Acta Alimentaria 52 (2023) 2, 220–234
DOI: 10.1556/066.2022.00258

AKADÉMIAI KIADÓ

Optimisation of spray drying parameters using mixed flow in whey powder production using response surface methodology (RSM)

A. Mouzai[1*], A. Kheroufi[1], L. Afoutni[1], W. Louaer[2], M.N. Zidoune[3] and H. Boughellout[1]

[1] Laboratoire de Génie Agro-Alimentaire (GENIAAL), Équipe Génie des Procédés Alimentaires, Biodiversité et Agroenvironnement (GPABAE), INATAA Université frères Mentouri Constantine 1, Constantine, 25000, Algeria

[2] Laboratoire d'Ingénierie de Procédés de l'Environnement, Université Salah Boubnider Constantine 3, Constantine, 25000, Algeria

[3] Laboratoire de nutrition et technologies alimentaires (L.N.T.A.), Équipe Transformation et Elaboration des Produits Agro-alimentaires (T.E.P.A.), INATAA Université frères Mentouri Constantine 1, Constantine, 25000, Algeria

ORIGINAL RESEARCH PAPER

Received: December 21, 2022 • Accepted: April 1, 2023

ABSTRACT

This work explored the impact of mixed flow spray drying on the physical and functional properties of whey powder without any subsequently added drying agent to increase whey utilisation. Spray drying was performed on a pilot scale using a mixed flow spray dryer. The effects of the inlet air temperature (150–210 °C) and feed flow rate (2–7 L h^{-1}) on several responses such as moisture content, yield, dispersibility, bulk density, and outlet air temperature were investigated using response surface methodology. In addition, with the optimised parameters, Carr index, Hausner ratio, solubility, wettability, hygroscopicity, degree of caking, crystallinity, and morphology of the obtained whey powder were determined. The investigation revealed that feed flow rate is the main parameter influencing all responses. The inlet air temperature significantly affected the bulk, tapped density, and outlet air temperature. The optimal inlet air temperature

---

[*] Corresponding author. Tel.: +213 552 36 30 51. E-mail: abdelghani.mouzai@umc.edu.dz

and feed flow rate for the production of whey powder were 182 °C and 3.2 L h^{-1}, respectively. Under these parameters the moisture content, yield, bulk density, hygroscopicity, and degree of caking of the obtained product were 28.6, 1.80%, 0.24 g cm^{-3}, 16.10 g H_2O/g powder, and 85.56%, respectively.

KEYWORDS

whey powder, spray drying, outlet air temperature, feed flow rate, functional properties, mixed flow

1. INTRODUCTION

Whey is considered as the most abundant by-product of cheese making in the dairy industry (Kheroufi et al., 2022) with high nutritional value. It is typically produced in high volumes, and its discharge as wastewater is environmentally hazardous. According to Fischer and Kleinschmidt (2021), about 212,211 × 10^6 kg of whey per year are produced.

Recently, the worldwide food insecurity has significantly increased due to population growth, socio-economic challenges, the consequences of climate change, and most recently the COVID-19 pandemic (Zhu et al., 2022) and also the Russian-Ukrainian conflict. So, the transition to a circular food system, where regenerative food production is favoured, is much required.

Spray drying aims to transform whey into a stable product for further use, making it a high value added product (Jafari et al., 2019). In addition, spray drying is eight times more economical than freeze drying and four times more economical than vacuum drying (Samsu and Zahir, 2020).

Whey powder is used for their functional properties in the cookie, pastry, bread-making, dairy beverage, chocolate, and meat industries (Selvamuthukumaran, 2019). In addition to its nutritional properties, whey powder improves the taste, aroma and texture of the finished product (Ghanimah, 2018).

The powder quality is influenced by several spray dryer operating parameters, and this is justified by the studies that have been carried out using the co-current spray dryer (Jafari et al., 2019; Samsu and Zahir, 2020).

Until today, there are a few scientific works on the influence of the operating parameters using the mixed flow dryer upon the powder's probiotic properties (Jiang et al., 2020), but no studies have been reported upon whey powder.

Hence, the goals of this work were to study the impact of drying conditions on the properties of whey powder using a reverse phase counter current system spray dryer and to determine the optimal process conditions to have a value added whey powder without the addition of any drying agent.

