Optimization of Spray Drying Conditions for Production of Functional Goat Milk Powder Enriched with Pomegranate Peel Extract (*Punica granatum* L.)

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ABSTRACT

Background: Pomegranate peel, a by-product of juice processing, is rich in bioactive compounds. Encapsulating its extract offers potential applications in food processing. Milk serves as an excellent carrier for such bioactive compounds due to its widespread consumption. Unlike cow milk, goat milk is more nutritious and provides additional health benefits. However, milk proteins lack phenolic compounds, making supplementation with pomegranate peel extract beneficial. Spray drying is a widely used, cost-effective encapsulation technique for functional food production.

Methods: This study aimed to optimize the addition of maltodextrin and the inlet temperature in the spray drying process to produce functional goat milk powder. A Response Surface Methodology (RSM) with a Central Composite Design (CCD) was employed. The independent variables included maltodextrin concentrations (5%, 10%, 15%) and inlet temperatures (160°C, 180°C, 200°C). The response variables were total phenolic content, antioxidant activity, solubility, hygroscopicity, bulk density and whiteness. The optimized functional goat milk powder was further characterized.

Result: The optimal conditions were determined as 12.10% maltodextrin concentration and an inlet temperature of 178.98°C. The resulting functional goat milk powder exhibited total phenolic content of 272.11 mg GAE/g, antioxidant activity of 55.36%, solubility of 95.55%, hygroscopicity of 1.91%, bulk density of 0.46 g/mL and whiteness of 76.89. Characteristics of optimized functional goat milk powder revealed water content of 3.85%, fat content of 26.76% and protein content of 31.23%. FTIR analysis identified the presence of a C=C functional group at absorption bands 1680.73 cm^{-1} and 480.38 cm^{-1}. SEM analysis showed agglomerated small particles, while Particle Size Analysis (PSA) indicated a monomodal distribution with an average particle size of 6.2293 µm.

Key words: Goat milk powder, Inlet temperature, Maltodextrin, Pomegranate peel extract, Spray drying.

INTRODUCTION

Consumers' increasing awareness of the close relationship between food and health is rapidly shaping innovation in the food industry. This has stimulated a growing interest in developing healthier, more sustainable products that incorporate naturally derived bioactive compounds (Rabadán *et al*., 2021). One source of these compounds is pomegranate peel, a by-product of juice processing, which constitutes about 26-30% of the fruit's total weight (Mo *et al*., 2022). Traditionally used in folk medicine, pomegranate peel contains higher concentrations of beneficial substances than the edible fruit portion, particularly polyphenols (Biesalski *et al*., 2009; Padayachee *et al*., 2016).

The major bioactive constituents in pomegranate peel include tannins, flavonoids and phenolic acids such as punicalagin (an ellagitannin), gallic acid, ellagic acid, caffeic acid and others (Mo *et al*., 2022). These compounds exhibit a broad range of biological activities-antioxidant, anti-inflammatory, anticancer, antibacterial, antiviral, cardiovascular protective and respiratory-modulating properties-making them highly attractive for functional food applications (Mo *et al*., 2022). The concentration of total phenolics in pomegranate peel can vary widely (18-510 mg/g dry matter), depending on factors such as solvent type, extraction method and pomegranate variety (García *et al*., 2021). Several studies have explored the incorporation of pomegranate peel extract as a functional ingredient in various food products. For instance, its application in cookie

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formulations has demonstrated an enhancement in nutritional attributes compared to conventional cookie (Kavitha *et al*., 2021). Additionally, the fortification of yogurt with pomegranate peel extract has been reported to improve its physicochemical characteristics and sensory acceptability, thereby increasing its nutritional value and consumer appeal (Bakhti *et al*., 2025).

