in various food formulations. The standard product for the market contains 35, 60–80 or 90 g protein/100 g.

The effect of heat treatment on whey protein has been widely studied and it is known that temperatures of 70 °C induce protein modifications related with unfolding and exposure of active sites. These alterations increase the probability of droplet flocculation and coalescence affecting negatively the foaming properties (Bernard et al., 2011). This temperature effect can be overcome through the use of surfactants, which can interact with active sites avoiding droplets flocculation and coalescence.

The aim of this work was to study the optimal conditions of both, spray drying and surfactant concentration, to obtain a whey protein concentrate powder to be applied in hot beverages according to optimize response based on industry criteria for foaming stability.

## 2. Materials and methods

### 2.1. Materials

The whey concentrate process was performed under licensee of COLANTA S.A (Medellin, Colombia). The whey was concentrated until reaching 40°Brix, pH 6.0 and an acidity of 0.90. The surfactant Tween 60 was obtained from PROES (Productos Especiales), Medellin, Colombia.

### 2.2. WPC powder preparation

After the whey protein was concentrated, it was mixed at 200 rpm with the surfactant Tween 60 at three concentrations: 1.50, 2.25 and 3.00 g/100 g. Then, to get the powder, WPC with different surfactant concentrations was dehydrated in a laboratory-scale spray dryer (BUCHI Mini Spray Dryer B-290, Flawil, Switzerland). Samples were pulverized with a co-current airflow produced by a blower. The airflow rate was 830.8–1051.75 L h<sup>-1</sup>, and the humidity level of the air was 18 g water (kg dry air)<sup>-1</sup>. The samples were dried for 3 h at three temperatures: 170, 190 and

210 °C. Finally, the WPC-surfactant powder was characterized as follows.

### 2.3. WPC powder characterization

The WPC powder characterization involved the determination of the moisture content, apparent density and particle size. The moisture content was determined according to 930.15 AOAC (1990). The apparent density of the WPC-surfactant powder was measured using the pycnometer method proposed by Caparino et al. (2012). Briefly, 5.0 g of the WPC-surfactant powder was placed into a 25 mL graduated glass cylinder, which was tapped manually by lifting and dropping the cylinder under its own weight until a negligible difference in the volume measurements was observed. The mass ( $m$ ) and the apparent (tapped) volume ( $V$ ) of the powder were used to calculate the powder density as  $m/V$  (kg/m<sup>3</sup>).

The particle size distribution of the WPC-surfactant powder 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  $\mu$ m.

### 2.4. Foaming properties

#### 2.4.1. Foaming capacity and stability

To produce the foam, 4.00 g of the WPC-surfactant was placed into a 250 mL beaker with 85 mL of distilled water at 80  $\pm$  1 °C. The resulting solution was whipped with a mechanical blender (Hamilton Beach) for 2 min at medium rate.

The foaming capacity was measured according to the method proposed by Constant (1991), with some modifications. 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 the visual method based on the modified methodology proposed by Wilson and Mundy (1984) at 80  $\pm$  1 °C. 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.

### 2.5. Sensory analysis

The foam forming solution that was obtained after the optimization underwent sensory measurements by nine trained panelists. The sensory analysis was performed based on the NTC3929 (NTC, 2009), approved by ISO6564:2005, where 0 is absence, 1 is very weak, 2 is weak, 3 is moderate, 4 is strong and 5 is intense. The panelists measured the attributes related to flavor and aroma. The panelists received a sample of the foam solution under the same conditions described in the foam formation section. The sensory study was conducted in the Food Sensory Analysis Laboratory at the Universidad de Antioquia, Colombia.

### 2.6. Statistical analysis

To estimate if temperature during drying process or surfactant concentration or a combination of both affect on the moisture content, apparent density, foaming capacity and foam stability whey protein powder were studied by a multifactorial statistical analysis (3<sup>2</sup> with two replicates) performed at 95% of significance (Table 1). In specific, two factors were considered: temperature ( $X_1 = T$ ) during spray drying and surfactant concentration ( $X_2 = C$ ).

**Table 1**

Experimental conditions applied to study the temperature and concentration using a complete design with two replicates.