2. MATERIALS AND METHODS

The whey used in this study came from GIPLAIT-Numidia, an Eastern Algerian dairy producer (Constantine, Algeria). It was recovered after draining during cheese manufacturing (soft chesse pate). Before use, whey was characterised by the determination of its total dry matter (AOAC method 934.01), fat (AOAC method 991.36), ash (AOAC method 930.05), and protein contents

(AOAC method 981.10), using the official methods of analysis (AOAC, 2000). The whey was dried in a pilot-scale spray dryer (model MP314, DALTALEB, France), with a stainless steel spray tower (diameter 1.1 m, height 2 m, conical base) and equipped with a support for the electric box, a supply tank, and a heater. The liquid was injected by sub-pressure, and the contact of the product and the drying air was subjected to a reverse phase counter current system (Fig. 1a). A bi-fluid nozzle of air and liquid injection ensured the spraying of the suspension in the drying chamber.

In the present work, response surface methodology (RSM) was performed with central composite design. The studied factors were inlet air temperature (IAT) ranged between 150 to 210 °C and feed flow rate (FFR) ranged between 2 and 7 L h^{-1}. These two parameters were used as independent variables for process optimisation to determine the effect of dryer operation on yield, moisture, dispersibility, bulk density, and outlet air temperature. The temperature of whey liquid was 35 °C. The drying air flow rate was set at 250 ± 20 $m^3 h^{-1}$, and the injection of air flow rate at the nozzle was maintained at 300 L h^{-1}. The operation conditions of the spray dryer were selected according to the preliminary tests and scientific works. A composite central design with 13 trials formed by 5 central points and 4 axial points to 2^2 complete factorial design was used (Table 1). A second-degree polynomial equation was used to relate the response variable (Y) to the independent variables (X).

$$\mathrm{Y} = B_{0+}\sum B_iX_{i+}\sum B_{ii}X_i^2+\sum B_{ij}X_iX_j \tag{1}$$

The desirability function method was used to optimise multiple responses simultaneously.

The resulting powders were stored in hermetically sealed boxes for further analysis.

Moisture content was determined by quantifying the weight loss (%) of 10 g of whey powder after oven drying at 102 °C for 5 h. The spray-dried powder yield was calculated as the ratio of the weight of powder collected after spraying to the total solids content of the feed (Jafari et al., 2019).

Solubility, dispersibility, wettability, bulk, and tapped density were determined according to the methods described by Schuck et al. (2012). The fluidity and cohesion of the whey powder, expressed by Carr Index (CI), and Hausner Ratio (HR) were determined from tapped and bulk density values of the powder according to the formulations giving by Seth et al. (2017). Hygroscopicity and caking degree were determined according to the methods given by Westergaad (2010). The outlet air temperature (OAT) was given automatically by means of a probe connected to the cyclone outlet for each combination of operating conditions. An X-ray diffractometer (Bruker AXS, D8 Advance, Germany) was used to determine the physical state of optimised whey powder constituents. The morphology of whey powder was examined using scanning electron microscopy (JEOL JSM-7001F, Germany), which was operated at an accelerating voltage of 12 kV. The experimental design and statistical analysis were performed using Minitab 18 software. Significant terms in the models were found by variance analysis ANOVA.

3. RESULTS AND DISCUSSION

Table 2 shows the physical and chemical characteristics of the whey used in this study. These characteristics are comparable to those noted by Yadav et al. (2015). The whey used is classified as a sweet whey with a pH > 6.