Milk and dairy products, due to their high nutrient content and widespread consumption, represent a promising carrier for incorporating pomegranate peel extracts (Augustin and Oliver, 2014). While cow milk remains prevalent worldwide, goat milk ranks third in global production after cow and buffalo milk (De Santis *et al*., 2019). The rising production of goat milk has been accompanied by a growing global awareness of its benefits, contributing to its increased use for both nutritional and medicinal purposes (Hammam *et al*., 2022), including superior digestibility and positive effects on lipid metabolism (Miller and Lu, 2019). Previous studies have shown that incorporating pomegranate peel extracts in camel milk products could boost antioxidant capacity, attributed partly to the presence of tannins (Mortazavi *et al*., 2021). However, similar encapsulation using goat milk as a matrix has been explored less.

Spray drying is the most popular, flexible and affordable encapsulation method among the food and pharmaceutical industries because it is easy to scale up, repeatable and preserves product quality and stability despite a short drying period (Desai and Park, 2005). The addition of maltodextrin for the spray drying process is needed to protect heat-sensitive nutrient components, thereby reducing the occurrence of the Maillard reaction (Masum *et al*., 2020). Another factor that plays a role in the spray drying process is the inlet temperature, which ranges from 150-220°C (Lipan *et al*., 2020).

Although numerous studies have examined spray-drying microencapsulation of pomegranate peel phenolic extracts using various encapsulating materials, such as orange juice by-products (Kaderides and Goula, 2019), maltodextrin and whey protein (Šavikin *et al*., 2021) and whey protein with β-cyclodextrin (Zhang *et al*., 2024). There is limited research exploring goat milk and maltodextrin as encapsulating agents. Therefore, the current study aimed to optimize the spray-drying conditions (maltodextrin concentration and inlet temperature) for producing a functional goat milk powder enriched with pomegranate peel extract. A Central Composite Design *via* Response Surface Methodology (RSM) was employed to determine the optimal combination of maltodextrin concentrations (5-15%) and inlet temperatures (160-200°C). The resulting optimized product was then characterized and compared to a control goat milk powder.

MATERIALS AND METHODS

Materials

This study used fresh goat milk from 1-year-old Etawa goats (*Capra aegagagrus hircus*) in Blimbing District, Malang, Indonesia. Red pomegranate peels (*Punica granatum*) were sourced from Mangaran Village, Situbondo, Indonesia. Additional ingredients included maltodextrin (Tereos FKS, DE 10-12), Folin-Ciocalteu reagent (Merck, Germany) and DPPH (TCI, Tokyo).

This study was conducted from September 2023 to May 2024 in the Faculty of Agricultural Technology, Brawijaya University, Indonesia.

Pomegranate peel extract preparation

Pomegranate peels were cleaned, cut and dried at 50±5°C for 24 hours using a food dehydrator. The dried peels were ground into fine powder (80-mesh sieve). A 1:10 ratio of pomegranate peel powder and 70% ethanol was extracted for 90 minutes with a magnetic stirrer. The extract was evaporated with a rotary evaporator and stored at 4°C, following Sandhya *et al*. (2018) with modifications.

Goat milk powder preparation

Fresh goat milk (500 mL) was pasteurized at 72°C for 15 seconds and cooled to room temperature. Pomegranate peel extract (5%) and maltodextrin (with the concentration according to the results of the design expert) were mixed at 150 rpm for 15 minutes using a magnetic stirrer. The mixture was then spray-dried at the inlet temperature according to the results of the design expert to produce functional goat milk powder (Sert *et al*., 2021). Optimization was verified using Response Surface Methodology (RSM) based on total phenol, antioxidant activity, solubility, hygroscopicity, bulk density and color.

Optimization analysis

Total phenolic content and antioxidant activity

Phenolic content was measured using the Folin–Ciocalteu assay (Derakhshan *et al*., 2018) and expressed as mg GAE/g. Samples (100 µL) were mixed with 500 µL Folin-Ciocalteu reagent and 400 µL sodium carbonate (7.5%), incubated for 30 minutes and analyzed at 765 nm using a UV-Vis spectrophotometer (Shimadzu UV-1280, Japan).