Trial N°	Surfactant concentration (g/100 g)	Temperature (°C)
1	1.50 (−1)	170 (−1)
2	3.00 (+1)	210 (+1)
3	2.25 (0)	170 (−1)
4	3.00 (+1)	190 (0)
5	1.50 (−1)	190 (0)
6	2.25 (0)	190 (0)
7	2.25 (0)	210 (+1)
8	3.00 (+1)	170 (−1)
9	1.50 (−1)	210 (+1)

Three levels (low, medium, high) were applied for each factor. The surfactant concentrations were 1.50, 2.25 and 3.00 g/100 g, and the drying temperatures were 170, 190 and 210 °C. The response variables for each treatment were the moisture content, apparent density, foaming capacity and foam stability which were investigated by using response surface methodology (RSM), fitting the experimental values to following second order equation:

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

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

### 3. Results and discussion

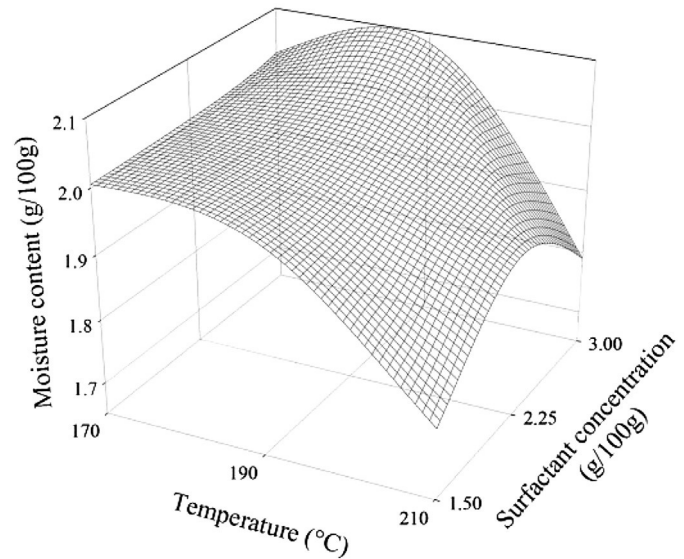
#### 3.1. WPC powder characterization

##### 3.1.1. Moisture content

The average moisture content of the powder obtained after spray drying was 1.95 g/100 g with a range of 1.69–2.14 g/100 g. The mean value is within the range of products dried by the spray method. For example, [Knecht and Van Den Brink \(1998\)](#) obtained a moisture content of 2.32 g/100 g for spray-dried milk, and [Keogh, Murray, and O’Kennedy \(2003\)](#) reached a moisture content of 2.79 g of water/100 g of saccharose.

Analyzing the experimental design, the moisture content behavior with respect to variations in the surfactant content and temperature during the spray-drying process is shown in [Fig. 1](#), which shows that there is a relationship between the moisture content and the surfactant concentration. It is possible to observe that the moisture content was not significantly affected by the surfactant content. However, a surfactant concentration of 2.50 g/100 g increased mildly the moisture content. According to [Frey and King \(1986\)](#), surfactants have the ability to affect mass transfer by reducing internal circulation, oscillation, turbulence, and interfacial movement in drop. This effect can be divided into: *hydrodynamic effect* and *interfacial resistance effects*. The first is related with changes in drop size and velocity, and effects on internal circulation, oscillation and turbulences. The second type, refer to surface blockage and solute–surfactant interaction, where surfactants act like barrier at air–water interface.

With respect to the temperature, the moisture content was decreased when the temperature of the process was higher (210 °C) due to an increase in the release of water molecules from when droplet. However, the moisture content did not present high



**Fig. 1.** Effect of temperature and surfactant concentration on the moisture content of the WPC powder.

difference between 170 and 210 °C, which can be attributed to surfactant content.

The model that describes the behavior of the moisture content ( $M$ ) with respect to the surfactant concentration ( $C$ ) and temperature during spray drying ( $T$ ) is presented in Equation (2):

$$M = -9.5 + 0.12 \times T + 0.94 \times C - 0.003 \times T^2 - 0.0011 \times TC - 0.16 \times C^2 \quad (2)$$

This model presented an  $R$ -square of 84.19% and adjusted  $R$ -square of 78.54%. However, a large  $R$ -square value does not always imply that the regression model is good ([Eren & Kaymak-Ertekin, 2007](#)). When the variables are increased, the  $R$ -square value will be increased, which does not necessarily indicate statistical significance. In this case, the adjusted  $R$ -square is more suitable to evaluate the model fit, and according to [Eren and Kaymak-Ertekin \(2007\)](#), it must be greater than 90%. Our results showed that the moisture content presented a lower  $R$ -square; therefore, this model could not be considered.

