https://doi.org/10.17113/ftb.64.03.26.9455 scientific note

# Comparative Analysis of Drying and Preservation Techniques in Whey Powder: Enhancing Physicochemical and Functional Properties

Running title: Comparative Analysis of Drying and Preservation Techniques in Whey Powder

Seçil Dede[1] and Sebnem Ozturkoglu-Budak[2]*

[1]Graduate School of Natural and Applied Sciences, Ankara University, 06110 Dışkapı, Altındağ, Ankara, Turkiye
[2]Department of Dairy Technology, Faculty of Agriculture, Ankara University, 06110 Dışkapı, Altındağ, Ankara, Turkiye

Received: 31 October 2025
Accepted: 24 July 2026



## SUMMARY

*Research background.* Whey is commonly used in powder form in various dairy and food products, including whey-based beverages, yogurts, sports supplements, and bakery items such as cakes and waffles. As a by-product of cheese production, incorporating whey into foods supports both economic and environmental sustainability while enhancing their nutritional and functional qualities, primarily due to its high serum protein content. This study investigates the effects of different technologies, such as spray-drying, freeze-drying, and encapsulation combined with freeze-drying,

*Corresponding author:
E-mail: budak@ankara.edu.tr

on the functional and nutritional characteristics of whey. The primary objective was to identify the most effective processing method for preserving the physicochemical and functional integrity of whey.

*Experimental approach.* Comprehensive analyses were conducted on individually processed and reconstituted whey samples, including the assessment of pH, titratable acidity, dry matter, protein, ash, and fat contents, as well as water-holding capacity, rheological behavior, color attributes, antioxidant activity, and total phenolic content. By comparing the analysis results, the most suitable application was identified based on technological, sensory, and functional criteria.

*Results and conclusions.* Encapsulation combined with freeze-drying was the most effective method, significantly enhancing functional properties. This treatment resulted in the highest antioxidant capacity (1.62 mM Trolox) and water-holding capacity (4.65 %), while largely preserving phenolic compounds (1.09 mg GAE/mL) and protein content (0.82 %). It also produced the highest consistency index (0.448), indicating improved structural stability. Freeze-drying alone effectively maintained phenolic content (1.13 mg GAE/mL), color (*L**=46.95), and nutritional quality, while improving water-holding capacity (3.91 %) compared to untreated whey. In contrast, spray-drying, despite its industrial advantages, resulted in notable reductions in phenolic content (0.82 mg GAE/mL) and antioxidant capacity (0.020 mM Trolox), along with a darker color (*L**=39.5), likely due to thermal degradation. All drying methods improved dry matter content, particularly in encapsulated samples (16.33 %). Overall, encapsulation combined with freeze-drying provided the best balance of functional, nutritional, and physicochemical properties, making it a promising approach for enhancing whey stability and shelf life.

*Novelty and scientific contribution.* These findings highlight the potential of alternative drying and preservation technologies, particularly encapsulation combined with freeze drying, for the sustainable valorization of whey in functional food applications.

**Key words:** whey; powder; encapsulation; freeze-drying; spray-drying

## INTRODUCTION

Whey is the liquid fraction separated from the curd during cheese production. Annually, a substantial amount of whey is generated as a by-product, estimated at approximately 121 million tons [*1*]. However, fluctuations in global supply lead to a significant portion of whey remaining unutilized, contributing to environmental pollution. Whey retains more than 50 % of the total nutrients present in milk [*2*], and due to its high organic content, improper disposal as wastewater poses a serious environmental threat [*3*]. Despite these environmental challenges, whey offers considerable nutritional benefits due to its high amount of serum proteins, lactose, vitamins, and various minerals [*4*]. Therefore, it is essential to explore alternative methods to convert whey into value-added products.

Powdered dairy products have gained increasing popularity worldwide due to their convenience, long shelf life, and safety. They are primarily used in manufacturing sectors such as confectionery, infant formula, enteral nutrition formulas, and bakery goods, and are extensively produced for export. Their stable characteristics make them well-suited for international trade. U.S. dairy export values rose by 25 %, driven by strong growth in key categories like skim milk powder, followed by significant contributions from the EU, New Zealand, Australia, and Belarus. The demand for various whey-based ingredients continues to grow, according to the USDA [*5*].

Whey powder can be produced depending on the drying technologies, such as spray-drying and freeze-drying. Freeze-drying helps preserve the physical and chemical properties of the product while ensuring microbial stability. Thus, it gained attention as an alternative drying technology providing improved flavor, aroma, and structure retention characteristics.

Powdered form is the favored commercial form due to its long shelf life and ease of handling. Whey drying technologies such as spray-drying, freeze-drying, and freeze-drying combined with encapsulation are widely used to improve the stability, handling, and functional applicability of whey-based ingredients. These techniques are based on different physicochemical principles that influence moisture removal, particle formation, and structural integrity of bioactive components. The most widely used method for producing dairy powder is atmospheric spray-drying (ASD), which rapidly evaporates water using high temperature and dry air. This method offers the advantage of a short processing time; however, it can also cause the degradation of bioactive compounds that are sensitive to heat [*6*]. Freeze drying (FD) is an alternative drying technique currently in use, which removes moisture through the process of sublimation. FD can preserve bioactive components because it operates at low temperatures (around −80 °C). However, this method is time-consuming and costly [*7*]. Therefore, a comparative evaluation of these drying approaches is essential to understand how processing conditions govern final product performance and potential food applications.