Antioxidant activity was assessed *via* DPPH radical scavenging (Wang *et al*., 2023). A 2 mL sample was mixed with 2 mL of 0.0003% DPPH solution in 95% ethanol, incubated for 30 minutes in the dark and analyzed at 517 nm using UV-Vis spectrophotometer (Shimadzu UV-1280, Japan). Ethanolic DPPH solution was used as the control.

DPPH scavenging activity (%) =

$$\frac{\text{Absorbamce of control - Absorbamce of extract}}{\text{Absorbamce of control}} \times 100\%$$

Solubility, hygroscopicity, bulk density and color (whiteness) analysis

Solubility, hygroscopicity and bulk density analysis was performed following the method described by Rohadi *et al*. (2020). The color of functional goat milk powder was measured using a color reader (Konica Minolta CR-10, Japan). The sample was placed in a plastic container or cup. Testing was carried out with a color reader; the sensor

part of the tool was attached to the flat part of the container. The device was turned on by pressing the Lab button and the results were recorded including the values (L, a and b) (Thakur *et al*., 2021). The color of goat milk powder was presented in whiteness value and calculated using the following equation:

$$\text{Whiteness} = 100 - [(100-L)^2 + a^2 + b^2]^{1/2}$$

Product characterization

The optimized functional goat milk powder obtained was characterized and compared with the control goat milk powder (without the addition of pomegranate peel extract).

Fourier transform infra-red (FTIR)

The FTIR spectra were acquired using a slightly modified version of the method outlined by Kennas *et al*. (2020). Each powder sample (1 mg) was mixed with 100 mg of potassium bromide (KBr) and tablets (1 cm in diameter) were formed by compressing the mixture. These tablets were then scanned using FTIR spectroscopy (Shimadzu, Japan) at a resolution of 2 cm^{-1} over the range of 4000 to 380 cm^{-1}.

Scanning electron microscopy (SEM)

A field emission SEM (Hitachi SU3500, Japan), equipped with a tungsten filament, was employed to analyze the surface morphology of the powder. The powder samples were prepared by mounting powders on the carbon adhesive tabs. Powders were then sputter-coated with gold at 18 mA for 60 s and finally examined at 2.00 kV accelerating voltage. The images were obtained at different magnifications to better observe the surface condition of the powders (Thakur *et al*., 2021).

Particle size analysis

The particle size of powder particles was determined using a particle size analyzer (Shimadzu, Japan) with Litesizer DLS 500 (serial number 82783594) coupled with a module type BM10 (serial number 82786489). The dry feeder dispersed powder particles through a venturi using compressed air. The particles were then conveyed into the measurement cell using a suction effect. The absorption and refractive index of powder particles were set at 0.01 and 1.45, respectively. The refractive index of air was set at 1.00 (Masum *et al*., 2020).

RESULTS AND DISCUSSION

Optimization effects on the physicochemical properties of functional goat milk powder

Table 1 presents the experimental results for the physico-chemical properties of functional goat milk powder, including total phenolic content, antioxidant activity, solubility, hygroscopicity, bulk density and whiteness.

Influence of maltodextrin concentration and inlet temperature

The relationship between maltodextrin concentration (X1) and inlet temperature (X2) on total phenolic content (Y1), antioxidant activity (Y2), solubility (Y3), hygroscopicity (Y4), bulk density (Y5) and whiteness (Y6) follows a quadratic regression model. Analysis of variance (ANOVA) results indicate a significant influence ($p \leq 0.05$). These interactions are visually represented through contour plots (Fig 1a-1f) and 3D surface plots (Fig 2a-2f).

Effects on total phenolic content and antioxidant activity

The addition of maltodextrin during spray drying enhances total phenolic content by forming hydrogen bonds, which protect bioactive compounds (Shishir and Chen, 2017; Navarro-Flores *et al*., 2020). Higher inlet temperatures improve antioxidant activity due to water loss, which concentrates antioxidants (Thakur *et al*., 2021). However, excessive temperatures can degrade bioactive compounds,

Table 1: Variable value of functional goat milk powder responses.

