

Whey Protein Concentrate admixture of Turmeric Extract: The Effect Of Drying Method

Concentrado Proteico do Soro do Leite Adicionado de Extrato de Cúrcuma: O Efeito do Método de Secagem

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This study aimed to characterize whey protein concentrates (WPC) added to turmeric extracts obtained by spray and foam mat drying (approximately 3.6 mg of turmeric was added to 30 g of whey protein). Drying parameters, morphology, particle size distribution, Raman spectroscopy, total phenolic compounds, curcumin quantification, and antioxidant capacity measurements were carried out. The spray drying process exhibited a higher drying efficiency, and was faster. The drying method significantly influences the morphology of the powders. There was a significant statistical difference between samples in relation to size distribution ($<1 \mu\text{m}$; d_{90}). Raman spectroscopy data showed that lactose remains in an amorphous state after only spray drying. WPC spray drying showed lower losses of curcumin (24.13 %) and total phenolics (57.14 %) compared with WPC dried by foam mat drying (40.00 %; 71.43 %, respectively). An increase in antioxidant capacity was observed for both spray-dried ($182 \pm 0 \text{ mmol trolox}/100 \text{ g sample}$) and foam mat-dried ($123 \pm 0 \text{ mmol Trolox}/100 \text{ g sample}$).

Keywords: Morphology; energetic balance; raman spectroscopy; antioxidant capacity; spray drying; foam mat drying.

1. Introduction

Turmeric (*Curcuma longa* L.), also known as yellow ginger or saffron, is a medicinal plant species native to India and Southeast Asia.¹⁻² Its rhizome is used by the food industry as a dye, flavoring, and seasoning component in processed foods and dairy products.² Furthermore, physiologically, turmeric protects cell components and prevents oxidation by neutralizing free radicals, promoting a balance between pro-oxidant and antioxidant compounds.³

Turmeric has an important constituent, a yellow pigment formed by a hydroxyl radical called curcumin.² The curcumin has been used as an antimicrobial, anti-inflammatory, antioxidant (free radical scavenging and metals chelating), and cardioprotective agent.²⁻⁴ This bioactive compound in turmeric is sensitive to alkaline conditions, heat, light, metal ions, enzymes, oxygen, and ascorbic acid. It is often encapsulated to improve its solubility, stability, and bioavailability.⁵⁻⁶

Whey protein is often studied as a natural vehicle for bioactive components for medical and food applications because of its ability to form gels and microcapsules. These characteristics allow it to control the release of bioactive/nutritional substances into the body.⁷⁻⁸ It is also worth mentioning that this milk protein is often used as a protein supplement to strengthen and promote muscle gain in athletes; moreover, it enhances physiological activity, improves immunomodulatory capacity, and antibacterial functions.⁹⁻¹⁰

A product manufactured of whey protein concentrate and turmeric extract can have enhanced nutritional benefits and would be a product with high market potential because of the benefits to the human body that both curcumin and whey protein provide (antimicrobial, antibacterial, anti-inflammatory, antioxidant, cardioprotective agent, physiological activity, improves immunomodulatory capacity), resulting in a product with differentiated nutritional properties.^{2-4;9-11}

The food industries make use of different drying methods. It is known that the selected drying method can affect the physicochemical, compositional, structural, and nutritional properties of the powder. In the literature, for drying dairy products, fruits, and vegetables, two methods are studied: foam mat drying and spray drying.¹²⁻¹⁵ Foam mat drying is a method that is simple, inexpensive, and accessible for firms with small budgets and commercial installations.¹²⁻¹⁴ However, spray drying, which requires considerable investments in physical space and

equipment, is widely used in food and pharmaceutical industries.¹⁵

In view of the above, the aim of this work is to characterize whey protein concentrate added to turmeric extract dried by two different methods: foam mat drying and spray drying.

2. Materials and Methods

2.1. Turmeric extract

Turmeric (*Curcuma longa* L.) was purchased from rural producers in the city of Viçosa, Minas Gerais, Brazil (Santa Cruz Waterfall, Lotus Site, Serra do Brigadeiro road, countryside without number, Zip code 36574-970). Representative samples were collected from different plants and only the rhizomes were used in this study.¹¹ The analytical method used for obtaining curcumin was based on NBR 13624 and is described in Gomes *et al.*¹¹

The turmeric (sanitized and peeled) was placed in a household pan, and water was added over it (enough amount to cover the sample). The turmeric was cooked for 20 minutes, starting from the moment the water boiled. After cooking, the contents of the pot were placed in a container until they reached room temperature. Subsequently, with the aid of a vertical mixer (Black&Decker® SB40), the cooked turmeric was crushed, and 70% alcohol was added little by little to extract the curcumin (enough quantity to crush the sample). The crushed mixture was covered with aluminum foil and allowed to rest for 4 hours. After this period, the mixture (turmeric, water and alcohol) was centrifuged (Multi-purpose® centrifuge, NF 1200/1200R) at 6000 rpm for 10 minutes. The supernatant was transferred and vacuum filtered in a Büchner funnel with filter paper and the alcohol was evaporated at $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 20 minutes in a rotary evaporator (QUIMIS® model Q344.1) coupled to a vacuum pump (TECNAL® model TE058). The extract was stored in an amber bottle in a freezer at $-18^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for use in the production of whey protein concentrate.

2.2. Drying of whey protein concentrate

Whey protein concentrate (WPC-SD) and whey protein concentrate added to turmeric extract (CWPC) were dried in a spray dryer (CWPC-SD) MSDi 1.0 (Labmaq do Brasil®, Brazil) equipped with a 1-mm spray nozzle and foam mat dryer (CWPC-FMD) PD-15 model food dehydrator (Polidryer®, Brazil). Foam mat drying is a technique where liquid products are converted into foam. Therefore, to promote good foam stability, the addition of an additive is necessary. Which consequently can increase stability during the drying process.¹⁶ According to De Paula,¹⁶ through tests carried out using different drying methods and different percentages of stabilizers, it was observed that the use of 8% of emulsifying agent resulted in greater

stability of the foam and better stability of the drying process. Such, the CWPC-FMD was added to $8\% \text{ w.w}^{-1}$ of an emulsifying agent (Emustab®, in proportion $20\% \text{ w.w}^{-1}$ carbohydrates and $26\% \text{ w.w}^{-1}$ lipids, where in the portion of 10.0 g (1 tablespoon) there are 1.0 g of carbohydrate, 2.3 g of total lipid and 2.2 g of saturated lipid). The blend was homogenized in a food processor (Philips Walita®) for 20 minutes at a high speed and room temperature until a thick foam formed. The foam was placed in aluminum trays covered with parchment paper, which were put into the foam mat dryer.