Moreover, temperature, temperature-squared and concentration-squared were significant terms ( $p < 0.05$ ) in the model. Thus, the temperature could be considered to be significantly associated with the moisture content. A minimal value of the moisture content is required to avoid microbiological contamination; therefore, the optimal condition to minimize the moisture content was reached at 210 °C and with surfactant concentration of 3.00 g/100 g.

##### 3.1.2. Apparent density

The apparent density of powder ranged between 0.155 and 0.181 g/cm<sup>3</sup> with average value of 0.167 g/cm<sup>3</sup>. This value is similar to those reported by [Yan and Barbosa \(2000\)](#) for low-fat powdered milk. [Fig. 2](#) shows the effects of the surfactant concentration and temperature on the apparent density. It is observed that the surfactant concentration has a relevant effect when low drying temperatures were applied, increasing notoriously the apparent density when high content of surfactant was incorporated. However, at high temperatures the surfactant content was not relevant on apparent density, probably because at high temperatures the molecular interaction was favored which increase the mass but allows getting particles with low volume.

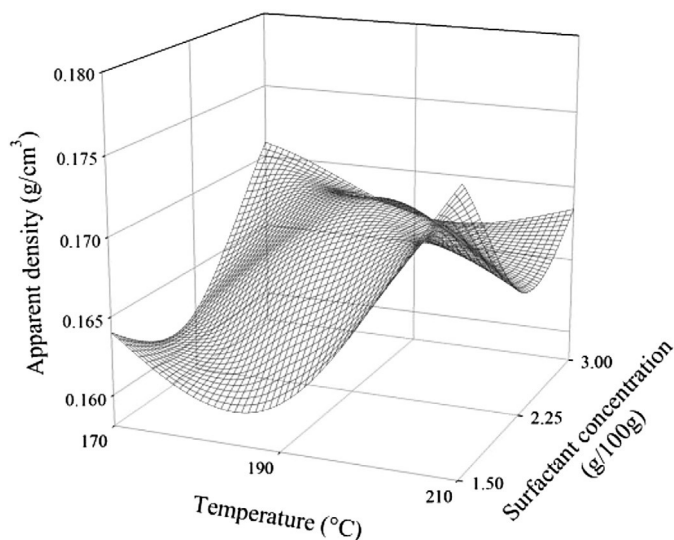


Fig. 2. Effect of temperature and surfactant concentration on the apparent density of the WPC powder.

To avoid high packaging costs due to a higher volume, a higher density is required. The optimal conditions to obtain the appropriate density ( $0.181 \text{ g/cm}^3$ ) were a higher temperature ( $210 \text{ }^\circ\text{C}$ ) and lower surfactant concentration ( $1.50 \text{ g/100 g}$ ).

The equation that represents the apparent density ( $D$ ) of the fitted model is presented in Equation (3):

$$D = 0.49 - 0.0038 \times T + 0.012 \times C + 0.000012 \times T^2 - 0.00027 \times TC + 0.0088 \times C^2 \quad (3)$$

The  $R$ -square was 67.45%, and the adjusted  $R$ -square was 55.83%, both of which are lower than the acceptable level of 90%. Therefore, the model could not be considered to adequately fit the effects of the temperature and surfactant concentration on the apparent density. Moreover, the effects of temperature, temperature-squared, the combination of temperature and concentration and concentration-squared were significant with  $p$  values less than 5%.

### 3.1.3. Particle size analysis

Fig. 3 presents the mean particle size distribution obtained for the WPC powder. The particle distribution ranged between 20 and 180  $\mu\text{m}$ , with 80% of the particles ranging between 20 and 40  $\mu\text{m}$ . Studies conducted by Keogh et al. (2003) showed that the mean particle size for milk concentrate, which varied between 43.5 and 121  $\mu\text{m}$ , depended on the amount of total solids, which varied between 42.8 and 52.3  $\mu\text{m}$ . The results obtained herein are in concordance with those obtained by Keogh et al. (2003) in terms of the total solid content and particle size; however, the same compounds that are in milk, such as fats and proteins, are not present in the whey. Other studies, such as that by Sootitawattawat (2005), reported that spray-dried milk samples with 20 g total solids/100 g had a particle size that varied between 23 and 70  $\mu\text{m}$ .

