

Comparison of bioactive compounds and nutrient contents in whey protein concentrate admixture of turmeric extract produced by spray drying and foam mat drying

Jaqueline Vieira Piovezana Gomes^a, Lívyia Alves de Oliveira^a,
Stephanie Michelin Santana Pereira^a, Aline Rosignoli da Conceição^a, Pamella
Cristine Anunciação^{a,*}, Eliana Carla Gomes de Souza^a, Ítalo Tuler Perrone^b,
Mateus da Silva Junqueira^c, Helena Maria Pinheiro Sant'Ana^a, Ceres Mattos Della Lucia^a

^a Department of Nutrition and Health, Universidade Federal de Viçosa (UFV), Viçosa, Minas Gerais, Brazil

^b Faculty of Pharmacy, Universidade Federal de Juiz de Fora (UFJF), Juiz de Fora, Minas Gerais, Brazil

^c Department of Food Engineering, Universidade Federal de São João Del-Rei (UFSJ), 35701970 São João Del-Rei, Minas Gerais, Brazil

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ABSTRACT

We developed a whey protein admixture of turmeric extract by spray drying (TWPC-SD) and by foam mat drying (TWPC-FMD) and compared its bioactive compounds and nutrients contents. TWPC samples were evaluated for preference and acceptability. Vitamins and carotenoids were determined by high-performance liquid chromatography. Total phenolics, curcumin and antioxidant capacity were determined by spectrophotometry. Centesimal composition was performed according to the Association of Official Analytical Chemists. Chemical elements were determined by inductively coupled plasma optical emission spectrometry. TWPC containing 3.6 mg of curcumin showed good acceptability. Carotenoids and riboflavin were not detected in either TWPC. Vitamin C content was maintained, and antioxidant capacity was increased in both products ($p < 0.05$). TWPC-SD showed higher total phenolic and curcumin contents compared to TWPC-FMD ($p < 0.05$). Thus, the TWPC-SD is a good alternative for human consumption since it showed good sensory acceptability and its nutrients and bioactive compounds can contribute to human health.

1. Introduction

Whey accounts for 80–90% of the total milk volume used during cheese production and contains approximately 55% of the nutrients of milk (soluble proteins, lactose, vitamins, minerals and a minimum amount of fat). It is used by the food industry for the preparation of several products, such as milk drinks, ricotta, cookies, flour, animal food, powdered whey, and condensed whey (Alves et al., 2014).

Whey can be concentrated through membrane separation technology, which leads to the formation of protein products, which can be used as ingredients to improve the techno-functional properties of the food (solubility, gelatinization, viscosity, emulsification, foaming) (Alves et al., 2014). However, whey shows high perishability and low stability, and therefore drying methods are commonly employed to increase its

shelf life. Among them, spray drying and foam mat drying (Alves et al., 2014; Hardy & Jideani, 2017) are noteworthy.

Spray drying is a process of transforming a fluid into a dry product in a single operation with little effect on quality, being presented as a common method for encapsulation in the food industry (Begum & Deka, 2017). On the other hand, in foam mat drying, liquid foods are transformed into powder using emulsifying agents, with the advantages of being a simple method, with lower operating cost, in addition to allowing the use of lower temperatures during drying (Hardy & Jideani, 2017).

Whey protein has been studied and consumed by athletes and practitioners of physical activity because its content of branched-chain amino acids (leucine, isoleucine, and valine), bioactive peptides and calcium, being considered a product that shows physiological and

* Corresponding author at: Departamento de Nutrição e Saúde, Universidade Federal de Viçosa, Avenida Peter Henry Rolfs - Campus Universitário, 36571-900 Viçosa, MG, Brazil.

E-mail address: pamella.anunciacao@ufv.br (P.C. Anunciação).

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functional properties that intensify hypertrophy (Davies, Carson, & Jakeman, 2018).