Approximately 3.6 mg of turmeric was added to 30 g of whey protein (serving size). As turmeric has a distinctive, pungent flavor, an artificial pineapple flavoring agent (ingredients list: Sugar, cornstarch, citric acid, artificial flavoring, and artificial coloring [yellow tartrazine and dusky yellow], where: in the portion of 50 g (5 tablespoons) there are 28 g of carbohydrate, 7 g of protein, 2 g of total lipid, 248 mg of sodium) was added to the sample. One gram of artificial pineapple flavoring was added to every three grams of CWPC according to product label instructions. The amount of turmeric extract added to the WPC was calculated using the mean recommended daily intake of curcumin ($0\text{--}3 \text{ mg.kg}^{-1}$ body weight/day).¹⁷

Whey was ultrafiltered (GEA®, Germany) and its retentate was divided into two aliquots to be dried according to the aforementioned methods. During the drying processes, CWPC water activity (a_w) was monitored at 25°C using a dalmachyrometer Aqualab (Decagon® 3TE, Decagon Devices Inc., USA) for both methods. Water activity was measured every hour (in replicates) until $a_w = 0.20 \pm 0.10$ in the foam mat equipment. However, for the spray dryer, a_w analysis was performed only at the end of the drying period, as the method does not allow sample access while the equipment is in use.¹⁸

For comparison purposes, a HygroPalm22-A thermohygrometer probe (Rotronic®, Switzerland) was placed onto both the spray dryer and foam mat dryer to monitor temperature ($^{\circ}\text{C}$), relative humidity (%), and absolute humidity ($\text{kg water.kg dry air}^{-1}$) in inlet and outlet air.¹⁹

Each formulated product (WPC-SD, CWPC-SD, CWPC-FMD) was dried in three replicates ($n=3 \times 3$).

To perform the analysis (by scanning electron microscopy, particle size distribution, and Raman spectroscopy), the turmeric extract was freeze-dried (TE-FD) in a freeze-dryer (FreeZone 2.5 Liter Benchtop Freeze Dry System with PTFE-coated collector).

Figure 1 schematically summarizes the production flowchart performed in the present work.

2.3. Drying parameters

The drying parameters of water activity (dalmachyrometer Aqualab, Decagon® 3TE, Decagon Devices Inc., USA), evaporated water, drying duration, and inlet and outlet

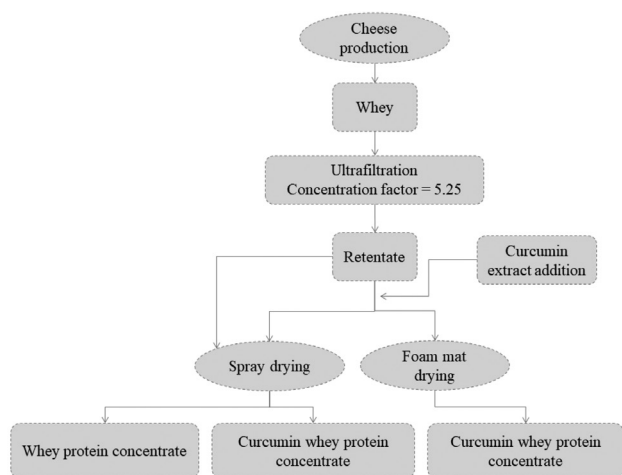


Figure 1. Sample production flowchart.

air temperature (HygroPalm22-A thermohygrometer probe, Rotronic®, Switzerland) were analyzed to compare the powders subjected to spray and foam mat drying. The parameters followed the methodology proposed by Gomes *et al.*,¹¹.

2.4. Morphology characteristics

The morphology characteristics of the powders (TE-FD, WPC-SD, CWPC-SD, and CWPC-FMD) were determined using a scanning electron microscope (Hitachi®, TM3000, Tokyo, Japan). At 500x magnification.

2.5. Particle size distribution

Particle size distribution was determined using a Beckman Coulter LS 13 320 laser diffraction analyzer (Beckman Coulter®, FL, USA) coupled to a liquid analysis module (Beckman Coulter®, FL, USA). The TE-FD, WPC-SD, CWPC-SD, and CWPC-FMD were tested without prior rehydration. Sufficient amounts of the powder were slowly added to the reservoir containing water at room temperature until an opaque mixture was obtained. Five series of data were collected in the region 0.375 – 2000 μm at 90 second intervals (1.5, 3.0, 4.5, 6.0, and 7.5 minutes). The results were obtained using the Fraunhofer approximation for total solubility. To determine their size, the data were given as the percentage (%) of volume that the particles occupied. Statistical analysis of the data was carried out using Beckman Coulter software (Particle Characterization, version 5.03). The analysis was performed in triplicate.

2.6. Raman spectroscopy

Raman spectra were obtained for TE-FD, WPC-SD, CWPC-SD, and CWPC-FMD using a FT-Raman spectrometer (Bruker®, model RFS 100) equipped with a Ge detector cooled using liquid nitrogen and a laser (Nd: YAG with 1064 nm frequency). A 90-mW beam was directed at

the samples. A strong signal-to-noise ratio was obtained for all spectra using an average of 512 scans. These were collected at a spectral resolution of 4 cm⁻¹ using OPUS 6.0 software.²⁰ The analysis was performed in triplicate.

2.7. Curcumin quantification

0.10 g of the following samples were added to 100 mL volumetric flasks: CWPC-SD, CWPC-FMD, and control sample (liquid whey with turmeric extract and pineapple flavoring in the same amounts added to the CWPC samples). Then, 60 mL of glacial acetic acid was added to the flasks and stirred at 180 rpm (Dubnoff®, model MA-093) at 90 °C for 1 h. 2 g of boric acid and 2 g of oxalic acid were then added and stirred for 10 minutes. After returning to room temperature, they were filled with glacial acetic acid and homogenized. Aliquots of 5 mL were transferred to 50 mL volumetric flasks, which were then filled with glacial acetic acid.