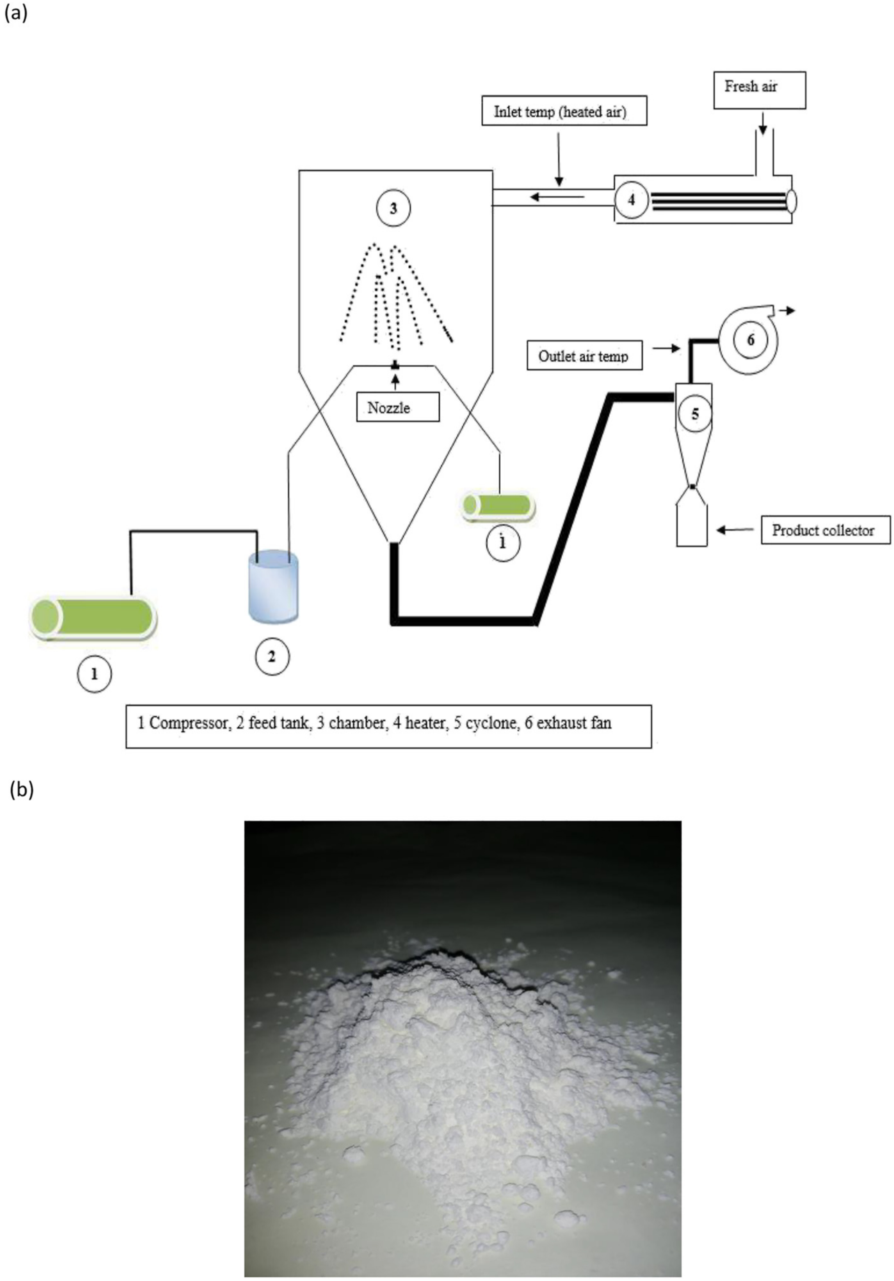

Fig. 1. (a): Diagram of the spray dryer, (b): Image of whey powder after optimisation

Table 1. RSM conditions of spray drying

Experiment N°	Inlet air temperature (°C)	Feed flow rate (L h^{-1})
1	150 (−1)	2 (–1)
2	210 (+1)	2 (–1)
3	150 (–1)	7 (+1)
4	210 (+1)	7 (+1)
5	137 (–1.41)	4.5 (0)
6	222 (+1.41)	4.5 (0)
7	180 (0)	1 (–1.41)
8	180 (0)	8 (+1.41)
9	180 (0)	4.5 (0)
10	180 (0)	4.5 (0)
11	180 (0)	4.5 (0)
12	180 (0)	4.5 (0)
13	180 (0)	4.5 (0)

Table 2. Physicochemical characterisation of whey

Parameter	M ± SD
pH	6.34 ± 0.2
Acidity (D°)	14.5 ± 1.5
Total dry matter (g L^{-1})	52.40 ± 1.13
Fat content (g L^{-1})	5.5 ± 0.05
Ash (g L^{-1})	5.28 ± 0.07
Proteins (g L^{-1})	7.73 ± 0.36
Density (kg L^{-1})	1.063
Colour parameters:	
L	52.23 ± 0.46
a	−18.70 ± 0.26
b	27.43 ± 0.05