Variables		Response					
Maltodextrin concentration (%)	Inlet temperature (°C)	Total phenolic content (mg GAE/g)	Antioxidant activity (%)	Solubility (%)	Hygroscopicity (%)	Bulk density (g/mL)	Color (whiteness)
5	160	268.96	51.75	90.84	2.16	0.28	71.28
15	160	270.87	53.85	95.96	1.51	0.47	77.26
5	200	269.81	52.05	96.31	2.34	0.22	75.93
15	200	271.19	54.22	93.16	1.23	0.41	80.82
2.928	180	267.79	50.77	88.93	2.64	0.27	69.77
17.071	180	270.45	53.48	93.09	1.25	0.49	80.85
10	151.716	271.18	54.14	92.04	1.76	0.33	77.17
10	208.284	271.31	53.69	96.31	1.67	0.21	75.02
10	180	272.69	55.72	96.43	1.44	0.31	75.22
10	180	272.62	55.66	97.82	1.35	0.32	74.61
10	180	272.51	55.50	92.16	1.32	0.37	75.42
10	180	272.75	55.74	96.34	1.31	0.31	76.00
10	180	272.68	55.81	96.31	1.29	0.38	75.67

reducing their effectiveness (Thakur *et al*., 2021). A combination of 10% maltodextrin and 180°C inlet temperature minimizes thermal degradation by forming a protective barrier (Nadali *et al*., 2022). Additionally, pomegranate peel extract contributes significantly to the phenolic content, with key bioactive compounds including ellagitannins, ellagic acid and gallic acid (Khan *et al*., 2017).

Effects on solubility and hygroscopicity

Solubility increases with maltodextrin concentration, as maltodextrin acts as a filler and enhances powder dispersibility (Kalušević *et al*., 2017). Maltodextrin up to 15% maintains a non-hygroscopic nature in the powder (Samsu and Zahir, 2020). It helps control moisture absorption, ensuring a drier product with stable texture (Bednarska and Janiszewska-Turak, 2020). Higher drying temperatures result in lower moisture content, enhancing stability and reducing hygroscopicity (Shishir and Chen, 2017). The formation of a protective maltodextrin layer further reduces exposure to humidity (Sidlagatta *et al*., 2020). High-temperature drying reduces particle size, resulting in a finer powder with greater surface area for rehydration (Masum *et al*., 2019; Leyva-Porras *et al*., 2019). However, protein aggregation due to β-lactoglobulin and casein interactions can negatively affect solubility (Thakur *et al*., 2021).

Effects on bulk density

Bulk density increases with maltodextrin concentration as additional particles fill the matrix, reducing empty space (Teo *et al*., 2021). Conversely, higher inlet temperatures reduce bulk density due to rapid water evaporation, leading to porous structures (Thakur *et al*., 2021; Djaafar *et al*., 2018). The viscosity of the maltodextrin solution decreases at higher temperatures, allowing more efficient water removal and lower bulk density (Saha *et al*., 2019).

Effects on color

Maltodextrin enhances whiteness by preventing Maillard reactions and oxidation during drying (Sarabandi *et al*., 2019; Neves *et al*., 2019). However, at inlet temperatures above 140°C, the Maillard reaction between lactose and proteins