Encapsulation protects sensitive compounds from environmental factors and allows for controlled release. It is an effective method used to improve the stability of bioactive compounds by enclosing the core substance within a protective barrier, commonly known as a wall material. Spray-drying and freeze-drying are the most widely used methods for producing dry encapsulated powders

[*8*]. Freeze-drying is particularly suitable for preserving heat-sensitive compounds, as it reduces the risk of degradation caused by high temperatures. However, this method involves high energy consumption and long processing times [*9*]. On the other hand, spray-drying is the preferred method in the food industry due to its adaptability, continuous production capabilities, and cost efficiency. In both spray- and freeze-drying, the encapsulation process involves coating whey with a protective layer. In spray drying, this emulsion is sprayed into a heated chamber, where the solvent quickly evaporates, leaving behind dry particles. However, freeze-drying begins with freezing the emulsion and then lowering the pressure so the frozen solvent transitions directly from solid to gas, creating a porous matrix [*10*].

This study aimed to identify the most effective method for preserving the nutritional and functional characteristics of whey. Whey powders obtained via spray-drying, freeze-drying, and encapsulation followed by freeze-drying were compared in terms of antioxidant capacity, total phenolic content, physicochemical properties, including pH, titration acidity (percentage of lactic acid), protein content, and water-holding capacity.

## MATERIALS AND METHODS

### *Materials*

The liquid whey used in this study was sweet whey obtained as a by-product of white cheese production (rennet-coagulated cheese) at the Dairy Plant of Ankara University (Ankara, Turkey). Liquid cheese whey is a greenish-yellow, nutrient-rich aqueous byproduct of cheesemaking, comprising 80–90 % of the total milk volume. It is composed of ~94 % water, lactose, soluble proteins (lactalbumin/lactoglobulin), and minerals. Whey samples obtained from three separate cheese productions conducted at one-week intervals were collected and subjected to processing and analysis. The applied processes included spray-drying, freeze-drying, and encapsulation followed by freeze-drying to produce powdered cheese whey. Powdered cheese whey is a dried byproduct of cheesemaking created by removing water from liquid whey using the technologies of spray-drying or freeze-drying. All processing and measurements were performed in triplicate.

### *Spray-dried whey powder*

The commercially available whey powder produced through atmospheric spray-drying was obtained from Enka (Konya, Turkiye). A rotary atomizer operating at 12000 rpm (GEA Niro Atomizer, GEA Process Engineering A/S, Søborg, Denmark) was employed for the spray-drying of the samples.

The process parameters were set as follows: feed temperature of 63 °C, inlet air temperature of 180 °C, and outlet air temperature of 70 °C. The air flow rate was maintained at 30,000 kg/h, with a drying duration ranging between 40 and 50 s. Whey powders were obtained from 3 consecutive batches to ensure reproducibility.

*Freeze-drying process*

The whey samples were frozen at −80 °C and then freeze-dried for 24 h under a vacuum pressure of 0.5 mbar and at −58 °C (Christ, Alpha 1-2 LDplus, Osterode am Harz, Germany). The dried samples were ground and stored at −20°C until reconstitution.

*Encapsulation process*

The freeze-dried samples were also used for the encapsulation treatments. A mixture of 8 % maltodextrin (MD) and 2 % gum arabic (GA) (Sigma-Aldrich, St. Louis, USA) was used as the coating material. The MD solution was prepared by mixing at 200 rpm for 24 h at 28 °C, while GA was mixed at 200 rpm for 2 h at 30 °C [*11*]. A 1:10 ratio of core material and coating material (1 g dry (powder) whey and 10 g coating material) was used. Encapsulation was carried out for 5 min at 100 rpm using an UltraTurrax homogenizer (Heidolph, DIAX 900 Ultraturrax, Schwabach, Germany). Dried capsuls were obtained after a second freeze-drying process for 48 h at the same conditions.

*Reconstitution process*

Whey powders obtained by spray-drying, freeze-drying, and encapsulation followed by freeze-drying were reconstituted with sterile distilled water to obtain solutions containing 10 % (*m/V*) total solids. This standardization was applied to ensure a comparable testing matrix across all treatments, based on the solid-not-fat (SNF) content of the bovine milk from which each whey sample was derived.

*Physicochemical analyses*

Physicochemical properties of untreated whey and reconstituted whey powder samples were analysed according to the methods given below. The pH was measured by direct insertion of a pH meter (Starter300, Ohaus, PR China). Titratable acidity (expressed as % lactic acid) was determined according to the AOAC method [*12*]. Dry matter content was analyzed using the gravimetric method at 102 °C [*12*]. Fat content and total nitrogen (TN) content were determined using the Gerber–Van

Gulik method and the Kjeldahl method, respectively [*12*]. The nitrogen content was converted to protein content by applying a conversion factor of 6.38. Ash content was obtained by incineration of the sample placed in a muffle furnace at 550 °C for 6 h. The water-holding capacity (WHC) of the samples (25 g) was determined using the centrifugation method described by Isanga and Zhang [*13*]. Samples were centrifuged at 8000×*g* for 15 min at 4 °C using a Sigma 3–18K centrifuge (Sartorius AG, Göttingen, Germany). WHC (in %) was calculated using the following equation:

$$\mathrm{WHC} = (1 - \frac{m1}{m2}) \cdot\ 100 \qquad /1/$$

where $m_1$ is the mass of the supernatant obtained after centrifugation, and $m_2$ is the initial mass of the sample.