M±SD: Mean and Standard Deviation

3.1. Moisture content

The moisture content (MC) of the powder varied from 2.12 to 9.92% (Fig. 2b). The highest MC was noted at 180 °C IAT and 8 L h^{-1} FFR. This high MC is probably due to the short contact time between liquid droplets and hot air, leading to a low heat and material transfer (Manickavasagan et al., 2015). According to the design, the linear and quadratic effects of feed flow rate (B), the quadratic effects of inlet air temperature (A), and the interaction between the two factors had a significant effect on moisture content ($P < 0.05$). According to Fig. 2b, the MC of whey powder is proportional to the FFR.

3.2. Yield

According to Fig. 2a, the yield rate varied from 0.00 to 40.04%. No whey powder was obtained at IAT 150 °C and FFR 7 L h^{-1}. A low yield rate can be explained by the stickiness and deposition

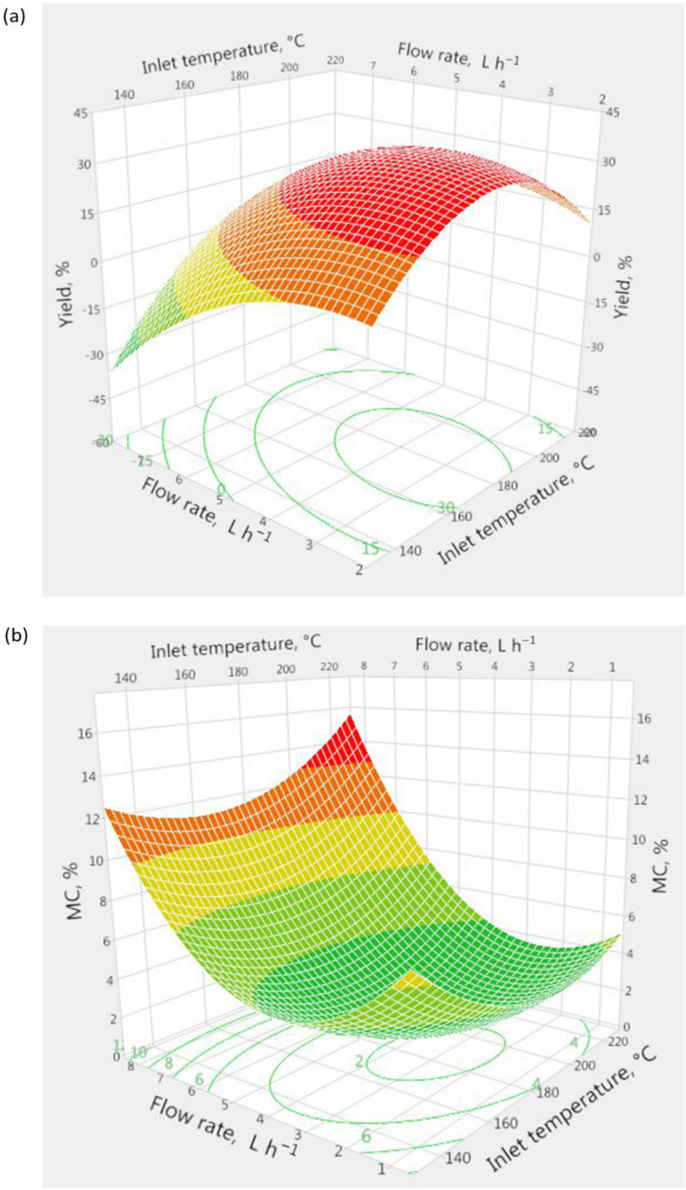

Fig. 2. Response surface plots for (a): yield, (b): moisture content, (c): bulk density, (d): tapped density, (e): dispersibility, and (f): outlet air temperature as a function of inlet air temperature and feed flow rate