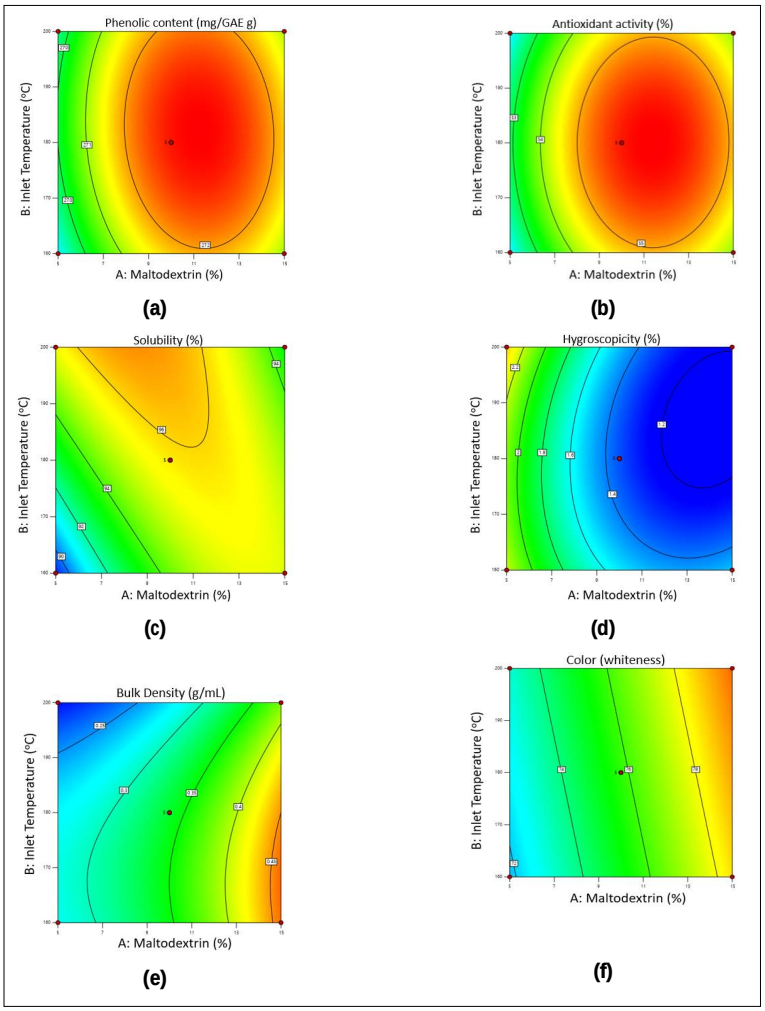


Fig 1: Contour graphic plot of total phenolic content (a), antioxidant activity (b), solubility (c), hygroscopicity (d), bulk density (e), color whiteness (f).

causes browning (Deshwal *et al*., 2020). Maltodextrin stabilizes pigment molecules, but excessive heat alters molecular interactions, reducing whiteness (Chng *et al*., 2020; Thakur *et al*., 2021).

Characterization of optimized functional goat milk powder

Optimization and verification

The optimization of functional goat milk powder was conducted using a maltodextrin concentration of 12.10% and an inlet temperature of 178.98°C, yielding a desirability value of 0.888. The verification results for total phenolic content, antioxidant activity, solubility, hygroscopicity, bulk density and whiteness are summarized in Table 2.

Fourier transform infrared spectroscopy (ftir) analysis

FTIR analysis identified distinct absorption bands in the optimized functional goat milk powder, particularly at 1680.73 cm^{-1} and 480.38 cm^{-1}, indicating the presence of C=C bonds associated with alkenes and aromatic compounds