*Rheology analyses*

Rheology analyses were performed using a Kinexus Pro+ rheometer (Malvern Panalytical, Malvern, UK) with a 40 mm stainless steel 4º conical geometry spindle and a 1 mm gap. The temperature of the frequency sweep measurements was set at 5 °C. The shear rate was set in the range of 0.1–300 $s^{-1}$. The flow curves were fitted to the power law model by using a non-linear regression. The power law model equation was used:

$$\sigma=K\cdot(\gamma)^n \qquad /2/$$

where $\sigma$ is the shear stress (Pa), $K$ is the consistency index (Pa·$s^n$), $\dot{\gamma}$ is the shear rate ($s^{-1}$), and $n$ is the flow behavior index (dimensionless).

*Color determination*

Color measurements were performed using a portable color spectrophotometer (Ser-Lab, SL400, Turkiye) based on the CIE *L*a*b** space value. Following calibration with white and black backgrounds, samples were individually placed into the container, and color measurements were recorded for *L** (lightness), *a** (red-green), and *b** (yellow-blue) values.

*Functional properties*

Determination of total phenolic content

Total phenolic content determination of whey and reconstituted whey samples was performed according to the Folin-Ciocalteu method. A sample volume of 5 mL was mixed with 25 mL of ethanol:acetic acid:water (50:42:8) mixture (Sigma-Aldrich, St. Louis, USA) and centrifuged at 8000 rpm for 15 min. The clear supernatant obtained was used as the extracted sample for total phenolic analysis. During the analyses, 100 μL of the extracted sample, 500 μL of Folin-Ciocalteu reagent (Merck KGaA, Darmstadt, Germany), 1.5 mL of 20 % $Na_2CO_3$, and 7.9 mL of distilled water were mixed in a test tube. The mixtures were vortexed and kept in a dark place at room temperature for an incubation period of 1 h. After this duration, the absorbances were read in triplicate at 760 nm in a UV-Vis spectrophotometer (PerkinElmer, Lambda 25, Waltham, USA). Calculations were done using a gallic acid standard curve. For this purpose, 5 mg/mL gallic acid (Sigma-Aldrich, St. Louis, USA) was prepared as the main stock solution, and working solutions were prepared at concentrations of 0.05–4 mg/mL. The values were expressed in mg GAE/mL ($R^2$=0.97).

Determination of antioxidant activity

The antioxidation activities of whey and reconstituted whey samples were determined by the 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical scavenging method. 100 μL of the extracted sample was added to 2 mL of DPPH solution (0.2 mM DPPH in 95 % methanol; Sigma-Aldrich, St. Louis, USA)) and kept in the dark for 30 min at 4 °C. At the end of the incubation, the absorbances were determined at 517 nm by a UV/VIS spectrophotometer (PerkinElmer, Lambda 25, Waltham, USA). Distilled water was used as a blank. The inhibition percentage was estimated using the following equation:

$$\text{Inhibition} = \left(\frac{A\ \text{blank} - A\ \text{sample}}{A\ \text{blank}}\right) \cdot 100 \qquad /3/$$

where $A_{\text{blank}}$ is the absorbance of the DPPH solution with water instead of the sample, and $A_{\text{sample}}$ is the absorbance of the DPPH solution with the sample. Each value was compared with Trolox standards prepared at concentrations of 0.05–5.00 mM. The measured absorbances were converted to μM Trolox equivalent antioxidant capacity (TEAC).

DPPH radical scavenging activity, representing antioxidant activity, was expressed as mM Trolox equivalent antioxidant capacity (TEAC) using a Trolox standard curve prepared with concentrations ranging from 0.05 to 2.50 mM ($R^2$=0.99).

*Statistical analyses*

Statistical analyses were carried out using the Minitab package program v. 17 (State College, USA). Analysis of variance (ANOVA) was performed to determine statistical differences among samples ($p<0.05$). Tukey's post-hoc multiple comparison test was applied to determine which specific treatment caused the differences.

## RESULTS AND DISCUSSION

*pH value and physicochemical properties*

The pH values and other chemical properties of all whey samples are shown in Table 1. The pH values were found to be at similar levels across all treatment groups (6.49–6.64), with no statistically significant differences observed ($p>0.05$). This result indicates that the different drying methods did not alter the buffering capacity of whey and that pH remained stable. However, titratable acidity was significantly higher in the encapsulated samples (0.16 %) ($p<0.05$). This increase can be attributed to the acidic contributions of coating materials such as maltodextrin and gum arabic, as well as the more controlled release of organic acids within the microencapsulated structures [*14*]. Encapsulation has been shown to effectively preserve the acidic structure of whey and may even increase the concentration of its acidic components [*15*]. In contrast, spray-dried samples exhibited low acidity (0.09 %), comparable to untreated whey, indicating organic acid loss due to heat exposure during drying.

Although all samples were reconstituted to a standardized 10 % (*m*/*V*) total solids level, the measured dry matter content varied among treatments due to differences in powder composition and matrix structure. Freeze-dried and spray-dried whey powders showed lower dry matter values (8.32 and 8.72 %, respectively), which may be attributed to their higher solubility and more complete rehydration, resulting in a more homogeneous dispersion of solids in the aqueous phase. In contrast, the encapsulation-assisted freeze-dried sample exhibited significantly higher dry matter content (16.33 %), which can be explained by the presence of carrier materials used during encapsulation. These carriers increase the total solid fraction and may not fully dissolve or disperse during reconstitution, thereby contributing to higher measured dry matter values. Therefore, the observed differences reflect variations in powder composition and rehydration behavior rather than inconsistencies in the reconstitution protocol. Dry matter content also correlated positively with water-holding capacity.