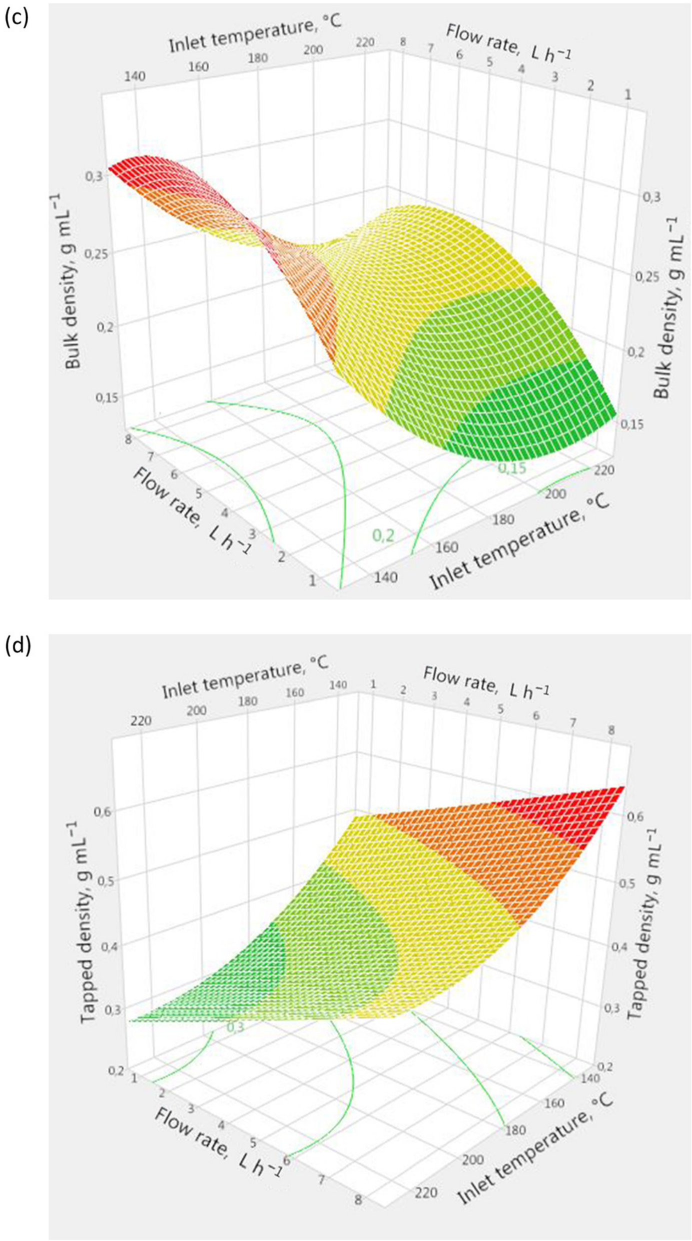

Fig. 2. continued

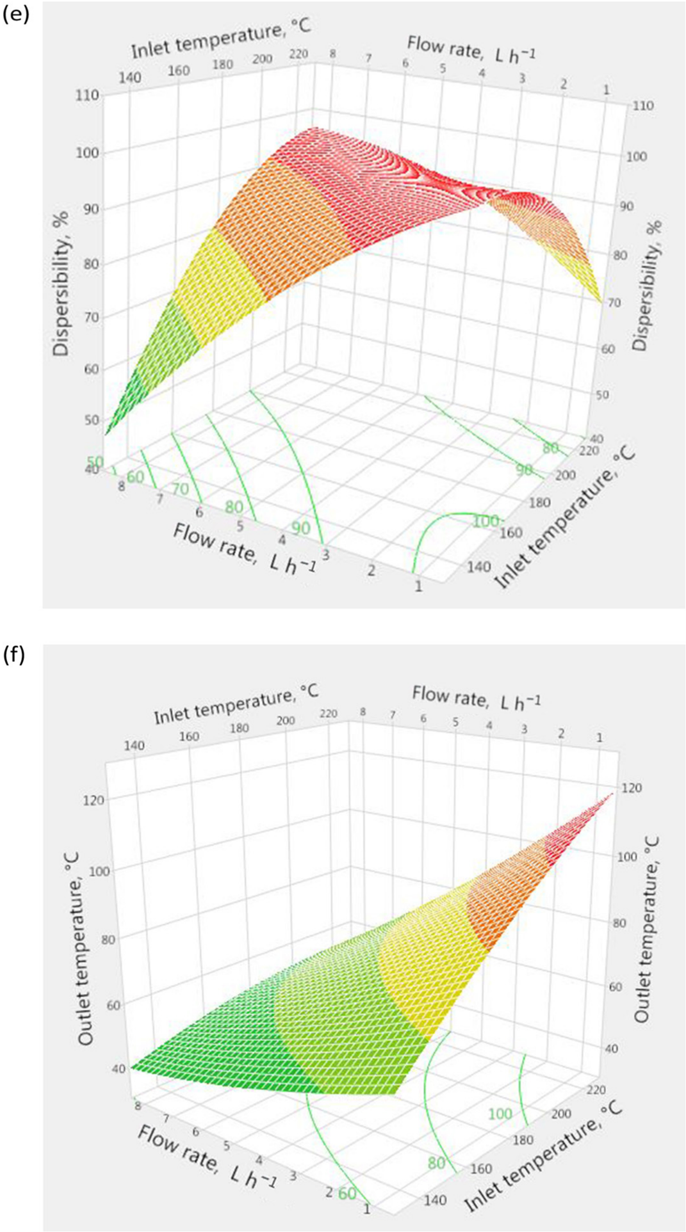

Fig. 2. continued

of powder particles on the walls of the drying chamber and the difficulty to collect these particles in the cyclone (Bhusari et al., 2014). Higher yield rate was noted for FFR of 4.5 L h^{-1} and IAT of 180 °C, which is probably due to the increase of the heat transfer efficiency. These results are lower than those obtained by Jafari et al. (2019). However, the increase of the FFR had a negative influence on the recovery of the extract in the cyclone. In addition, IAT had no significant effect (Table 3) on the yield rate ($P = 0.151$). Studies have shown similar results that the recovery rate of the powder was not significantly influenced by the IAT (Amiri-Rigi et al., 2011).

3.3. Bulk and tapped density

Figure 2 presents response surfaces of bulk and taped density of whey powder recuperated as a function of IAT and FFR. According to Fig. 2c, the BD of whey powder varied between 0.18 and 0.31 g mL^{-1}. These results are almost similar to those reported by Zouari et al. (2019). A low bulk density is due to a large volume and large particle size of powder (Bhusari et al., 2014). The IAT affects significantly the BD of whey powder ($P < 0.05$). The BD decreased with the increasing IAT, what was also reported by Saha et al. (2019). Figure 2c shows that the decrease in FFR generally caused a decrease in bulk density, due to the higher evaporation rate. A similar results were observed by Wang et al. (2015) for soy sauce powder. The tapped density (TD) of whey powder (Fig. 2d) varied from 0.32 to 0.55 g mL^{-1}, it was positively influenced by the FFR and negatively by the IAT.