Turmeric (*Curcuma longa* L.) is a medicinal plant native to India and Southeast Asia (Cañameres, García-Ramos, & Sanchez-Cortes, 2006). In the food industry, it is used as dye, flavoring and as an ingredient in the preparation of seasonings and processed products, including dairy products (Mohajeri, Behnam, Cicero, & Sahebkar, 2018). Its main phytochemical component is the polyphenolic compound known as curcumin (yellow pigment), which acts in the protection of cellular components and oxidative processes, combating free radicals, thus promoting the balance between pro-oxidant and antioxidant compounds (Cañameres et al., 2006; Mohajeri et al., 2018). However, curcumin is insoluble in water and ether. It is degraded in an alkaline solution and unstable to the presence of light, factors that usually limit its application in food (World Health Organization, 2004).

Since the whey protein concentrate admixture of turmeric extract (TWPC) has important functional and technological potential, it is important to evaluate which is the best drying method to obtain this product. Although foam mat drying is simple and low cost, our hypothesis is that spray drying, due to the shorter time employed, presents superiority in terms of preserving nutritional characteristics and greater industrial viability.

Thus, this study aimed to develop a TWPC through two drying methods (spray drying and foam mat drying) and compare its content of nutrients and bioactive compounds.

2. Materials and methods

2.1. Samples

2.1.1. Turmeric and turmeric extract

About 10 kg of turmeric was purchased from rural producers in the city of Viçosa, Minas Gerais, Brazil. The samples of turmeric were collected from different plants to obtain a representative sample. Only the rhizome was used to conduct this study. Turmeric was collected in this rhizome's natural habitat and the collection took place in December 2017.

The samples were transported to the laboratory in plastic bags protected from light since some bioactive compounds analyzed are sensitive to light oxidation. At the laboratory, the rhizomes were selected, sanitized, peeled and packed in polyethylene plastic bags in the approximate amount of 250 g. The packages were sealed, identified and stored in a freezer ($-18\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$) for up to six months. Throughout this process, the samples were protected from sunlight and artificial light, using amber glass, aluminum foil and blackout curtains.

The extract preparation was based on the analytical method of NBR 13624 (Lara, 1984), whose principle is the extraction of curcumin with ethanol, dilution, and spectrophotometric reading as described below.

The turmeric (sanitized and peeled) was weighed, placed in a domestic pan, and water was added over it (enough volume to cover the sample). The turmeric was then cooked for 20 min, measured from the moment at which the water reached boiling point. After cooking, the contents of the pan were placed in a container to cool down to room temperature.

Subsequently, with the aid of a vertical mixer SB40 (Black & Decker®, Towson, USA), the cooked turmeric was crushed, and alcohol (70%) was gradually added to extract curcumin (enough to crush the sample). The crushed mixture was covered with aluminum foil and allowed to settle down for 4 h.

After that period, the mixture (turmeric, water, and alcohol) was centrifuged (Nive® NF 1200 / 1200R, Ancara, Turkey) at 6000 rpm for 10 min. The supernatant was vacuum filtered using a Büchner funnel fitted with filter paper. The ethanol was evaporated from the mixture at $70\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ for 20 min using a rotary evaporator (QUIMIS® model Q344.1, São Paulo, Brazil) coupled to the vacuum pump (Tecnal® model TE058, São Paulo, Brazil). The extract was stored in an amber bottle in a

freezer ($-18\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$) for its use in the production of turmeric extract admixture of whey protein concentrate (TWPC) and for nutritional and technological analyses.

2.1.2. Whey

Whey was purchased and ultrafiltered (ultrafiltrator, GEA®, São Paulo, Brazil) in a dairy plant and transported immediately to the laboratory.

2.2. Pilot drying in spray drying

2.2.1. Development of TWPC in two dosages of curcumin

The pilot study was needed since the behavior of the turmeric extract added to the WPC in the spray drying was not known. This method has been chosen for being the most used in industrial production of whey protein, and also for its lowest operating cost.

Two formulations of WPC were developed and tested: one with addition of 7.2 mg and another with addition of 3.6 mg of curcumin per 30 g of whey protein portion. It was of interest to see whether, in the TWPC, the pungent and bitter taste of turmeric would impair the acceptance of the product by potential consumers, and whether this flavor would be perceptible to the point of influencing one's individual choice. To increase acceptability, the products were developed with and without artificial pineapple aroma, totaling four TWPC samples. For each 3 g of TWPC, 1 g of artificial pineapple flavored powder was added (composition: sugar, corn starch, acidulant, artificial flavoring, artificial colorings: tartrazine yellow and twilight yellow). The amount of flavor was calculated according to the product packaging guidelines.