Absorbance levels were measured using an spectrophotometer at 540 nm (ThermoScientific®, USA, UV-Vis spectrophotometer Evolution 60S, detector type was dual silicon photodiodes, light source was xenon lamp and the cuvette used was made of quartz).

To plot the curcumin curve standard curcumin (Sigma-Aldrich®) was dissolved in glacial acetic acid. The curcumin concentrations used to plot the curve were: 0.17, 1.34, 3.04, 5.56, 13.21, 25.67, 32.74 and 34.04 μl/mL. The absorbance levels were recorded at 540 nm to determine an analytical curve (coefficient of variation $R^2 = 1.0$).

The analysis was performed in triplicate.

2.8. Total phenolic compound

For analysis, 2 g of the control sample (liquid whey with turmeric extract and pineapple flavoring in the same amounts added to the CWPC samples) was measured on a liquid scale. For CWPC-SD and CWPC-FMD, 1.78 g of distilled water and 0.22 g of each powdered sample were weighed, to obtain a total of 2 g (wet matter). Then, 20 mL of acetone was added to the sample, and the mixtures were stirred (Dubnoff®, model MA-093) at 180 rpm for 15 minutes and centrifuged (Multi-purpose® centrifuge, NF 1200/1200R) at 14000 rpm for 15 minutes. Supernatant fractions were transferred to amber bottles and stored at -18 ± 1 °C. Aliquots of the extracts were used to estimate total phenolic compounds and antioxidant capacity.²¹

The total phenolic concentration was estimated using a Folin–Ciocalteu reagent.²² In test tubes, 500 μL of each extract were added to a 500 μL Folin–Ciocalteu 20% solution and 500 μL sodium carbonate solution (7.5% in water) and stirred by vortex (Biosan®, Brazil). After 30 min in the dark, the absorbance levels were determined using a spectrophotometer (ThermoScientific®, Evolution 60S, USA) at 765 nm.

An analytical curve of gallic acid (Sigma-Aldrich®, Germany) (0.005–0.10 mg·mL⁻¹) was used to quantify

the phenolic compounds. The results were expressed in milligrams of gallic acid equivalents.100 g⁻¹ of the sample (mg GAE.100 g⁻¹).

The analysis was performed in triplicate.

2.9. Antioxidant capacity

Antioxidant capacities for the samples were determined for CWPC-SD, CWPC-FMD, and the control sample (liquid whey with turmeric extract and pineapple flavoring in the same amounts added to the CWPC samples). The sample radical removal activity (RRA) levels were determined using the 1,1-diphenyl-2-picryl-hydrazyl (DPPH) radical method.²² A 100- μ L aliquot of each sample was added to 1500 μ L of 0.1 mM DPPH methanolic solution (Sigma-Aldrich®, Germany) and stirred by vortex. After 30 minutes of standing, the absorbance levels were determined using a spectrophotometer (ThermoScientific®, Evolution 60S, USA) at 517 nm. An analytical curve was developed using 50–100 mmol.L⁻¹ of trolox solution. The RRA was expressed in mmol of Trolox equivalent.100 g⁻¹ of sample (mmoltrolox.100 g⁻¹).

The analysis was performed in triplicate.

2.10. Statistical analysis

In the present work there are more than two treatments (n=3), therefore, in this case it is necessary to apply a test to compare the means of the treatments. The tests chosen were: Dunnet test and Tukey test (p<0.05). Tukey's test was performed using statpages and Dunnet's test using IBM SPSS Statistics version 20.

3. Results and Discussion

3.1. Drying parameters

Table 1 compares the drying parameters of the two different methods, spray drying and foam mat drying.

The water mass that evaporated by dry air was 5.1 times higher during the spray drying process compared with the foam mat drying process (22.24 g of water·kg of dry air⁻¹ and

4.35 g of water·kg of dry air⁻¹, respectively). In addition, the average drying duration for foam mat drying was 7.7 times longer than for spray drying (Table 1). This demonstrates that the spray drying process exhibited a higher drying efficiency, and was faster than the foam mat drying process. The spray dryer evaporated water mass to dry air mass ratio is in accordance with previous studies using a pilot spray dryer.²³⁻²⁴ Silva *et al.*²⁴ evaporated fluid milk using the same pilot dryer and obtained 24.4 g of water·kg⁻¹ of dry air.

The stability of dehydrated dairy products is directly related to product composition, water activity, temperature, and relative humidity during storage.²⁵⁻²⁶ Dried dairy products must have water activity levels between 0.10 and 0.20.²⁷ In our study, foam mat drying produced a powder with higher water activity (aw = 0.25 \pm 0.05) than the spray-dried powder (aw = 0.16 \pm 0.01). Therefore, it can be verified that the powder produced via spray drying exhibited the closest water activity to that considered ideal for dried dairy products such as WPC.

Spray drying is widely used to encapsulate natural compounds and microorganisms because, among other advantages, the average particle drying time ranges from 6 to 17 s.²⁸⁻²⁹ Thus, despite the drying temperature being higher, the heat exposure time is shorter, which allows for better component conservation conditions.³⁰ Hence, TWPC-FMD was determined to be less energy efficient than TWPC-SD.

Schuck *et al.*³¹ described the properties of air during drying using the Moillier diagram. In Figure 2, the data obtained from the experiment performed here was used in conjunction with the Moillier diagram.

Applying the diagram to the data from this experiment, the ambient inlet air for the spray dryer before heating is identified by number (1). At this point, the ambient air has a temperature of 25 \pm 3 °C and an absolute air humidity of 10.0 \pm 0.6 g·H₂O·kg⁻¹ of dry air. When passing through the air heating system of the spray dryer, the properties of the air shift to those represented by number (2) on the graph, with a temperature of 160 \pm 1 °C. From this point, the heated air comes into contact with the droplets of whey protein concentrate coming from the atomizer. A change in the physical properties of the air is observed after contact with the atomized droplets of the whey protein concentrate, making this air colder and more humid, decreasing its

Table 1. Comparison of different methods and parameters of drying for whey protein concentrate, and whey protein concentrate with turmeric extract added

Attribute	Drying method	
	Spray drying	Foam mat drying
Evaporated water (g of water·kg of dry air ⁻¹)	22.24 \pm 0.21 ^b	4.35 \pm 0.73 ^a
Drying duration (minutes)	70 \pm 10 ^a	540 \pm 30 ^b
Water activity (a _w)	0.16 \pm 0.01 ^a	0.25 \pm 0.05 ^a
Inlet air temperature (°C)	160 \pm 1 ^b	70 \pm 1 ^a
Outlet air temperature (°C)	91 \pm 2 ^b	68 \pm 2 ^a

*Data expressed as mean \pm standard deviation (n=3). **Different superscript letters within the same line indicate significant differences by Tukey's test (p<0.05).