The protein content was highest in the untreated whey (0.87 %), but decreased to as low as 0.76 % in the spray-dried samples. This reduction is associated with protein denaturation caused by high temperatures, which affects the secondary and tertiary structures of proteins. Freeze-drying and encapsulation, being carried out at lower temperatures or in protective environments, resulted in more limited protein loss [*16*]. Ghanimah *et al*. [*17*] also reported that higher protein levels cause increased functionality. Farahmandpour *et al.* [*18*] investigated the effects of whey protein supplementation on inflammatory markers and oxidative stress, highlighting that protein processing methods can influence nutritional value. They reported that proteins processed at lower temperatures retained more functional properties compared to those exposed to higher temperatures. The study also emphasized that encapsulation helps preserve the biochemical integrity of proteins by preventing denaturation. The reduction in protein content also had an indirect effect on the phenolic compound content and antioxidant activity of whey, as protein–phenolic interactions can influence antioxidant stability [*19*].

Ash content was found to be the lowest in the untreated whey (0.43 %), while significantly higher values were observed in all processed samples (0.64–0.67) ($p<0.05$). This increase can be explained by water removal during drying, leading to a concentration of minerals [*20*]. However, a noteworthy point is that despite the increase in ash content, there was a tendency for phenolic compound levels to decrease. This suggests that while thermal processing helps retain mineral content, it may lead to the degradation of heat-sensitive phenolic compounds.

No significant differences were observed among the samples with respect to fat content (0.40 %). These findings indicate that whey fats exhibit stability under the applied thermal and mechanical treatments and are not markedly influenced by the processing method.

Water-holding capacity increased significantly, particularly in the encapsulated samples (4.65 %). This enhancement is attributed to the hydrophilic nature of coating agents such as maltodextrin and gum arabic. Spray-dried whey exhibited a similarly high water-holding capacity (4.60 %); however, this capacity is associated more with the presence of carrier materials (8 % maltodextrin (MD) and 2 % gum arabic (GA)) than with protein content. Freeze-dried whey, on the other hand, demonstrated a lower water-holding capacity (3.91 %). These differences are directly related to variations in dry matter and protein content, indicating that the processing technique substantially influences the microstructure and water-binding capacity of the product. Farahmandpour *et al.* [*18*] also reported that the water retention capacity of whey products subjected to different heat treatments and additives ranged from 3.75 to 4.70 %, which is consistent with the present findings and demonstrates that encapsulation and spray-drying processes enhance the physical properties of whey. It has been determined that encapsulation increases the capacity to bind water molecules.

*Rheological properties*

As shown in Table 2, significant differences were observed among the samples in terms of consistency index (*K*) and flow behavior index (*n*). Encapsulated+ freeze-dried whey exhibited the highest consistency index (0.448 Pa·s), it was followed by spray-dried (0.190 Pa·s) and freeze-dried (0.130 Pa·s) whey, respectively. The untreated sample displayed the lowest value (0.020 Pa·s). Similarly, McEntee *et al.* [*21*] determined strong gelation properties in reconstituted powders (5 % *m*/*m* protein) following heat treatment at 90 °C. The elevated consistency is directly linked to increased dry matter content, enhanced water retention, and the dense nature of the encapsulated matrix. Regarding flow behavior, spray-dried whey showed a flow behavior index value closest to Newtonian behavior (*n*=0.696), while both encapsulated and freeze-dried samples exhibited lower *n* values, indicating pseudoplastic behavior. This suggests that the method of whey processing has a significant effect on the physical properties of the final product. Treatments such as freeze-drying, encapsulation, and spray-drying enhance the consistency of reconstituted whey, thereby influencing its flow and viscosity.

Onay-Ucar *et al.* [*22*] examined the rheological properties of whey and reported that the consistency index (*K*) increased to 0.45 % with encapsulation, compared to 0.025 % in untreated samples. Flow behaviour index (*n*) was determined highest in spray-dried reconstituted whey showing the increased flowability compared to freze-dried and encapsulated+freze dried reconstituted wheys. Similarly, Gausemel *et al.* [*23*] found that flow behaviour index (*n*) of spray dried whey powder processed at low temperatures demonstrated better flowability. In another study, Zhou *et al*. [*24*] evaluated the flowability of spray- and freeze-dried milk powders and reported Spray-dried milk powders generally exhibit superior flowability compared to freeze-dried powders. This difference is primarily due to the formation of small, spherical, and dense particles during spray-drying, which reduces interparticle friction. In contrast, freeze-drying often results in porous, irregular, sheet-like, or larger, more cohesive particles that hinder flow.

*Color properties*

The *L**, *a**, and *b** values obtained from color analysis (Table 3) indicate that processing methods significantly influenced the color profile of whey. Encapsulated whey showed the highest lightness value (*L**=55.7), whereas spray-dried whey exhibited the lowest (*L**=39.5), corresponding to a darker color. These differences can be attributed to Maillard reactions, polyphenol oxidation, and variations in particle size. Furthermore, caramelization and browning reactions likely occurred during spray-drying as a result of the high processing temperatures. These findings suggest that the whey processing method has a significant impact on its color properties. Encapsulation produces a lighter, more yellow product, whereas the commercial form appears darker and more neutral, reflecting the direct effects of additives or processing techniques.