For the calculation of the amount of turmeric extract to be added to WPC, the mean of acceptable daily intake of curcumin of 0–3 mg/kg body weight/day was considered (World Health Organization, 2004). The choice of the artificial pineapple aroma was based on the yellowish coloration presented by the developed TWPC.

The turmeric extract admixture of concentrated fluid whey was dried in a spray dryer (GEA®, São Paulo, Brazil) with inlet air temperature of $180\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ and outlet air temperature of $85\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ to obtain the TWPC for sensory analysis.

2.2.2. Sensory analysis

The four TWPC samples were evaluated for preference and acceptability on nonconsecutive days by untrained judges. The study was approved by the Research Ethics Committee with Human Beings (2,568,251) of the Universidade Federal de Viçosa (Minas Gerais, Brazil).

The preference evaluation was performed using the Friedman method, as suggested by Carneiro and Minim (2006). The samples were evaluated by 67 individuals (23.88% men and 76.12% women). On the other hand, the evaluation of acceptability was carried out only to the most preferred sample, using an hedonic scale of nine points, in a range from "Like extremely" to "Dislike extremely", scored by the participants (53 individuals – 41.51% men and 58.49% women).

In both tests, the samples were diluted (3 g TWPC in 30 mL of mineral water) and served in 50 mL disposable cups (refrigerated temperature), being presented simultaneously, coded with a random three-digit number. In addition to the samples, a glass of mineral water was used to clean the taste buds between the analysis of each product formulation (Carneiro & Minim, 2006; Reis & Minim, 2006).

According to the results of the sensory analysis, the sample containing 3.6 mg of curcumin with an artificial pineapple aroma was the most preferred by the volunteers (46.4%) and had a good acceptability (6.13 points), being located between the hedonic terms "Like slightly" and "Like moderately". So, this sample was selected to conduct the development of TWPC by two drying methods.

2.3. Development of TWPC by two drying methods

Water activity (a_w) was monitored during TWPC drying of the two methods at 25 °C using an Acqualab® thermohygrometer (Decagon 3TE, Decagon Devices Inc., USA). During foam mat drying, a_w was measured every hour until the final value of $a_w = 0.20 \pm 0.1$. In spray drying, a_w measurement was performed only at the end of the process, since this method does not allow access to the sample without turning the equipment off (Perrone, Simeao, Rodrigues Junior, Stephani, & de Carvalho, 2013).

Fig. 1 shows the flowchart of the drying process of turmeric extract admixture of whey protein concentrate (TWPC) by spray drying (SD) and foam mat drying (FMD).

2.3.1. Development of TWPC by spray drying

The drying procedure was conducted in a spray dryer model MSD1 1.0 (Labmaq®, São Paulo, Brazil) equipped with a one mm-diameter spray nozzle. The air and product flow rates, as well as air temperature, were defined by initial tests, following the methodology proposed by Perrone et al. (2013). In a plastic beaker, the retentate was weighed, and turmeric extract was added (3.6 mg curcumin for a 30 g portion of TWPC), with 6 to 6.5% yield, on average, after drying (de Oliveira Moreira, Junior, & Francisquini, 2017). The drying rate was 1 kg/h and the sample dried at 45 °C. After this drying period, the dried material was removed from the apparatus with the aid of plastic spatula. Then, the artificial pineapple aroma was added. The powder was stored in plastic packaging, wrapped in foil and stored in a freezer $-18 \text{ °C} \pm 1 \text{ °C}$.

2.3.2. Development of TWPC by foam mat drying

Initially the retentate was weighed in a plastic beaker, and turmeric extract (3.6 mg curcumin for a 30 g portion of TWPC) was added, with 6 to 6.5% yield, on average, after drying (de Oliveira Moreira, 2017). Then 8% of the emulsifying agent (composed of 20% carbohydrate and 26% lipid) was added (De Paula, 2015). The emulsifying agent was used to obtain the foam and to promote stability. By using a domestic shaker (Philips Walita®, São Paulo, Brazil), the blend was homogenized for 20 min at full speed until a consistent foam was obtained.