3.4. Dispersibility

The dispersibility varied between 82.45 and 97.09% (Fig. 2a), which is within the range noted by Bhusari et al. (2014). According to the statistical analysis, the dispersibility was affected negatively by the feed flow rate ($P < 0.05$).

3.5. Outlet air temperature

According to Fig. 2f, the OAT values ranged from 42.4 to 96.3 °C, this variation was due to changes in the IAT values and the feed flow rate used. A low outlet air temperature was obtained when whey was dried at an IAT of 150 °C and a FFR of 7 L h^{-1}, whereas a very high outlet air temperature was noted at a very high IAT 210 °C and a low FFR 2 L h^{-1}.

The analysis of variance Table 3 shows a positive linear effect ($P < 0.05$) of the IAT on the OAT, while the FFR showed a negative linear effect. No quadratic impact was reported on the OAT ($P > 0.05$).

3.6. Optimisation

The conditions of the spray drying process have been optimised to obtain maximal yield, low MC, high dispersibility, high bulk density, and minimal tapped density. In order to avoid powder sticking and to have a low moisture content of the powder at the outlet cyclone, we have maximised the outlet air temperature. Desirability functions were used for the optimisation, the most desirable solutions for the optimal process conditions were found as follows: an inlet air temperature of 182 °C and feed flow rate of 3.2 L h^{-1}.