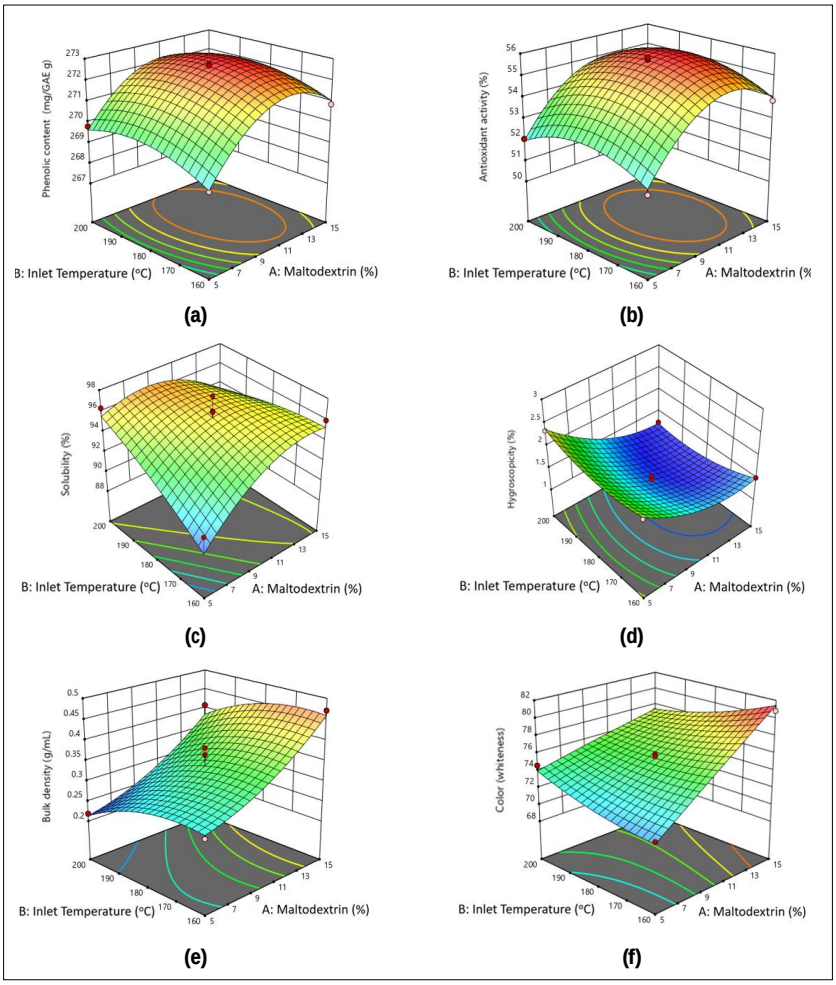


Fig 2: The 3D graphic surface of phenolic content (a), antioxidant activity (b), solubility (c), hygroscopicity (d), bulk density (e), color (whiteness) (f).

Table 2: Simultaneously optimized process conditions with prediction target and verification responses.

Parameter	Criteria	Prediction	Verification
Maltodextrin concentration, %	In range	-	-
Inlet temperature, °C	In range	-	-
Total fenol, mg GAE/g	Maximize	272.70	272.57±0.12
Antioxidant activity, %	Maximize	55.79	55.70±0.27
Solubility, %	Maximize	95.85	95.85±0.11
Hygroscopicity, %	Minimize	1.20	1.91±0.08
Bulk density, g/mL	In range	0.38	0.40±0.03
Color whiteness	Maximize	76.95	77.16±0.50

Table 3: Comparison of characteristics of goat milk powder.

Peak IR (spectrum infrared)	Goat milk powder control		Optimized functional goat milk powder	
	Wavelength (cm^{-1})	Function clusters	Wavelength (cm^{-1})	Function clusters
1.	3293.72	O-H	3297.31	O-H
2.	2921.05	C-H	2923.20	C-H
3.	2853.55	C-H	2853.55	C-H
4.	2360.96	O=C=O	2362.40	O=C=O
5.	-	-	1680.73	C=C
6.	1643.44	C=O	1644.87	C=O
7.	1643.63	C=O	1642.91	C=O
8.	1548.13	C-N	1536.64	C-N
9.	1453.34	C-H	1454.78	C-H
10.	1413.85	C-H	1416.00	C-H
11.	1365.74	C-H	1375.07	C-H
12.	1242.23	C-O	1240.08	C-O
13.	1149.61	C-O-C	1149.61	C-O-C
14.	1075.65	C-O	1077.08	C-O
15.	1015.33	C-O	1018.92	C-O
16.	932.75	C-OH	931.32	C-OH
17.	893.98	C-OH	894.70	C-OH
18.	853.77	C-OH	850.18	C-OH
19.	760.42	C-OH	761.86	C-OH
20.	702.26	C-OH	703.69	C-OH
21.	567.98	C-OH	573.73	C-OH
22.	517.00	C-OH	522.03	C-OH
23.	-	-	480.38	Organic component