The resulting foam was placed in aluminum trays covered with parchment paper and placed in a food dehydrator model PD-15

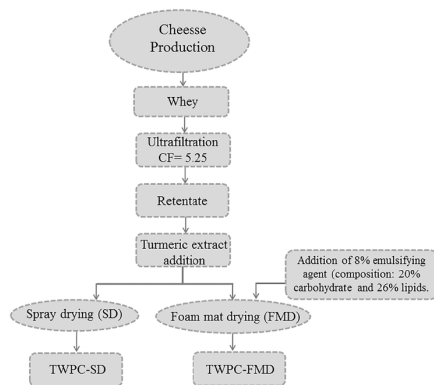


Fig. 1. Flowchart of sample production, in which Cf: concentration factor; TWPC-FMD: whey protein admixture of turmeric extract produced by foam mat drying; TWPC-SD: whey protein admixture of turmeric extract produced by spray drying.

(Polidryer®, Viçosa, Brazil) with air circulation, at 70 °C (Da Cruz, 2013). The trays were kept in the equipment until all the foam was converted to powder (9 h on average). At this time, the dried material was removed from the trays with a plastic spatula. Then, for each 3 g of TWPC, 1 g of the artificial pineapple aroma was added. The powder was stored in plastic containers, which were wrapped in aluminum foil and stored in a freezer $-18 \text{ °C} \pm 1 \text{ °C}$.

2.4. Analysis of vitamins, carotenoids, and bioactive compounds

Analyses were performed on the following samples: TWPC-SD, TWPC-FMD, and control (mixing of fluid whey, plus turmeric extract and pineapple aroma in the same amount added to the developed TWPCs). Curcumin was not analyzed in the fluid whey since this product is not a source of this compound. Riboflavin was analyzed only in samples of animal origin because they are considered major sources of this vitamin. The analyses were performed in quadruplicates.

2.4.1. Vitamin C (ascorbic acid)

The conditions used for the extraction and analysis of ascorbic acid (AA) were those optimized by (Campos, Ribeiro, Della Lucia, Pinheiro-Sant'Ana, & Stringheta, 2009). Five grams of samples (0.55 g of the powder sample diluted in 4.45 g of water) were added to 15 mL of extracting solution (metaphosphoric acid 3%, acetic acid 8%, sulfuric acid 0.3 mol/L and 1 mM EDTA) and homogenized in a micro-grinder (IKA® T18 basic Ultra Turrax, Staufen, Germany) for 5 min. After homogenization, the samples were centrifuged (Nüve®, NF 1200/1200R, Ancara, Turkey) at 4000 rpm for 15 min. The supernatant was vacuum filtered in a Büchner funnel with filter paper. Then, the filtrate was transferred to a volumetric flask and the volume completed to 25 mL with ultrapure water.

AA were analyzed using a high-performance liquid chromatography (HPLC) system (Shimadzu, SCL 10AT VP model, Japan) and diode-array detector (DAD) (Shimadzu, SPD-M10A, Japan). The following conditions were used: C-18 Synergi Hydro chromatographic column, 250 × 4.6 mm, 4 µm, fitted with a Phenomenex C18 guard column, 4 mm × 3 mm; mobile phase composed of 1 mM NaH_2PO_4 , 1 mM EDTA, pH 3.0, flow rate of 1.0 mL/min; running time of 7 min and injection volume of 10 µL. The chromatograms were obtained at 245 nm.

AA identification was performed by comparing the retention times and the absorption spectrum obtained for the commercial standard (Proquímios®, São Paulo, Brazil) and for the samples, analyzed under the same conditions. Quantification was performed by external standardization, using analytical curves constructed from injection, in duplicate, of six different concentrations of standard solutions. Ascorbic acid was expressed in mg/100 g of sample.