Figure 1b shows the image of the whey powder obtained after optimisation. The values of moisture, dispersibility, and solubility of whey powder under the optimum conditions were

Table 3. Effects of linear, quadratic, and interaction terms for each response variable treated by ANOVA

Source	Yield (%)		Moisture content (%)		Bulk density (g mL^{-1})		Tapped density (g mL^{-1})		Dispersibility (%)		Outlet temperature (°C)	
	Coeff	*P* value	Coeff	*P* value	Coeff	*P* value	Coeff	*P* value	Coeff	*P* value	Coeff	*P* value
Model	32.77	<0.0001*	2.160	<0.000*	0.24000	<0.0001	0.4100	<0.0001*	95.36	<0.0001*	70.05	<0.0001
A	1.88	0.151	−0.026	0.862	−0.02574	0.008*	−0.0597	0.034*	1.02	0.369	11.581	<0.0001*
B	−7.36	<0.0001*	2.334	<0.0001*	0.02043	0.020*	0.0532	0.051	−5.31	0.002*	−14.687	<0.0001*
A^2	−9.71	<0.0001*	0.771	<0.0001*	0.01544	0.051	0.0211	0.357	– 4.25	0.006*	−1.876	0.087
B^2	−9.17	<0.0001*	1.998	<0.000*	−0.01772	0.031*	0.0007	0.973	−2.86	0.031*	0.522	0.584
AB	4.43	0.031*	0.553	0.047*	0.0020	0.849	0.0018	0.961	4.88	0.026*	−2.35	0.088
R^2	95.86%		99%		87.69%		69.30%		87.50%		98.63%	

Coeff: Model coefficient; A: inlet air temperature (°C), B: feed flow rate (L h^{-1})

* Statistically significant values (*P* value <0.05)

1.80%, 99%, and 97.22%, respectively. Our values are comparable to the values published by other authors (Westergaad, 2010; Schuck et al., 2012). The wettability of optimised whey powder was higher than that indicated by Schuck et al. (2012). The Hausner index (HI) and Carr index (CI) expressing fluidity and cohesion of optimised whey powder were 1.70 and 41.37%, respectively. So, we can say that the powder obtained was very difficult to flow in accordance with the classification given by Reddy et al. (2014).

The quantity of water absorbed by the optimised whey powder was 16.10%, which classifies our powder as a hygroscopic product (Schuck et al., 2012). The caking degree (CD) of whey powder was 85.56%. According to Westergaad (2010), the powder is classified as, non-caking if the CD is less than 10%, slightly caking when the CD varies between 10.1 and 20%, highly caking if the CD is more than 20.1% and less than 50%, and extremely sticky if the CD is higher than 50.1%. Therefore, the optimised whey powder was classified as extremely caking powder. The commercial whey powder showed significant crystalline lactose peaks, while the optimised whey powder showed a lactose amorphous state (Fig. 3). Žolnere and Ciproviča (2019) reported that during drying of milk permeate and sweet and acid whey permeate, the lactose had an amorphous state instead of the crystalline form of α -monohydrate of lactose. The morphology of optimised and whey powder (Fig. 4) by SEM showed that the particles of optimised powder had different sizes, smooth and/or cracked into spherical shape, and agglomerated structure (Fig. 4a). This result is similar to those reported by Jafari et al. (2019). Indeed, the commercial powder has a crystalline form (Fig. 4b).