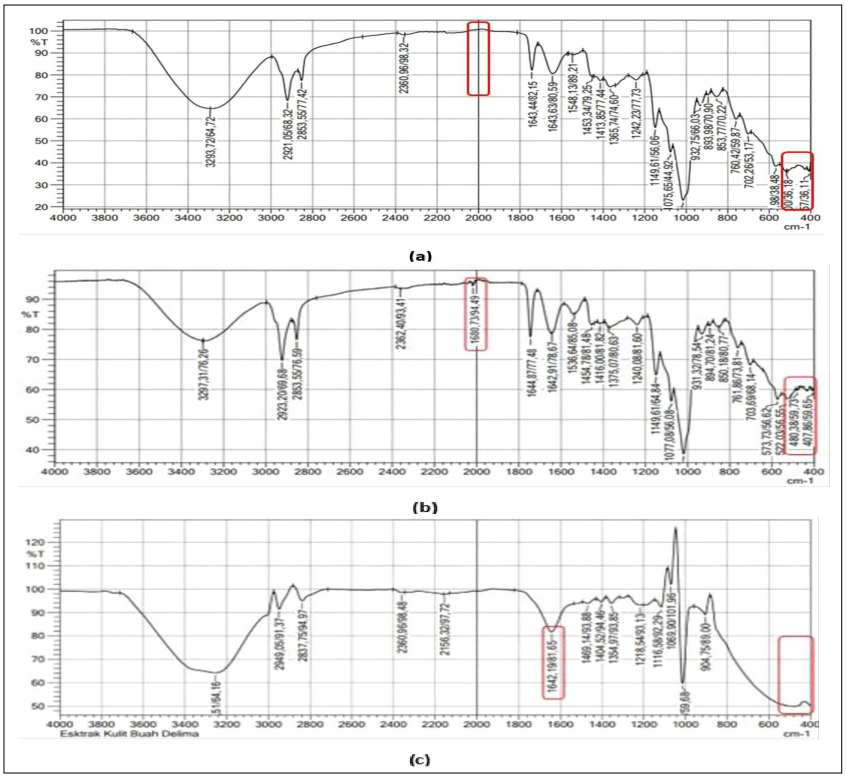


Fig 3: Results of FTIR analysis of control goat milk powder (a), optimized functional goat milk powder (b), pomegranate peel extract (c).

(Table 3 and Fig 3). These spectral shifts suggest the successful incorporation of phenolic compounds from pomegranate peel extract, such as punicalagin and ferulic acid (Bertolo *et al*., 2020; Sehari *et al*., 2022). Additionally, organic components, including minerals and oxygen-containing compounds, were detected, confirming the contribution of bioactive compounds from the extract (Ben-Ali *et al*., 2018).

Microstructure analysis

SEM revealed that the addition of pomegranate peel extract led to the formation of agglomerates due to interactions between polyphenols, tannins and milk proteins (Fig 4). These interactions resulted in the formation of a matrix around the granules, influencing particle clustering. However, the slight variations in agglomerate shape did not significantly affect solubility, hygroscopicity, or bulk density. The presence of micropores in the agglomerates enhances water absorption, which can impact the bulk density of the powder (Zhong *et al*., 2016; AlYammahi *et al*., 2023; Thakur *et al*., 2021).

Particle size distribution

Particle size analysis using PSA indicated an average particle size of 6.1755 µm for the control and 6.2293 µm for the optimized functional goat milk powder (Fig 5). The slight increase in particle size is attributed to polyphenol-protein interactions, particularly with tannins, which promote protein aggregation. This results in the formation of larger, less water-soluble protein-tannin complexes, influencing the powder's physical properties (Slim *et al*., 2019; Trigueros *et al*., 2014). The observed particle sizes are comparable to those reported for spray-dried camel milk powder at an inlet temperature of 170°C (Deshwal *et al*., 2020).

These findings confirm that the optimization process successfully enhanced the functional properties of goat milk powder by incorporating bioactive compounds from pomegranate peel extract, leading to improved antioxidant activity, structural modifications and stable physical characteristics.