2.4.2. Carotenoids

The carotenoids (α -carotene and β -carotene) were extracted according to Rodriguez-Amaya (2001). Three grams of samples (0.33 g of the powder samples diluted in 2.67 g of distilled water) were homogenized in 20 mL of cooled acetone with a micro-grinder (IKA® T18 basic Ultra Turrax®, Staufen, Germany) for 2 min. The obtained suspension was vacuum filtered in a Büchner funnel with filter paper, keeping the residue in the extraction tube. Then, the extraction procedure was repeated for two more times, adding 20 mL of cooled acetone to the residue, with subsequent homogenization and vacuum filtration.

Subsequently, the carotenoids were partitioned from acetone to petroleum ether. The filtrate was transferred, in three fractions, to a separatory funnel containing 50 mL of cooled petroleum ether. After transferring each fraction, approximately 100 mL of distilled water was added for phase separation (carotenoids-petroleum ether and acetone-water), with the lower phase (acetone-water) being discarded. Anhydrous sodium sulfate was added to the ether extract to remove any water residue. Then, the extract was concentrated on a rotary evaporator (QUIMIS® model Q344.1, São Paulo, Brazil) at $35 \pm 2 \text{ °C}$,

transferred to a 25 mL volumetric flask, the volume completed with petroleum ether, and stored in an amber glass at $-18 \pm 2 \text{ }^\circ\text{C}$.

Carotenoid analyzes were performed according to Sant'Ana et al. (1998), using HPLC-DAD system. An aliquot of 2 mL of the sample was evaporated under nitrogen gas flow, and then recovered in 2 mL of HPLC grade acetone. The extract was filtered in filter units (0.45 μm , Millipore, Massachusetts, USA).

The following chromatographic conditions were used: Phenomenex® Gemini chromatographic column (250 \times 4 mm i.d., 5 μm) fitted with a Phenomenex® ODS guard column (C18, (4 mm \times 3 mm)); mobile phase composed of methanol: ethyl acetate: acetonitrile (80:10:10); isocratic elution; flow rate of 2.0 mL/min; volume injection of 30 μL ; run time of 12 min. The chromatograms were obtained at 450 nm.

The identification of carotenoids was performed by comparing the retention times and the absorption spectra of authentic standards (Sigma-Aldrich®, Germany) and samples analyzed under the same conditions. Quantification was performed by external standardization, using analytical curves constructed from the injection, in duplicate, of six different concentrations of standard solutions. Carotenoids were expressed in mg/100 g of sample.

2.4.3. Curcumin

Curcumin was extracted using the methodology of NBR 13,624 (Lara, 1984). The absorbance was measured in the Evolution 60S spectrophotometer (Thermo Scientific®, USA) at 540 nm. For the construction of the curcumin standard curve, 0.10 g of the curcumin standard (Sigma-Aldrich®, Germany) was dissolved in glacial acetic acid, in a 1000 mL flask, with the aid of heat. From this solution, aliquots of 0.2; 0.5; 1; 2; 5; 10; 15; 20 and 25 mL were transferred to 100 mL volumetric flasks, and their volumes were completed with glacial acetic acid. The absorbance of each solution was read to construct the standard curve.

2.4.4. Riboflavin

The determination of riboflavin was performed according to the method proposed by Bianchini and Penteadó (2000), with modifications. We used a solution of trichloroacetic acid (TCA) to a final concentration of 8% (20 mL serum fluid milk and 20 mL of 16% TCA solution). For the TWPC-SD and TWPC-FMD powder samples, dilution was performed based on the moisture of the fluid whey (2.2 g of powder sample diluted in 17.8 g of distilled water). The material was vacuum filtered in a Büchner funnel using filter paper and slowly neutralized with 10% NaOH to pH 6.0–6.5, using a pipette and magnetic stirring.

The volume of the extract was completed to 100 mL with ultrapure water, using a volumetric flask and then transferred to an amber glass bottle and kept refrigerated ($6 \pm 2 \text{ }^\circ\text{C}$), until the moment of analysis by HPLC. Prior to injection, the extract was filtered in filter units (0.45 μm , Millipore, USA).

The chromatographic conditions used were: HPLC system (Shimadzu, SCL-10AD VP, Japan), fluorescence detector (Shimadzu, RF10AXL, Japan) (456 nm excitation and 525 nm emission); C18 VP-ODS Phenomenex Gemini column (250 \times 4 mm, 5 μm); mobile phase composed of methanol: ultrapure water (40:60); flow rate of 1 mL/min, run time of 5 min.