The smaller particle size obtained herein can be caused by drying conditions as a lower flux can produce large-sized drops and therefore large particles, whereas a higher flux can diminish the drop size and thus produce smaller particle sizes with lower moisture content because the water molecules in small droplets can be easily released during drying. According to Gharsallaoui, Roudaut, Chambin, Voilley, and Saurel (2007), the particle size and morphology of a powder is directly related to the physico-

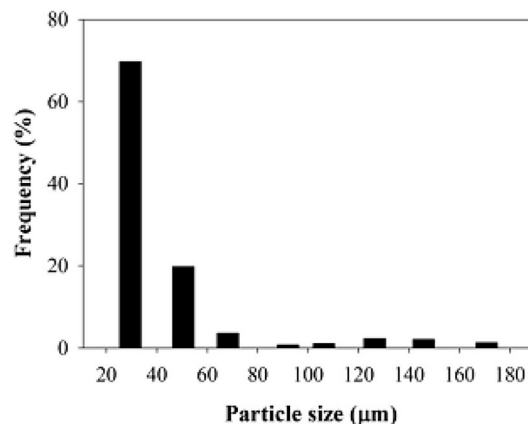


Fig. 3. Particle size distribution for the WPC powder obtained from the average of all treatments.

chemical changes produced in the particles during spray drying due to the moisture content and temperature used in the process.

### 3.2. Foaming capacity and foam stability

The average values obtained for the foaming capacity and foam stability were 5.51 mL and 5.28 min, respectively. Fig. 4 shows that a higher foaming capacity was obtained at  $170 \text{ }^\circ\text{C}$  but that at higher temperatures, the foaming capacity and stability tended to decrease, which may be due to the thermodenaturation of the protein during spray drying. At temperatures greater than  $180 \text{ }^\circ\text{C}$ , the foaming capacity and stability were also affected by the surfactant concentration. For instance, foaming capacity, at  $210 \text{ }^\circ\text{C}$  with a high content of surfactant, was close to 6.30 mL. This is an interesting result since surfactant molecules could be considered as protection agents when higher temperatures were applied. According to Yoshii et al. (2008) the addition of some surfactant offers a way to prevent direct contact of protein material with the high temperature during protein spray drying, since surface molecules tend to distribute on the surface of the particles, protecting the protein from thermal damage. In this work they found that enzyme

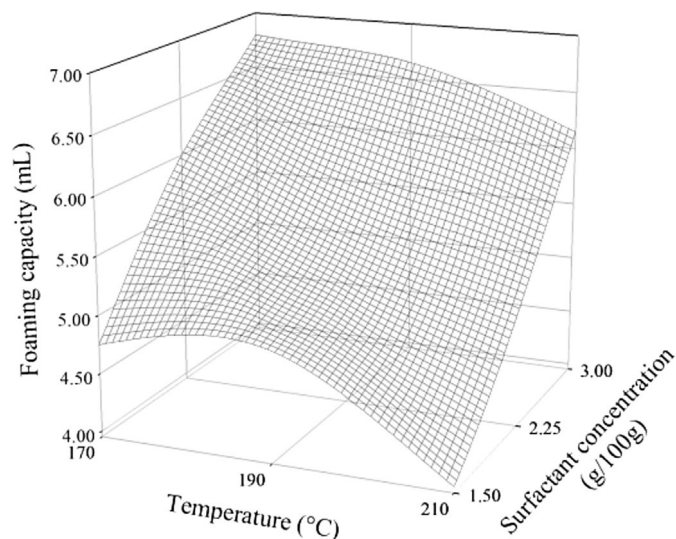


Fig. 4. Effect of temperature and surfactant concentration on the foaming capacity of the WPC powder.

retention was dependent on surfactant content, which was able to protect the enzymes from denaturation during spray drying.

According to Bernard et al. (2011), heating whey protein higher than 70 °C induces denaturation and, consequently, an unfolding of the whey protein molecules, exposing their reactive sites increasing the foam stabilization.

Analyzing the results obtained by Ayadi, Khemakhem, Belgith, and Attia (2008) in egg whites and whole eggs that were spray dried it is possible to understand the behavior of WPC obtained under our conditions. In that case, the flow rate influences the foaming capacity with temperature. For a lower flow rate (0.2 L/h), for example, the foaming capacity was higher than higher flow rate (0.3 L/h), but a higher temperature (125 °C) reduced the foaming capacity from 2.7 mL/mL at 120 °C to 2.3 mL/mL at 125 °C. The improvement of foaming capacity can be attributed to the partial protein unfolding that occurs during the heating process, which enhances the protein chain flexibility as long as aggregates are not formed.