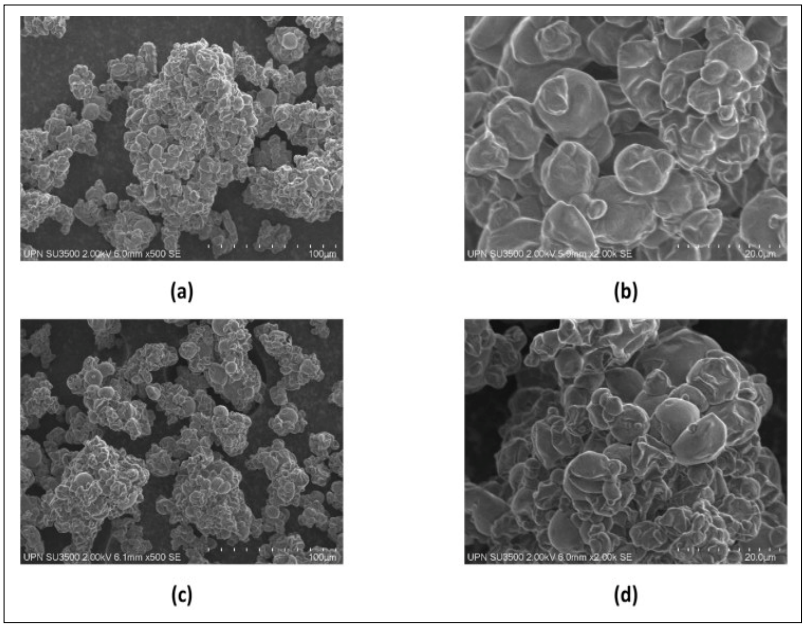


Fig 4: Microstructure of control goat milk powder 500× (a), 2000× (b) and optimized functional goat milk powder 500× (c), 2000× (d).

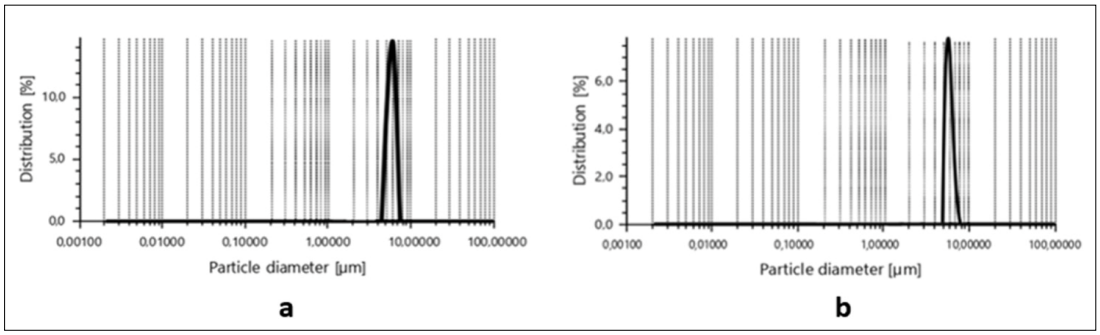


Fig 5: Particle size results of control goat milk powder (a) and optimized functional goat milk powder (b).

CONCLUSION

The encapsulation of pomegranate peel extract in functional goat milk powder was influenced by variations in maltodextrin concentration and inlet temperature during the spray drying process. The optimal conditions were identified at 12.10% maltodextrin concentration and 178.98°C inlet temperature, yielding a product with a total phenol content of 272.57 mg GAE/g, 55.70% antioxidant activity, 95.85% solubility, 1.91% hygroscopicity, 0.40 g/mL bulk density and a whiteness (L value) of 77.16. SEM analysis revealed agglomerated microstructures with uniform, grouped small particles. FTIR analysis indicated additional functional groups associated with phenolic compounds such as punicalagin, ellagic acid, p-coumaric acid and ferulic acid, while PSA analysis showed a monomodal particle size distribution, with average sizes of 6.1755 µm for the control and 6.2293 µm for the optimized product.

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Disclaimers

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Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this article. No funding or sponsorship influenced the design of the study, data collection, analysis, decision to publish, or preparation of the manuscript.

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