The identification of riboflavin was performed by comparing the retention time of the commercial standard (Sigma-Aldrich, Germany) and samples analyzed under the same conditions.

2.4.5. Determination of total phenolic compounds and antioxidant capacity

2.4.5.1. Obtaining extracts. Two grams of the samples (0.22 g of powdered sample diluted in 1.78 g of distilled water) were added to 20 mL of acetone solution at 70%. The mixture was stirred in a metabolic bath (Marconi® model MA-093, São Paulo, Brazil) (180 rpm, for 15 min). The samples were centrifuged (Nüve® centrifuge, NF 1200 / 1200R, Ankara, Turkey) for 15 min. The extracts were stored in amber

bottles at $-18 \pm 1 \text{ }^\circ\text{C}$. Aliquots of the extracts were used for the estimation of total phenolics and antioxidant capacity (Bloor, 2001).

2.4.5.2. Estimation of total phenolic concentration. The concentration of total phenolics was estimated using the Folin-Ciocalteu reagent (Singleton, Orthofer, & Lamuela-Raventós, 1999). The absorbance was read in a spectrophotometer (Thermo Scientific®, Evolution 60S, USA), at 765 nm. The results were expressed in milligrams of gallic acid equivalents per 100 gram of sample (mg GAE/100 g).

2.4.5.3. Determination of antioxidant capacity. The radical removal activity (RRA) of the samples was determined using the DPPH (1,1-diphenyl-2-picryl-hydrazyl) radical method (Bloor, 2001). The absorbance was read in a spectrophotometer (Thermo Scientific®, Evolution 60S, USA) at 517 nm. The RRA was expressed in mmol of Trolox equivalent/100 g of sample (mmol Trolox/100 g).

2.5. Analysis of the centesimal composition

In order to verify if the addition of turmeric extract would modify the whey protein composition, we performed the analysis of the centesimal composition, according to the Association of Official Analytical Chemists (2012), in triplicate. The moisture content was determined by the gravimetric method at 105 $^\circ\text{C}$ until constant weight. The method 991.20 (N \times 6.38) was used to determine protein, and lactose was evaluated by the method ISO 22,662 / IDF198, lipids by the method 989.05 and ashes by the method 942.05 (Association of Official Analytical Chemists, 2012). The results were expressed as dry matter.

2.6. Determination of chemical elements

Nitroperchloric digestion of the chemical elements followed the methodology of Sarruge and Haag (1974). The determination of phosphorus-P occurred by calorimetry using the ascorbic acid method (Braga & Defelipo, 1974); potassium-K by flame photometry; and sulfur-S by turbidimetry (Alvarez, Dias, Ribeiro, & Souza, 2001). The other chemical elements (calcium-Ca, magnesium-Mg, sulfur-S, copper-Cu, iron-Fe, zinc-Zn, manganese-Mn, sodium-Na, chromium-Cr) were determined by inductively coupled plasma optical emission spectrometry (ICP-OES).

Analytical curves of the chemical elements were performed using increasing concentrations of the multi-element standard solution, according to the expected concentration. Results were expressed as mg/100 g.

2.7. Statistical analysis of data

A randomized blocks design was used. Data normality was evaluated by the Shapiro-Wilk test. To compare the concentration of vitamins and the bioactive compounds in the two developed TWPC (spray drying and foam mat), data were analyzed by ANOVA, followed by the Dunnett test ($\alpha = 0.05$). Statistical analyses were performed using SPSS software version 20.0.

3. Results and discussion

3.1. Concentration of vitamins and bioactive compounds in the developed products

3.1.1. Vitamin C

There was no difference in AA concentration between the control and the TWPC-FMD (Table 1). There was an increase in AA concentration in TWPC-SD when compared to control. The artificial aroma of pineapple had 127.53 mg/100 g of AA.

Vitamin C is the most chemically degradable (thermosensitive)

Table 1

Occurrence and concentration of vitamin C, carotenoids, riboflavin, curcumin, total phenolic compounds and antioxidant capacity of whey protein concentrates admixture by spray drying and foam mat drying.