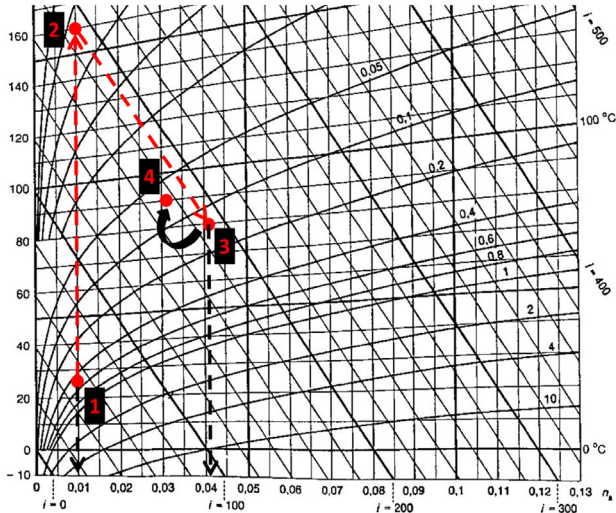


Figure 2. Moillier diagram applied to the data obtained from the experiment. (1) is ambient inlet air for spray dryer before heating, (2) is the point at which heated air comes into contact with droplets of whey protein concentrate from the atomizer, (3) is the outlet air in equilibrium with the whey protein concentrate powder (at the final moment during drying), (4) is the deviation indicating the difference between the drying of pure water drying of a food.

temperature, and increasing its absolute humidity. These two changes occur simultaneously, and, considering that all the energy from this process is transferred from the kinetic energy of the air molecules to the potential energy of the water molecules of the whey protein concentrate, then the process is isenthalpic. The consequence of this is the change in the air properties from number (2) to number (3) on the graph. If we consider that in number (3), the outlet air is in equilibrium with the whey protein concentrate powder, then, we define it as the end of drying. In number (3), it can be seen that the air temperature, initially at 160 °C, is now close to 80 °C, which is the exit air temperature (T_{as}) of the chamber. By extending the point indicated by number (3) to the curve of 100 % relative humidity, we find the wet bulb temperature (T_{bu}). In this way, the temperature of the whey protein concentrate particles during drying must be located between T_{as} and T_{bu} . From the point indicated by number (3), it is also possible to observe the absolute humidity of the air at the end of drying (A_{Ho}), with an approximate value of 41 g·H₂O·kg⁻¹ of dry air. The difference between A_{Ho} and A_{Hi} (41 - 10 = 31 g·H₂O·kg⁻¹ of dry air) indicates the theoretical mass of water evaporated per kilogram of dry air. The point indicated by number (3) on the curve corresponds to a relative humidity of 16% or a water activity equal to 0.16, with 0.16 ± 0.01 being the average value for the dry products in this experiment for the spray dryer method. Considering the properties of the air in the spray dryer measured during the drying process, a theoretical evaporated water mass per gram of dry air would be expected to be 31 g·H₂O·kg⁻¹; however, the experimental evaporated water mass was 22.24 ± 0.21 g·H₂O·kg⁻¹. The difference from the theoretical value for the obtained value is related to the deviation indicated by point 4 in Figure 2,

and consists of the difference between the drying of pure water and the drying of a food.

3.2. Morphology characteristics and particle size distribution

Figure 3 shows both the microscopy and particle size distribution of the samples.

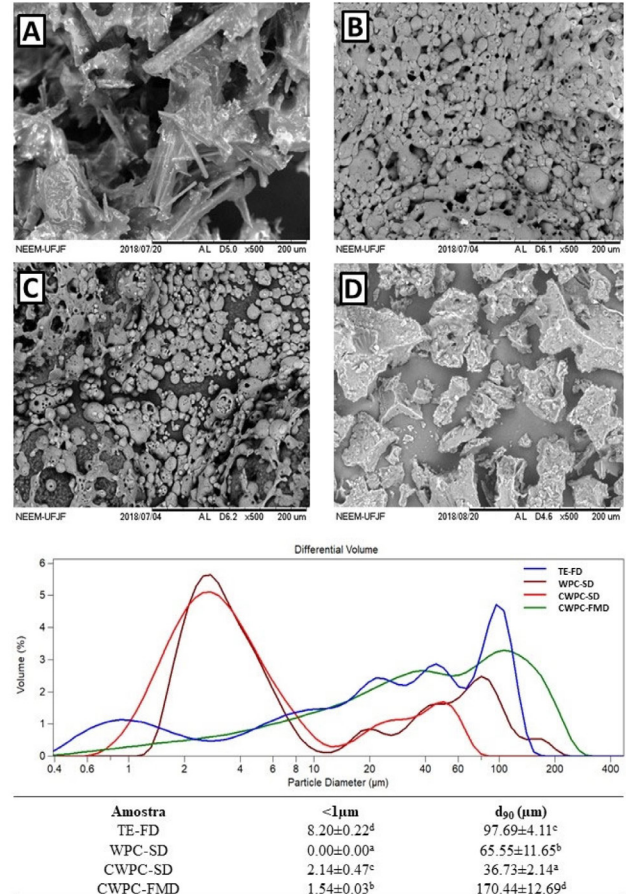


Figure 3. Results of particle size analysis and scanning electron microscopy (x500): (A) turmeric extract freeze-dried (TE-FD); (B) whey protein concentrate (WPC-SD); (C) whey protein added to turmeric extract produced by spray drying (CWPC-SD); (D) whey protein added to turmeric extract produced by foam mat drying (CWPC-FMD). *Data expressed as mean ± standard deviation (n=3). **Different superscript letters within the same column indicate significant differences by Tukey's test (p<0.05). ***The particle size distribution graphs correspond to analysis time 1 (1.5 minutes), whereas the result found in the table (<1 µm and d₉₀) is based on the average of the results found in the five analysis times (1.5, 3.0, 4.5, 6.0, and 7.5 minutes).