With respect to foam stability (FS), Fig. 5 demonstrates that the FS was increased linearly with the Tween 60 surfactant concentration. In general, temperature played an important role in the foam stability, at 170 and 190 °C the foam stability was kept constant and decreased when the powder was at 210 °C. However, the surfactant content increases the foam stability even at 210 °C. According to Damodaran (1997), those polymeric species increase foam stabilization because they form an elastic interfacial film which increases the film viscoelastic properties, stabilizing the foam against gas diffusion during foaming and gravitational drain.

Foaming capacity and stability are related to the surfactant concentration because a higher amount of hydrophilic molecules is oriented to diminish the surface tension in the solution, thus allowing the formation of air bubble (Langevin, 2000; Monteux, Fuller, & Bergeron, 2004; Mukerjee, 1977; Wang & Yoon, 2006).

Other authors, such as Eisner, Jeelani, Bernhard, and Windhab (2007), have studied foam stability using non-ionic surfactant compounds, showing that the foam stability was significantly increased by inducing a lower surface tension in the solution. Cornec et al. (1998) found that a higher surfactant concentration improved the foam stability (Aken, 2003; Ariyaprakai, Limpachoti, & Pradipasena, 2013; Zhang, Xia, Liu, Xu, & Zhou, 2013).

The foaming capacity (FC) and stability (FS) are modeled according to the following equations:

$$FC = -7.11 + 0.14 \times T + 0.78 \times C - 0.00042 \times T^2 - 0.00060 \times TC + 0.12 \times C^2 \quad (4)$$

$$FS = -159.5 + 1.67 \times T + 9.63 \times C - 0.0044 \times T^2 - 0.026 \times TC - 0.56 \times C^2 \quad (5)$$

For the FC, the *R*-square was 88.76%, and the adjusted *R*-square was 84.74%; the significant term of the model was concentration. For the FS, the *R*-square reached 96.82%, and the adjusted *R*-square was 95.68%; the significant terms were concentration, temperature, the combination of temperature and concentration and temperature-squared ( $p < 0.05$ ). Thus, the FS equation can be considered adequate to model the foam stability when temperature and surfactant concentration are considered as processing variables.

The aim of this study was to identify the optimal conditions of temperature and surfactant concentration (Tween 60) to develop a foaming base for hot beverages. The values for foaming capacity and stability were determined considering that the optimal foaming in hot beverages is below 4 min, which is the typical consumption time. The optimal conditions were related to the minimal values of foam stability, which were near to 2.15 min. Thus, the optimal conditions of temperature and surfactant concentration were 210 °C and 1.5 g/100 g, respectively, resulting in a foaming capacity of 3.80 mL and moisture content of 1.82 g/100 g and apparent density of 0.181 g/cm<sup>3</sup>.

### 3.3. Sensory analysis

The WPC obtained under optimal conditions (210 °C and 1.50 g of surfactant/100 g of whey) were underwent to sensory analysis. The results obtained from the trained panel showed that dairy, cooked and, whey flavors were predominate (Fig. 6). The cooked taste was most likely due to the interaction between the proteins and sugars during the thermal process, which is the first mechanism of aromatic compound formation according to Mortenson, Vickers, and Reineccius (2008).

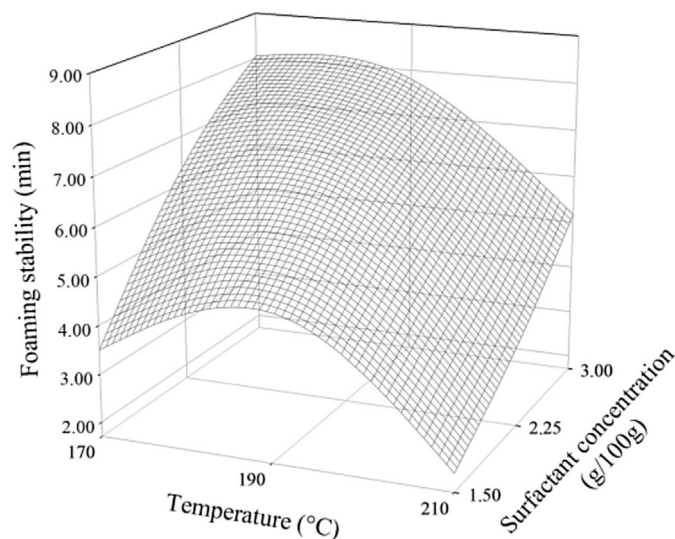


Fig. 5. Effect of temperature and surfactant concentration on the foaming stability of the WPC powder.