Samples					
Compounds	Control	TWPC-FMD	% of losses	TWPC-SD	% of losses
Vitamin C (mg de AA/100 g)	11.42 ± 0.23 ^B	11.49 ± 0.93 ^{Ba}	–	12.50 ± 0.47 ^{Aa}	–
Carotenoids	nd	nd	–	nd	–
Riboflavin (mg/100 g)	nd	nd	–	nd	–
Curcumin (mg/100 g)	1.45 ± 0.03 ^A	0.87 ± 0.12 ^{Bb}	40.0	1.10 ± 0.22 ^{Ba}	24.13
Total Phenolics (mg GAE/100 g)	7.00 ± 0.00 ^A	2.00 ± 0.00 ^{Bb}	71.43	4.00 ± 0.01 ^{Ba}	57.14
Antioxidant capacity (mmol trolox/100 g)	6.00 ± 0.00 ^B	123.00 ± 0.29 ^{Ab}	–	182.00 ± 0.39 ^{Aa}	–

^aMean ± standard deviation of quadruplicates, as wet matter. Means followed by the same lowercase letter in the lines do not differ by the t-Test. Means followed by the same capital letter in the lines do not differ by Dunnett's test ($p < 0.05$). AA: ascorbic acid; Control: fluid whey, plus extract of turmeric and pineapple aroma; TWPC-SD: whey protein concentrate with turmeric extract added by spray drying; TWPC-FMD: whey protein concentrate with turmeric extract added by foam mat drying; GAE: equivalents of gallic acid; nd: not detected.

among vitamins; its retention is considered an index of maintenance of nutritional quality during processing and storage (Cunha, Silva, Costa, Teodoro, & Koblitz, 2014). Since the pineapple aroma was added after drying TWPC, maintaining the concentration of AA in the developed products and control indicates that the extraction and analysis of this compound were performed efficiently. According to Miller, Jarvis, and McBean (2006), in dried whey protein, there is on average 0.1 mg/100 g of vitamin C (wet matter). This value was lower than that found in the samples analyzed in our study. That is, in our samples, the highest concentration of this compound can be explained by the addition of the artificial pineapple aroma (added after drying) in the developed products.

3.1.2. Carotenoids and riboflavin

No carotenoids and riboflavin were detected in either TWPC-SD or TWPC-FMD (Table 1). Ultrafiltration is used for retention of macromolecules and colloids present in a solution and it is used in the dairy industry, among other applications, to concentrate the protein fraction of whey, which is retained by the membranes (retentate or concentrate - fraction studied in the present study) (Fig. 1). The fraction that permeates the membranes during ultrafiltration is called permeate, and B vitamins and carotenoids are found in this fraction. This is due to the hydrophilic character and the low molar mass (Catarino, Martins, Duarte, Prudêncio, & de Pinho, 2013), which justifies the non-detection of riboflavin and carotenoids in the present study.

3.1.3. Curcumin

There was a reduction in curcumin concentration in the powders compared to the control mixture (fluid whey, turmeric extract and artificial pineapple aroma), with a consequent reduction in total phenolics (Table 1), since curcumin is a phenolic compound (Mohajeri et al., 2018) (Table 1). Higher curcumin contents were found in TWPC-SD (retention of 75.87%) when compared to TWPC-FMD (retention of 60%).

3.1.4. Phenolic compounds

Total phenolic losses were 71.43% (TWPC-FMD) and 57.14% (TWPC-SD), respectively (Table 1). The reduction of total phenolics after spray drying was also reported by Lim, Ma, and Dolan (2011) in

blueberry (losses between 8.22 and 17.52%). In another study (Senica, Veberic, Grabnar, Stampar, & Jakopic, 2016), the authors observed reductions of total phenolic compounds after drying the samples, with losses of 84.4% (green persimmon) and 86.66% (ripe persimmon), higher than our results.

The antioxidant characteristic of phenolic compounds makes them susceptible to oxidation degradation, which can be influenced by the presence of oxygen, light, and heat, explaining the losses found (Davey & Montagu, 2000).