The turmeric extract sample demonstrated a morphology (Figure 3-A) similar to that found in literature.³² Moreover, the morphological characteristics of WPC-SD and CWPC-SD (Figure 3-B and 3-C) had the characteristics of spherical and bound particles. This attribute is typical in spray drying, which can result in better rehydration capacity, easier turmeric extract incorporation, and improved encapsulation.³³⁻³⁴ The addition of turmeric did not influence

the morphology of the products formulated via spray drying.

In contrast, CWPC-FMD exhibited broken, irregularly shaped, and separated particles, which are detrimental to both the rehydration process and the encapsulation of the selected bioactive compound.³⁵ This CWPC-FMD phenomenon, characteristic of the drying process used, is more prominent in the powder's physical structure. This occurs because the foam mat dryer does not have the atomizer, which is responsible for the formation of spherical particles in the spray dryer.^{11,36-37} In summary, the modification of the drying method exerted an important influence on the morphology of the analyzed powders.

From the data obtained, it can be inferred that there was a statistical difference between all analyzed samples in relation to the analyzed parameters of $<1 \mu\text{m}$ and d_{90} . The WPC-SD showed 0% of its particles in the region of $<1 \mu\text{m}$, whereas the turmeric extract exhibited 8.20% of its particles in the region of $<1 \mu\text{m}$. Furthermore, the addition of turmeric shifted the particle size distribution curve, resulting in a positive effect by increasing the number of particles in the $<1 \mu\text{m}$ region from 0% in WPC-SD to 2.14% in CWPC-SD. Finally, comparing the different types of drying, drying using a spray dryer (CWPC-SD) allowed for a greater number of particles to be in the region below $1 \mu\text{m}$ in comparison with the product formulated using a foam mat dryer (CWPC-FMD). More specific analyses need to be performed, but the CWPC-SD findings indicate good encapsulation capacity as curcumin has lipophilic characteristics. If the turmeric extract was not properly encapsulated in WPC, the particle would have behaved like a "supernatant," reducing the powder's rehydration capacity (wettability, shrinkage, dispersibility, and solubility).³⁸⁻³⁹

The d_{90} data correspond to 90% of the particles having values equal to or less than the result obtained. It is known that the higher the d_{90} value, the lower is the powder reconstitution efficiency.³⁶⁻³⁷ The highest d_{90} value found in the present work was obtained for the CWPC-FMD ($170.44 \pm 12.69 \mu\text{m}$) sample, followed by the TE-FD ($97.69 \pm 4.11 \mu\text{m}$). Considering this, it can be inferred that this product exhibited the worst rehydration capacity. Conversely, the lowest values of d_{90} were found in the products formulated using a spray dryer ($65.55 \pm 11.65 \mu\text{m}$ WPC-SD and $36.73 \pm 2.14 \mu\text{m}$ CWPC-SD). Among the two samples, CWPC-SD presented the lowest d_{90} , making it the powder with the best rehydration efficiency. This result demonstrates that the addition of turmeric resulted in a decrease in the d_{90} value of the powder, facilitating the rehydration process, which is a demonstrated benefit resulting from the addition of this extract.

Notably, when comparing the product formulated using the spray dryer and foam mat dryer, there was a reduction of 4.64 times for the d_{90} value. This allows us to conclude that the modification of the drying process exerted a positive influence on this evaluated parameter, leading to a significant

lowering of d_{90} with a consequent improvement in the powder rehydration capacity.

Whey protein concentrate was evaluated in relation to the d_{90} parameter by Jambrak *et al.*⁴⁰; the authors found values between 1.04 and $324.08 \mu\text{m}$. Onwulata *et al.*⁴¹ analyzed the particle size of commercial samples of whey protein concentrate and obtained results between 53 and $382 \mu\text{m}$. Notably, the particle size distribution values can vary significantly among the different types of powders due to the different dryers used.

3.3. Raman spectroscopy

Figure 4 displays the results of Raman spectroscopy.

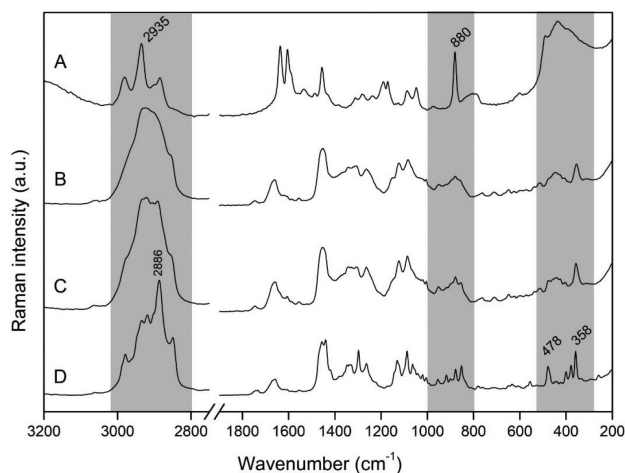


Figure 4. Raman spectroscopy: (A) Turmeric extract freeze-dried (TE-FD); (B) whey protein concentrate (WPC-SD); (C) whey protein added to turmeric extract produced by spray drying (CWPC-SD); (D) whey protein added to turmeric extract produced by foam mat drying (CWPC-FMD).