Başyiğit and Karaaslan [*25*] compared the color characteristics of wild mushrooms dried by freeze-drying, vacuum-drying, cabinet-drying, and solar-energy drying. The highest *L** value (58.15) was observed in the freeze-drying method, while the lowest (42.15) occurred in solar-energy drying. These differences were attributed to enzymatic browning resulting from varying oxygen levels. Overall, freeze-drying was identified as the most effective technique for preserving color characteristics.

In spray-drying, discoloration may occur due to high inlet air temperatures or prolonged exposure of the powder to the dryer, which can lower *L** values [*26*]. This effect has also been attributed to the concentration of low-moisture samples at very high temperatures and to Maillard reactions induced by heat. For both drying techniques, the *a** parameter showed low positive values, indicating a reddish hue, while the *b** parameter showed low positive values, indicating a yellow hue. The *a** and *b** values of the lyophilized powder were 7.39 and 13.23, respectively, whereas the spray-dried powder showed values of 6.86 and 16.16, corresponding to lower redness.

*Functional properties*

Phenolic content

As shown in Table 4, the total phenolic content was similar in the untreated and freeze-dried whey samples (1.15 and 1.13 mg GAE/mL, respectively), but decreased significantly in the spray-dried samples (0.82 mg GAE/mL). In contrast, the encapsulated whey exhibited an intermediate phenolic content (1.09 mg GAE/mL). These losses are primarily attributed to degradation caused by thermal processing. Since high temperatures can lead to the degradation of phenolic compounds, freeze-drying at low temperatures preserves most of these compounds [*27*]. Consequently, the phenolic content of freeze-dried whey remained similar to that of untreated whey. Encapsulation, on the other hand, can help preserve active compounds while altering their bioavailability. Studies have shown that although encapsulation can increase the bioavailability of phenolic compounds, it may also prevent some compounds from existing in free form, resulting in a lower measured phenolic content [*28*]. In addition, unlike the other samples, the whey obtained by encapsulation contains a coating material, which likely contributes to the lower measured phenolic content in the analyzed sample. Due to the high temperatures involved, the spray-drying method may cause the degradation of heat-sensitive phenolic compounds, while also facilitating temperature-related Maillard reactions that can further contribute to phenolic compound loss [*29*]. Onay-Ucar *et al.* [*22*] reported the phenolic content of original whey as 1.20 mg GAE/mL and that of its freeze-dried form as 1.15 mg GAE/mL. In spray-dried whey samples, the phenolic content decreased to 0.78 mg GAE/mL. Li *et al.* [*30*] evaluated the interaction between whey protein and polyphenolic compounds and reported that phenolic content could reach up to 1.12 mg GAE/mL using encapsulation. Furthermore, this value was maintained between 1.10 and 1.15 mg GAE/mL when low-temperature lyophilization was applied. In contrast, Farahmandpour *et al.* [*18*] reported that high-temperature treatments led to greater loss of phenolic compounds, reducing the content to 0.85 mg GAE/mL in whey processed by spray-drying.

Antioxidant activity

Total antioxidant activity, while closely associated with phenolic content, was measured at the highest level in the encapsulated whey (1.62 mM TEAC). This finding suggests that encapsulation can effectively protect bioactive compounds from oxidation and enhance their bioavailability. In contrast, the antioxidant capacity of the spray-dried sample dropped to 0.58 mM TEAC, indicating that high processing temperatures led to structural degradation of antioxidant compounds.

Freeze-dried whey exhibited moderate antioxidant activity, with a value of 1.40 TEAC mM Trolox, while untreated whey showed a similar level of 1.30 TEAC mM Trolox. Although antioxidant capacity is partially preserved in both forms, these values were lower than those observed in the encapsulated whey sample. Freeze-drying, performed at low temperatures under vacuum, minimizes thermal damage to antioxidant components. In contrast, powdered whey produced by spray-drying showed the lowest antioxidant activity at 0.58 TEAC mM Trolox, suggesting that high-temperature treatments generally result in the loss of antioxidant compounds. The elevated temperatures (180 °C) used during spray-drying can cause oxidation of these components, reducing their functional properties [*29*].

As a result, the whey processing method was found to have a significant impact on antioxidant content. Encapsulation enhances the preservation of antioxidant compounds, whereas spray-drying generally leads to their loss. Onay-Ucar *et al.* [*22*] examined the antioxidant capacity of different whey protein fractions and reported values ranging from 1.2 to 1.5 TEAC mM Trolox. These values are consistent with the antioxidant capacities measured in this study for untreated whey (1.30 TEAC mM Trolox) and lyophilized whey (1.40 TEAC mM Trolox). The ability of lyophilization to preserve antioxidant activity is attributed to its low-temperature process, which minimizes the degradation of phenolic and other bioactive compounds. However, findings from Onay-Ucar *et al.* [*22*] also indicate that high-temperature methods, such as spray-drying, can reduce antioxidant capacity.

## CONCLUSIONS

In this study, the physical, chemical, and functional properties of whey samples processed by three different techniques, such as spray-drying, freeze-drying, and encapsulation, were comparatively evaluated. The results indicated that these methods differently influenced the structural integrity and functional quality of whey.