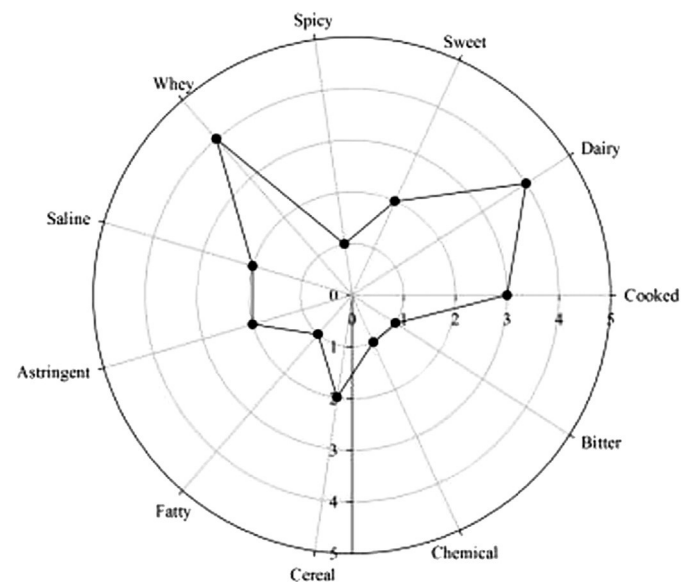


Fig. 6. Aroma profile for foams based on whey protein.

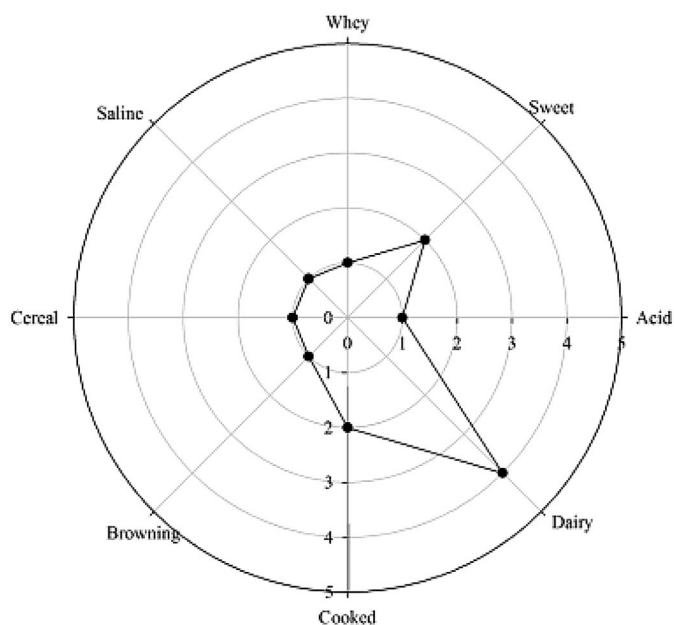


Fig. 7. Flavor profile for foams based on whey protein.

With respect to the taste of the foam, some trends toward dairy, sweet, and cooked tastes were identified, together with some metallic notes and a residual bitter taste (Fig. 7). According to the USDEC (2003), whey was described as a soft product with low sweet tastes that allowed other tastes and aromas, such as fruit and chocolate, to be released. However, other authors such as Drake, Karagul-Yuceer, Cadwallader, Civille, and Tong (2003) described some metallic tastes with a strong astringency, in accordance with our results.

#### 4. Conclusions

In this work, we demonstrated that controlling the spray-drying temperature and surfactant (Tween 60) concentration, whey protein concentrate could be developed from the waste by-product of dairy industry with foaming properties that can be used in hot drinks such as café or chocolate. The obtained WPC has the moisture content, density and particle size properties typical for this type of product, which allowed it to have a foaming capacity and stability adequate for use in hot beverages. The optimal conditions of the temperature and surfactant concentration were 210 °C and 1.50 g/100 g, respectively, leading to a foaming stability of 2.15 min, which is adequate considering the hot beverage industry criteria. Under these optimal conditions of temperature and surfactant concentration, foaming capacity of 3.80 mL, apparent density of 0.181 g/cm<sup>3</sup> and moisture content of 1.82 g/100 g were obtained and can be considered positively because allow reducing the packaging volume. The foaming capacity and stability were according to consumer demand and comparable with the sensory characteristics of dairy products (flavor and taste).

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