Peaks in the 2986 cm^{-1} and 2935 cm^{-1} regions were found for all samples (TE-FD, WPC-SD, CWPC-SD, and CWPC-FMD). These can be attributed to Raman scattering bands characteristic of C–H stretch vibrations.²⁰ In addition, a peak was observed in the region of 880 cm^{-1} for the turmeric extract sample. Generally, bands between 880 cm^{-1} and 710 cm^{-1} can be attributed to different aromatic and skeletal movements outside of the COH plane (which may explain the 880 cm^{-1} peak in the CWPC spectrum).⁴²

The CWPC-SD spectrum (Figure 4-C) differed from the WPC-SD spectrum (Figure 4-B), which can be attributed to an overlap of C–H groups with the turmeric extract. Moreover, data showed that lactose remains in an amorphous state even after spray drying (Figure 4-C). Lactose in an amorphous state helps encapsulate the turmeric extract and preserve its properties. This could in turn protect the curcumin and prolong its shelf life. In contrast, the loss of the amorphous structure observed in the CWPC-FMD analysis resulted in lactose crystallization and in an unstable powder that compromised the powder's shelf life. Both of these factors are often associated with foam mat drying.⁴³

Table 2. Concentration and percent loss of total phenolic compounds, curcumin, and antioxidant capacity (on wet matter) of whey protein added to turmeric extract produced via foam mat drying and spray drying in comparison with the control sample

Parameters	Control sample	Samples			
		CWPC-SD	% Losses	CWPC-FMD	% Losses
Curcumin quantification (mg.100 g ⁻¹ sample)	1.45 ± 0.03 ^b	1.10 ± 0.22 ^a	24.13	0.87 ± 0.12 ^a	40.00
Total phenolics (mg GAE.100 g ⁻¹ sample)	7.00 ± 0.00 ^b	4.00 ± 0.01 ^a	57.14	2.00 ± 0.00 ^a	71.43
Antioxidant capacity (mmol Trolox.100 g ⁻¹ sample)	6 ± 0 ^b	182 ± 0 ^a	-	123 ± 0 ^a	-

*Data expressed as mean ± standard deviation (n=3). **Data expressed as wet basis. ***Different superscript letters within the same line indicate significant differences by the Dunnett test (p<0.05); where the control sample is the liquid whey with turmeric extract and pineapple flavoring added in equal amounts to the CWPC samples; CWPC-SD is whey protein added to turmeric extract produced by spray drying; CWPC-FMD is whey protein added to turmeric extract produced by foam mat drying; and GAE are the equivalents of gallic acid.

Thus, CWPC-SD preserved the amorphous state of lactose, which indicates better curcumin encapsulation compared with the powder obtained by foam mat drying.

3.4. Curcumin quantification, total phenolic compound, and antioxidant capacity

Table 2 shows the results found for the analyzed samples (control sample, CWPC-SD, and CWPC-FMD) for the quantification of curcumin, total phenolic compounds, and antioxidant capacity.

Reduced curcumin contents were observed in CWPC-SD and CWPC-FMD in comparison with the control sample. This also led to a total phenolic reduction because curcumin is a phenolic compound. Notably, there was a higher retention of curcumin in the products dried by spray dryer. The total phenolic losses were 57.14% (CWPC-SD) and 71.43% (CWPC-FMD) (Table 2). In study of Richter *et al.*,⁴⁴ the phenolic content in lychees decreased when the vacuum drying temperature increased. In another study Djendoubi *et al.*⁴⁵ the influence of drying temperature on the total phenolic concentration in dried pears was evaluated and a phenolic reduction was also observed.

The phenolic compound antioxidant characteristics make them susceptible to oxidation. Oxidation is influenced by the presence of oxygen, light, and heat, which explain the losses recorded.⁴⁶

However, an increase in antioxidant capacity for both CWPC samples (CWPC-SD: 1.82 mmol Trolox.100 g⁻¹ and CWPC-FMD: 1.23 mmol Trolox/100 g) was observed in comparison with the control sample (0.06 ± 0.00 mmol trolox.100 g⁻¹ sample). Moreover, a previous study on the effects of drying Tommy mango in a foam mat dryer using different experimental parameters (drying temperature and stabilizing agents), showed increased antioxidant capacity for the treated samples compared to the control sample.⁴⁷ This increase can be attributed to the formation of new antioxidant compounds and improvement of the antioxidant capacity of naturally occurring compounds.⁴⁸

Analyzing the results from Table 2, a higher concentration of total phenolics and curcumin, and an increase in antioxidant capacity in CWPC-SD compared to

CWPC-FMD were also observed. This result demonstrates that spray drying is less aggressive and has a lesser effect on the nutritional characteristics of the final powder compared to foam mat drying. In addition, it is worth mentioning that both SD and FMD aided curcumin encapsulation since the antioxidant capacities for both were higher than that of the control sample.

4. Conclusion

The properties of the air obtained during drying showed higher evaporative capacity, less thermal damage, and shorter residence time in the equipment for the product formulated by the spray drying process in comparison with the product formulated via foam mat drying. The spray drying technique was more efficient than the foam mat dryer drying technique in terms of solubility, loss of phenolics/curcumin and antioxidant capacity. This is because, the product formed via spray drying exhibited greater solubility, lower loss of phenolics and curcumin, and greater antioxidant capacity.

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References

- Jyotirmayee, B.; Mahalik, G.; Traditional Uses and Variation in Curcumin Content in Varieties of Curcuma-the Saffron of India. *Ambient Science* **2022**, *09*, 1. [[Crossref](#)]
- Mohajeri, M.; Behnam, B.; Cicero, A. F.; Sahebkar, A.; Protective effects of curcumin against aflatoxicosis: A comprehensive