Encapsulation combined with freeze-drying was found to be the most effective method for preserving the nutritional value and functional properties of whey. This technique enhanced whey's functional quality by producing the highest values for parameters such as dry matter content, water-holding capacity, consistency, and antioxidant activity. These results suggest that carrier materials, such as maltodextrin and gum arabic, contribute both to the structural stability and the protection of bioactive compounds. Freeze-drying, performed at low temperatures, preserved the stability of proteins and phenolic compounds, maintaining antioxidant capacity and proving to be highly suitable for heat-sensitive substances. However, it was less effective than encapsulation in terms of water-holding capacity and consistency. Although spray-drying is rapid and convenient, it causes significant losses in protein content, phenolic compounds, and antioxidant activity due to the high temperatures involved. Therefore, careful consideration is required when applying this method, particularly for products where the preservation of heat-sensitive components is critical.

Encapsulation combined with freeze-drying was identified as the most effective method for preserving the nutritional value and enhancing the functional properties of whey. This approach yielded the highest dry matter content (16.33 %), water-holding capacity (4.65 %), consistency index (0.448), and antioxidant activity (1.62 mM Trolox). In addition, it maintained relatively high phenolic content (1.09 GAE/mL) and protein levels (0.82 %), indicating effective protection of bioactive compounds. These findings suggest that carrier materials such as maltodextrin and gum arabic enhance structural stability while protecting sensitive compounds during processing. Freeze-drying alone also proved to be a suitable method for heat-sensitive components, preserving phenolic content (1.13 mg GAE/mL), antioxidant capacity (1.40 mM Trolox), and protein content (0.81 %), while maintaining color ($L^*$=46.95) and improving water-holding capacity (3.91 %) compared to untreated whey. However, its effectiveness in improving consistency (0.130) and water-holding capacity remained lower than that of encapsulation. In contrast, spray-drying, despite its operational advantages, resulted in notable reductions in protein content (0.76 %), phenolic compounds (0.82 mg GAE/mL), and antioxidant activity (0.58 mM Trolox), along with decreased lightness (*L**=39.5), indicating quality deterioration due to high processing temperatures. Therefore, while spray-drying offers economic and practical benefits, its application should be carefully optimized, particularly for products requiring the preservation of heat-sensitive bioactive components.

In conclusion, while encapsulation emerges as the preferred method for enhancing the functional and nutritional properties of whey, the choice of processing technique should depend on the intended application of the product, such as a functional food additive, texture enhancer, or bioactive carrier. The advantages and limitations of each method should be carefully considered within a comprehensive evaluation framework.

## ACKNOWLEDGEMENTS

The authors gratefully acknowledge ENKA Inc. (Konya, Turkey) for supplying spray-dried whey powder.

## FUNDING

No funding was received to support this study from any organization.

## CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

## AUTHORS' CONTRIBUTION

S. Dede carried out the main experiments, prepared tables and figures, and wrote the first draft of the manuscript. S. Ozturkoglu-Budak supervised the entire study, conceived and designed the research, provided resources, and finalised the manuscript for submission. Both authors read and approved the final version of the manuscript.

## ORCID ID

S. Ozturkoglu-Budak https://orcid.org/0000-0003-1275-9025

## REFERENCES

*1.* Petruccioli M, Raviv M, Di Silvestro, R, Dinelli G. Agriculture and agro-industrial wastes, by-products, and wastewaters: Origin, characteristics, and potential in bio-based compounds production. In: M. Moo-Young (Ed.), Comprehensive Biotechnol. 2019:477–90; Elsevier.
https://doi.org/10.1016/B978-0-08-088504-9.00389-5

*2.* Zandona E, Blažić M, Režek Jambrak A. Whey utilisation: Sustainable uses and environmental approach. Food Technol. Biotechnol. 2021;59(2):147–61.
https://doi.org/10.17113/ftb.59.02.21.6968

*3.* Ozel B, McClements DJ, Arikan C, Kaner O, Oztop MH. Challenges in dried whey powder production: Quality problems. Food Res Int. 2022;160:111682.
https://doi.org/10.1016/j.foodres.2022.111682

*4.* Zeng X, Wang Y, Yang S, Liu Y, Li X, Liu D. The functionalities and applications of whey/whey protein in fermented foods: a review. Food Sci Biotechnol. 2023; 33(4):769-90.
https://doi.org/10.1007/s10068-023-01460-5

*5.* Rosa E, Prudencio ES. A comprehensive approach about comparison between drying technologies and powdered dairy products. Food Res Int. 2023;173:113326.
https://doi.org/10.1016/j.foodres.2023.113326

*6.* Kim S-H, Chang Y-H, Kwak HS. Physicochemical properties of reconstituted milk made from freeze-dried milk powder or spray-dried milk powder. Food Sci Anim Resour. 2010;30:28–35.
https://doi.org/10.5851/kosfa.2010.30.1.28

*7.* Yao Y, Zhao G, Yan Y, Chen C, Sun C, Zou X, Jin Q, Wang X. Effects of freeze drying and spray drying on the microstructure and composition of milk fat globules. RSC Adv. 2016; 6(4):2520–9.
https://doi.org/10.1039/C5RA22323G

*8.* Eun J-B, Maruf A, Das PR, Nam SH. A review of encapsulation of carotenoids using spray drying and freeze drying. Crit Rev Food Sci. 2020;60:3547–72.
https://doi.org/10.1080/10408398.2019.1698511