3.1.5. Antioxidant capacity

The increase in antioxidant capacity was observed in the products developed by the two drying methods (Table 1). This increase was also reported by Mechlouch et al. (2012) after comparing three drying methods (microwave, outdoor drying, and direct sunlight drying) to obtain high-quality tomatoes. This is due to the inactivation of peroxidases, which show pro-oxidant activity, or the formation of new antioxidant compounds, or the improvement of the antioxidant capacity of naturally occurring compounds (Davey & Montagu, 2000).

To date, no other studies have been conducted to evaluate the concentration of vitamins, total phenolics, and antioxidant capacity in WPC.

In general, TWPC-SD showed lower losses of vitamin C, total phenolic compounds and curcumin, demonstrating that the drying method used is more efficient for the production of TWPC and in the preservation of these compounds, probably because of the shorter time and temperature of the particle that is employed in this process.

3.2. Centesimal composition

The analysis of the centesimal composition was carried out only in the developed product that showed lower nutritional losses after the drying methods employed. Thus, this analysis was performed only in the TWPC-SD, which presented 52.01% of lactose, 33.76% of proteins, 7.93% of ashes, 3.63% of lipids and 4.1% of moisture (data on dry matter). All values found for the TWPC-SD centesimal composition are as expected for WPC according to the methodologies employed by the Institute (2018), in which whey protein concentrate (WPC-34) must contain at least 33.5% protein, a maximum of 52% lactose, up to 5% milk fat, 6.5–8.0% ash and maximum moisture of 6% (dry matter).

3.3. Chemical elements

The most abundant chemical elements in TWPC-SD (as dry matter) were K (1025 mg/100 g), P (266.5 mg/100 g), Ca (310 mg/100 g), Na (121 mg/100 g) and Mg (57.5 mg/100 g), a finding that is in agreement with that described by Carvalho (2007) for whey protein. Our findings were lower than those verified by Baldasso, Barros, and Tessaro (2011) for sweet whey protein: P (931 mg/100 g); K (2080 mg/100 g); Ca (796 mg/100 g); Mg (176 mg/100 g); Zn (1.97 mg/100 g) and Na (1079 mg/100 g). This lower concentration of minerals can be explained by the whey ultrafiltration to obtain the retentate, since this process promotes variation in the concentration ratio between components due to protein retention and selective permeation of lactose, minerals, water, and compounds of low molar mass (Baldasso et al., 2011). There are few studies in the literature that have evaluated mineral composition in WPC, which demonstrates the pioneering nature of the present study.

No heavy metals (nickel, lead, and cadmium) were found in the analyzed sample. The ingestion of heavy metals is toxic to the body, being bioaccumulative, leading to adverse effects to human health in the long term, besides causing chronic intoxication effects and neurological alterations (Rocha, 2009).

4. Conclusion

Vitamin C was preserved, and total antioxidant capacity was increased in both TWPC developed. TWPC-SD showed characteristic

centesimal composition of WPC. Also, TWPC-SD showed lower losses of phenolic compounds and curcumin, demonstrating that this drying method is more efficient to produce TWPC and to preserve these compounds.

Thus, TWPC produced by spray-drying is a good alternative for the food industry and for human consumption since it showed good sensory acceptability and important bioactive compounds.

CREdIT authorship contribution statement

Jaqueline Vieira Piovezana Gomes: Conceptualization, Methodology, Investigation, Writing - original draft. **Lívia Alves de Oliveira:** Investigation. **Stephanie Michelin Santana Pereira:** Investigation. **Aline Rosignoli da Conceição:** Investigation. **Pamella Cristine Anunciação:** Conceptualization, Writing - review & editing. **Eliana Carla Gomes de Souza:** Conceptualization, Methodology, Writing - review & editing. **Ítalo Tuler Perrone:** Conceptualization, Methodology, Resources, Writing - review & editing, Supervision. **Mateus Silva Junqueira:** Resources, Writing - review & editing, Supervision. **Helena Maria Pinheiro Sant'Ana:** Conceptualization, Methodology, Resources, Writing - review & editing, Supervision. **Ceres Mattos Della Lucia:** Conceptualization, Methodology, Resources, Writing - review & editing, Supervision, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.foodchem.2020.128772>.

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