- review. *Journal of Cellular Physiology* **2018**, 233, 4. [[Crossref](#)] [[PubMed](#)]
3. Akter, J.; Hossain, M. A.; Takara, K.; Islam, M. Z.; Hou, D. X.; Antioxidant activity of different species and varieties of turmeric (*Curcuma* spp): Isolation of active compounds. *Comparative Biochemistry and Physiology Part C: Toxicology & Pharmacology* **2019**, 215. [[Crossref](#)] [[PubMed](#)]
 4. Bordoloi, D.; Kunnumakkara, A. B.; In *Role of Nutraceuticals in Cancer Chemosensitization*. Elsevier: Amsterdã, 2018, cap. 2. [[Crossref](#)]
 5. Dar, O. A.; Malik, M. A.; Shahid-ul-Islam, P. G.; Hashmi, A. A.; In *Plant-Based Natural Products: Derivatives and Applications (edited by Shahid Ul-Islam)*. John Wiley & Sons: Nova Jersey, EUA, 2017, cap. 8. [[Crossref](#)]
 6. Hatcher, H.; Planalp, R.; Cho, J.; Torti, F.; Torti, S.; Curcumin: from ancient medicine to current clinical trials. *Cellular and Molecular Life Sciences* **2008**, 65, 11. [[Crossref](#)] [[PubMed](#)]
 7. Hu, Y.; Kou, G.; Chen, Q.; Li, Y.; Zhou, Z.; Protection and delivery of mandarin (*Citrus reticulata* Blanco) peel extracts by encapsulation of whey protein concentrate nanoparticles. *LWT - Food Science and Technology* **2019**, 99. [[Crossref](#)]
 8. Nishanthi, M.; Chandrapala, J.; Vasiljevic, T.; Impact of storage conditions on solubility, heat stability and emulsifying properties of selected spray dried whey protein concentrates. *LWT- Food Science and Technology* **2018**, 92. [[Crossref](#)]
 9. Bergia, R. E.; Hudson, J. L.; Campbell, W. W.; Effect of whey protein supplementation on body composition changes in women: A systematic review and meta-analysis. *Nutrition Reviews* **2018**, 76, 7. [[Crossref](#)] [[PubMed](#)]
 10. Naidu, S. T.; O uso de whey protein no manejo da síndrome consumptiva em pacientes HIV+. *International Journal of Nutrology* **2018**, 11, 02. [[Crossref](#)]
 11. Gomes, J. V. P.; de Oliveira, L. A.; Francisquini, J. D. A.; Anunciação, P. C.; Stephani, R.; de Oliveira, L. F. C.; Perrone, I. T.; Della Lucia, C. M.; Morphological characterization of whey protein concentrate admixture of microencapsulated curcumin by spray drying. *Journal of Food Processing and Preservation* **2020**, 45, 2. [[Crossref](#)]
 12. Chaux, G. A. M.; Santos, A. B.; Granda, R. D. M.; Mauro, M. A.; Foam mat drying of mango: Effect of processing parameters on the drying kinetic and product quality. *Drying Technology* **2017**, 35, 5. [[Crossref](#)]
 13. Hardy, Z.; Jideani, V. A.; Foam-mat drying technology: A review. *Critical Reviews in Food Science and Nutrition* **2017**, 57, 12. [[Crossref](#)] [[PubMed](#)]
 14. Salahi, M. R.; Mohebbi, M.; Taghizadeh, M.; Development of cantaloupe (*Cucumis melo*) pulp powder using foam-mat drying method: Effects of drying conditions on microstructural of mat and physicochemical properties of powder. *Drying Technology* **2017**, 35, 15. [[Crossref](#)]
 15. Schuck, P.; Jeantet, R.; Bhandari, B.; Chen, X. D.; Perrone, Í. T.; de Carvalho, A. F.; Fenelon, M.; Kelly, P. Recent advances in spray drying relevant to the dairy industry: A comprehensive critical review. *Drying Technology* **2016**, 34, 15. [[Crossref](#)]
 16. de Paula, R. R.; Dissertação de Mestrado, Universidade Federal do Espírito Santo, 2015. [[Link](#)]
 17. World Health Organization, Evaluation of certain food additives Geneva, 2004. [[Link](#)]
 18. Perrone, I. T.; Simeao, M.; Rodrigues Junior, P. H.; Stephani, R.; de Carvalho, A. F.; Influence of pilot spray dryer settings on moisture and water activity of whole milk powder. *Journal of Candido Tostes Dairy Institute* **2013**, 68, 393. [[Crossref](#)]
 19. Schuck, P.; Mejean, S.; Dolivet, A.; Jeantet, R. Thermohygro-metric sensor: A tool for optimizing the spray drying process. *Innovative Food Science & Emerging Technologies* **2005**, 6, 1. [[Crossref](#)]
 20. Almeida, M. R.; Oliveira, K. D. S.; Stephani, R.; de Oliveira, L. F. C.; Fourier-transform Raman analysis of milk powder: a potential method for rapid quality screening. *Journal of Raman Spectroscopy* **2011**, 42, 7. [[Crossref](#)]
 21. Bloor, S. J.; Overview of methods for analysis and identification of flavonoids. *Methods Enzymology* **2001**, 335. [[Crossref](#)] [[PubMed](#)]
 22. Singleton, V. L.; Orthofer, R.; Lamuela, R. R. M. Analysis of total phenols and other oxidation substrates and antioxidants by means of folin-ciocalteu reagent. *Methods in Enzymology* **1999**, 299. [[Crossref](#)] [[PubMed](#)]
 23. Fialho, T. L.; Martins, E.; Silveira, A. C. P.; Silva, C. R. d. J.; Perrone, Í. T.; Schuck, P.; de Carvalho, A. F. Lactose hydrolyzed milk powder: Thermodynamic characterization of the drying process. *Drying Technology* **2018**, 36, 8. [[Crossref](#)]
 24. Silva, C. R. D.; Martins, E.; Silveira, A. C. P.; Simeão, M.; Mendes, A. L.; Perrone, Í. T.; Schuck, P.; de Carvalho, A. F.; Thermodynamic characterization of single-stage spray dryers: Mass and energy balances for milk drying. *Drying Technology* **2017**, 35, 15. [[Crossref](#)]
 25. Li, R.; Lin, D.; Roos, Y. H.; Miao, S.; Glass transition, structural relaxation and stability of spray-dried amorphous food solids: A review. *Drying Technology* **2019**, 37, 3. [[Crossref](#)]
 26. Zouari, A.; Perrone, Í. T.; Schuck, P.; Gaucheron, F.; Dolivet, A.; Attia, H.; Ayadi, M. A.; Effect of outlet drying temperature and milk fat content on the physicochemical characteristics of spray-dried camel milk powder. *Drying Technology* **2019**, 37, 13. [[Crossref](#)]
 27. Schuck, P.; Jeantet, R.; Dolivet, A. Analytical Methods for Food and Dairy Powders. John Wiley & Sons: Nova Jersey, 1a. ed., 2012. [[Crossref](#)]
 28. Chandralekha, A.; Rani, A.; Tavanandi, H. A.; Amrutha, N.; Hebbar, U.; Raghavarao, K. S. M. S.; Role of carrier material in encapsulation of yeast (*Saccharomyces cerevisiae*) by spray drying. *Drying Technology* **2017**, 35, 8. [[Crossref](#)]
 29. Drosou, C. G.; Krokida, M. K.; Biliaderis, C. G.; Encapsulation of bioactive compounds through electrospinning/electrospraying and spray drying: A comparative assessment of food-related applications. *Drying Technology* **2017**, 35, 2. [[Crossref](#)]
 30. Schmitz S. I.; Foerst, P.; Kulozik, U.; Impact of the spray drying conditions and residence time distribution on lysine loss in spray dried infant formula. *Dairy Science & Technology* **2013**, 93. [[Crossref](#)]
 31. Schuck, P.; Jeantet, R.; Tanguy, G.; Méjean, S.; Gac, A.; Lefebvre, T.; Labussière E.; Martineau, C.; Energy consumption in the processing of dairy and feed powders by evaporation and drying. *Drying Technology* **2015**, 33, 2. [[Crossref](#)]