*9.* Elik A, Kocak Yanık D, Gögüş FA. Comparative study of encapsulation of carotenoid enriched-flaxseed oil and flaxseed oil by spray freeze-drying and spray drying techniques. LWT. 2021; 143:111153.
https://doi.org/10.1016/j.lwt.2021.111153

*10.* Drosou C, Krokida MA. Comparative study of encapsulation of β-carotene via spray-drying and freeze-drying techniques using pullulan and whey protein ısolate as wall material. Foods. 2024;13:1933.

https://doi.org/10.3390/foods13121933

*11.* Luca A, Çilek B, Hasırcı V, Şahin S, Sumnu G. Effect of degritting of phenolic extract from sour cherry pomace on encapsulation efficiency and production of nano-suspension. Food Bioprocess Technol. 2013;6(9):2494–502.
https://doi.org/10.1007/s11947-012-0880-z

*12.* AOAC. Official methods of analysis (16th ed.) Association of Official Analytical Chemists. 1995; Washington DC.

*13.* Isanga J, Zhang G. Production and evaluation of some physicochemical parameters of peanut milk yoghurt. LWT - Food Sci. Technol. 2019; 42(6):1132–38.
https://doi.org/10.1016/j.lwt.2009.01.014

*14.* González-Cuello RE, Pérez-Moreno JL, Rodríguez-Martínez A, Fernández-García P. Shelf life study of ice cream: Microbial viability and titratable acidity with microencapsulation techniques. Hikari J. 2024;15(3):123-30.
https://doi.org/10.12988/ces.2018.83132

*15*. Zabot GL, Schaefer Rodrigues F, Polano Ody L, Vinícius Tres M, Herrera E, Palacin H, *et al.* Encapsulation of bioactive compounds for food and agricultural applications. Polymers. 2022;14(19):4194.
https://doi.org/10.3390/polym14194194

*16.* Haque MA, Adhikari B. Drying and denaturation of proteins in spray drying process. In A. S. Mujumdar (Ed.), Handbook of Industrial Drying. Taylor & Francis Group: USA; 2015. pp. 971–84.
https://doi.org/10.1201/b17208-53

*17.* Ghanimah MA. Functional and technological aspects of whey powder and whey protein products. Int J Dairy Technol. 2018;71:454–9.
https://doi.org/10.1111/1471-0307.12436

*18*. Farahmandpour F, Haidari F, Heidari Z, Hajjarzadeh S, Ahangarpour A. Whey protein intervention and inflammatory factors and oxidative stress: Systematic review and meta-analysis of randomized controlled trials. Nutr Rev. 2025;83(4):609–21.
https://doi.org/10.1093/nutrit/nuae100

*19.* Guan H, Zhang W, Sun-Waterhouse D, Jiang Y, Li F, Waterhouse GIN, *et al.* Phenolic-protein interactions in foods and post ingestion: Switches empowering health outcomes. Trends Food Sci Tech. 2021;118:71-86.
https://doi.org/10.1016/j.tifs.2021.08.033.

*20.* Arkain B, Alibas I. Effect of different drying methods on bioactive potential and nutrients of medlar fruit. Sci Rep. 2025;15;10202.
https://doi.org/10.1038/s41598-025-86955-3

*21.* McEntee SA, Kelly AL, Lawless FN, McCarthy NA. Murphy EG. Effect of ultrafiltration of acid whey at pH 4.6 or 6.5 on whey concentrate composition and resultant powder properties. Int J Dairy Technol. 2025;78.
https://doi.org/10.1111/1471-0307.13116

*22.* Onay-Ucar E, Arda N, Pekmez M, Yılmaz AM, Boke-Sarıkahya N, Kırmızıgül S. Comparison of antioxidant capacity, protein profile and carbohydrate content of whey protein fractions. Food Chem. 2014;150:34-40.
https://doi.org/10.1016/j.foodchem.2013.10.119

*23.* Gausemel E, O'Mahony JA, Schüller RB, Liland K, Rukke EO, Skeie S, *et al.* Impact of spray drying temperatures on whey protein concentrate particle size, particle shape and related powder physical properties. Int J Dairy Technol.2025;78.
https://doi.org/10.1111/1471-0307.70052

*24.* Zhou S, Zhang X, Zhang J, Zang C, Fan R, Wang J, *et al*. Differences in physicochemical properties and proteomics analysis of spray- and freeze-dried milk powders from bovine, goat, and horse sources, J Dairy Sci. 2025;108 (2):1367-79.
https://doi.org/10.3168/jds.2024-25146

*25*. Başyiğit B, Karaaslan M. The effects of different drying techniques on the properties of desert truffle (*Terfezia boudieri* Chatin). Harran Univ Muh Dergi. 2020;5(3):239-46.
https://doi.org/10.46578/humder.822639

*26.* Huntington DA. The influence of the spray drying process on product properties. Drying Technol. 2004;22(6):1261-87.
https://doi.org/10.1081/DRT-120038730

*27.* Cheaib D, El Darra N, Rajha HN, El-Ghazzawi I, Maroun RG, Louka N. Effect of the extraction process on the biological activity of lyophilized apricot extracts recovered from apricot pomace. Antioxidants (Basel). 2018;14:7(1):11.
https://doi.org/10.3390/antiox7010011.