32. Thorat, A. A.; Dalvi, S. V.; Particle formation pathways and polymorphism of curcumin induced by ultrasound and additives during liquid antisolvent precipitation. *CrystEngComm* **2014**, *16*, 48. [[Crossref](#)]
33. Alves, A. I.; Rodrigues, M. Z.; Ribeiro Pinto, M. R. M.; Lago Vanzela, E. S.; Stringheta, P. C.; Perrone, Í. T.; Ramos, A. M.; Morphological characterization of pequi extract microencapsulated through spray drying. *International Journal of Food Properties* **2017**, *20*, 2. [[Crossref](#)]
34. Beran, M.; Drahorad, J.; Vltavsky, O.; Urban, M.; Laknerova, I.; Fronek, M.; Sova, J.; Ondracek, J.; Kralova, M.; Formankovas; Pilot-scale production and application of microparticulated plant proteins. *Journal of Nutrition and Food Sciences* **2018**, *8*, 1. [[Crossref](#)]
35. Franco, T. S.; Perussello, C. A.; Ellendersen, L. N.; Masson, M. L.; Effects of foam mat drying on physicochemical and microstructural properties of yacon juice powder. *LWT - Food Science and Technology* **2016**, *66*. [[Crossref](#)]
36. Queiroz, E. S.; Rezende, A. L. L.; Perrone, Í. T.; Francisquini, J. D. A.; de Carvalho, A. F.; Alves, N. M. G.; de Oliveira, L. F. C.; Stephani, R.; Spray drying and characterization of lactose-free goat milk. *LWT - Food Science and Technology* **2021**, *147*. [[Crossref](#)]
37. de Paula, I. L.; Teixeira, E. B. S.; Francisquini, J. D. A.; Stephani, R.; Perrone, Í. T.; de Carvalho, A. F.; de Oliveira, L. F. C.; Buffalo powder dairy products with and without lactose hydrolysis: Physical-chemical and technical-functional characterizations. *LWT - Food Science and Technology* **2021**, *151*, 112124. [[Crossref](#)]
38. Richard, B.; Le Page, J.-F.; Schuck, P.; André, C.; Jeantet, R.; Delaplace, G.; Towards a better control of dairy powder rehydration processes. *International Dairy Journal* **2013**, *31*, 1. [[Crossref](#)]
39. Shehzad, A.; Qureshi, M.; Anwar, M. N.; Lee, Y. S.; Multifunctional curcumin mediate multitherapeutic effects. *Journal of Food Science* **2017**, *82*, 9. [[Crossref](#)] [[PubMed](#)]
40. Jambrak, A. R.; Mason, T. J.; Lelas, V.; Paniwnyk, L.; Hecceg, Z.; Effect of ultrasound treatment on particle size and molecular weight of whey proteins. *Journal of Food Engineering* **2014**, *121*. [[Crossref](#)]
41. Onwulata, C. I.; Konstance, R. P.; Tomasula, P. M. Minimizing variations in functionality of whey protein concentrates from different sources. *Journal of Dairy Science* **2004**, *87*, 3. [[Crossref](#)] [[PubMed](#)]
42. Kolev, T. M.; Velcheva, E. A.; Stamboliyska, B. A.; Spitteller, M.; DFT and experimental studies of the structure and vibrational spectra of curcumin. *International Journal of Quantum Chemistry* **2005**, *102*. [[Crossref](#)]
43. Stephani, R.; Oliveira, K. D. S.; de Almeida, C. E. R.; Perrone, Í. T.; de Carvalho, A. F.; de Oliveira, L. F. C.; Almeida, M. R. Raman spectroscopy as a tool to identify modification of whey protein concentrate (WPC) during shelf life. *Food Packaging and Shelf Life* **2017**, *11*. [[Crossref](#)]
44. Richter R. F.; de Oliveira, A. C.; Gadelha, G. G. P.; de Abreu, M. B.; Soares, H. I.; Vacuum drying for extending litchi shelf-life: Vitamin C, total phenolics, texture and shelf-life assessment. *Plant Foods for Human Nutrition* **2017**, *72*, 2. [[Crossref](#)] [[PubMed](#)]
45. Djendoubi M. N.; Boudhrioua, N.; Kechaou, N.; Courtois, F.; Bonazzi, C.; Influence of air drying temperature on kinetics, physicochemical properties, total phenolic content and ascorbic acid of pears. *Food and Bioproducts Processing* **2012**, *90*, 3. [[Crossref](#)]
46. Davey, M. W.; Montagu, M. V.; Inzé, D.; Sanmartin, M.; Kanellis, A.; Smirnoff, N.; Benzie, J. J. I.; Strain, J. J.; Favell D.; Fletcher, J.; Plant L-ascorbic acid: chemistry, function, metabolism, bioavailability and effects of processing. *Journal of the Science of Food and Agriculture* **2000**, *80*, 7. [[Crossref](#)]
47. Lobo, F. A.; Nascimento, M. A.; Domingues, J. R.; Falcão, D. Q.; Hernanz, D.; Heredia, F. J.; de Lima Araujo, K. G.; Foam mat drying of Tommy Atkins mango: Effects of air temperature and concentrations of soy lecithin and carboxymethylcellulose on phenolic composition, mangiferin, and antioxidant capacity. *Food Chemistry* **2017**, *221*. [[Crossref](#)]
48. Turkmen, N.; Sari, F.; Velioglu, Y. S.; The effect of cooking methods on total phenolics and antioxidant activity of selected green vegetables. *Food Chemistry* **2005**, *93*, 4. [[CrossRef](#)]