*28.* Grgić J, Šelo G, Planinić M, Tišma M, Bucić-Kojić A. Role of the encapsulation in bioavailability of phenolic compounds. Antioxidants. 2020; 9(10):923.
https://doi.org/10.3390/antiox9100923

*29.* Gomes WF, França FRM, Denadai M, Andrade JKS, da Silva Oliveira EM, de Brito ES, *et al*. Effect of freeze- and spray-drying on physico-chemical characteristics, phenolic compounds and antioxidant activity of papaya pulp. J Food Sci Technol. 2018;55(6):2095-102. https://doi.org/10.1007/s13197-018-3124-z

*30*. Li Y, Zhou L, Zhang H, Liu G, Qin X. Preparation, characterization and antioxidant activity of glycosylated whey protein isolate/proanthocyanidin compounds. Foods. 2023;12(11):2153. https://doi.org/10.3390/foods12112153

Table 1. Chemical properties of whey with different treatments

| Property | Untreated whey | Freeze-dried RS whey | Encapsulation+freeze-dried RS whey | Spray-dried RS whey |
|---|---|---|---|---|
| pH | $(6.49\pm0.020)^{A}$ | $(6.50\pm0.025)^{A}$ | $(6.50\pm0.040)^{A}$ | $(6.64\pm0.010)^{A}$ |
| TA/% | $(0.09\pm0.003)^{B}$ | $(0.10\pm0.003)^{B}$ | $(0.16\pm0.005)^{A}$ | $(0.09\pm0.001)^{B}$ |
| *w*(dry matter)/% | $(6.36\pm0.020)^{D}$ | $(8.32\pm0.050)^{C}$ | $(16.33\pm0.055)^{A}$ | $(8.72\pm0.030)^{B}$ |
| *w*(protein)/% | $(0.87\pm0.010)^{A}$ | $(0.81\pm0.050)^{AB}$ | $(0.82\pm0.255)^{AB}$ | $(0.76\pm0.035)^{B}$ |
| *w*(ash)/% | $(0.43\pm0.000)^{B}$ | $(0.64\pm0.025)^{A}$ | $(0.64\pm0.015)^{A}$ | $(0.67\pm0.015)^{A}$ |
| *w*(fat)/% | $(0.40\pm0.002)^{B}$ | $(0.40\pm0.000)$ | $(0.40\pm0.000)$ | $(0.40\pm0.000)$ |
| WHC/% | $(2.72\pm0.045)^{C}$ | $(3.91\pm0.005)^{B}$ | $(4.65\pm0.045)^{A}$ | $(4.60\pm0.160)^{A}$ |

RS=reconstituted, TA=titration acidity (lactic acid percentage), WHC=water-holding capacity. Differences between all samples ($p<0.05$) are indicated by capital letters (A, B, C, D). The difference between values with the same capital letter (A) was not significant ($p>0.05$). Values are shown as mean±S.D.

Table 2. Rheological values of whey with different treatments

| Index | Untreated whey | Freeze-dried RS whey | Encapsulation+freeze-dried RS whey | Spray-dried RS whey |
|---|---|---|---|---|
| *K* | $(0.020\pm0.001)^{D}$ | $(0.130\pm0.000)^{C}$ | $(0.448\pm0.002)^{A}$ | $(0.190\pm0.001)^{B}$ |
| *n* | $(0.560\pm0.009)^{B}$ | $(0.247\pm0.000)^{C}$ | $(0.209\pm0.003)^{D}$ | $(0.696\pm0.006)^{A}$ |

RS=reconstituted, *K*=consistency index, *n*=flow behavior index. Differences between all samples ($p<0.05$) are indicated by capital letters (A, B, C, D). Values are shown as mean±S.D.

Table 3. Color values of whey with different treatments

| Parameter | Untreated whey | Freeze-dried RS whey | Encapsulation+freeze-dried RS whey | Spray-dried RS whey |
|---|---|---|---|---|
| *L** | (46.75±1.65)$^{B}$ | (46.95±0.15)$^{B}$ | (55.7±0.40)$^{A}$ | (39.5±0.00)$^{C}$ |
| *a** | (−2.05±0.05)$^{A}$ | (−2.3±0.00)$^{B}$ | (−2.95±0.05) $^{C}$ | (−2.8±0.00)$^{C}$ |
| *b** | (0.15±0.35)$^{A}$ | (0.3±0.40)$^{A}$ | (1.55±0.95)$^{A}$ | (1.7±0.00)$^{A}$ |

RS=reconstituted. Differences between all samples (p<0,05) were indicated by capital letters (A, B, C, D). The difference between values with the same capital letter (A) was not significant (p>0.05). Values are shown as mean±S.D.

Table 4. Functional properties of whey with different treatments

| Property | Untreated whey | Freeze-dried RS whey | Encapsulation+freeze-dried RS whey | Spray-dried RS whey |
|---|---|---|---|---|
| *γ*(phenolics as GAE)/(mg/mL) | (1.15±0.02)$^{A}$ | (1.13±0.11)$^{A}$ | (1.09±0.01)$^{B}$ | (0.82±0.01)$^{C}$ |
| Antioxidant capacity as *c*(TEAC)/mM | (1.30±0.015)$^{B}$ | (1.40±0.010)$^{B}$ | (1.62±0.025)$^{A}$ | (0.58±0.020)$^{C}$ |

RS=reconstituted, GAE=gallic acid equivalent, TEAC=Trolox equivalent antioxidant capacity. Differences between all samples (p<0.05) are indicated by capital letters (A, B, C, D). Values are shown as mean±S.D.