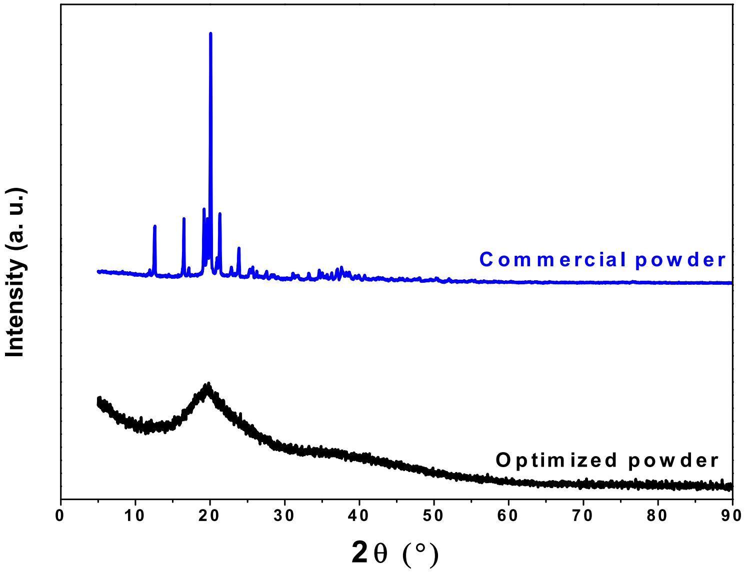

Fig. 3. X-ray diffraction patterns of whey powder

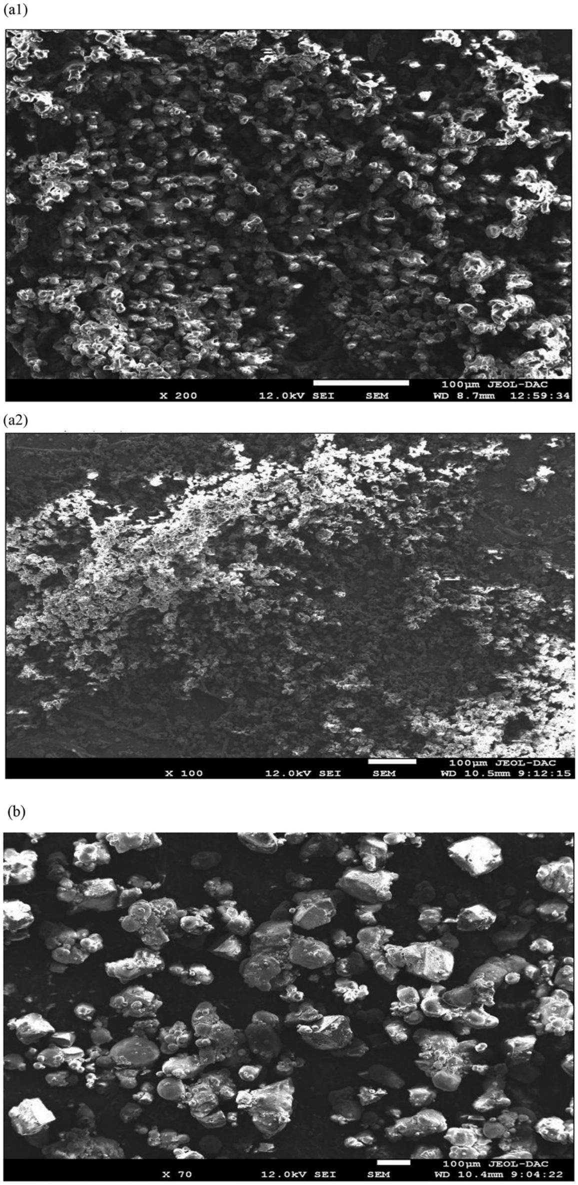

Fig. 4. The SEM of spray-dried whey powders (a1): whey powder obtained, × 200, (a2): optimised whey powder × 100, (b): commercial whey powder × 70

4. CONCLUSIONS

The results of this work showed that whey powder could be recovered without drying agent and without concentration by spray drying using mixed flow. All responses were principally influenced by the feed rate. Under the best spray drying conditions, i.e. inlet air temperature 182 °C and feed flow rate 3.2 L h^{-1}, the yield was lower than 50%, and the powder obtained was characterised by a water content of 1.80, solubility of 99, and dispersibility of 94.77% in accordance with the recommendations for a food powder. In this study, it was found that the wettability was higher than 120 s and the powder was very difficult to flow. In addition, the powder obtained was hygroscopic with high caking degree, having amorphous state, and different sizes of particles.

ABBREVIATIONS

IAT	Inlet air temperature
OAT	Outlet air temperature
FFR	Feed flow rate
MC	Moisture content
CD	Caking degree
BD	Bulk density
TD	Tapped density
CI	Carr index
DI	Dispersibility index
HR	Hausner ratio
RSM	Response surface methodology

ACKNOWLEDGMENTS

Authors gratefully acknowledge the cooperation of Dr. A. Boukhiar (University Boumerdes) and Dr. F. Moulai (University of Bejaia) in this study.

REFERENCES

Amiri-Rigi, A., Mohammadifar, M.A., Emam-Djomeh, Z., and Mohammadi, M. (2011). Response surface optimisation of spray dryer operational parameters for low-phenylalanine skim milk powder. *International Journal of Food Science & Technology*, 46(9): 1830–1839. https://doi.org/10.1111/j.1365-2621.2011.02688.x.

AOAC (2000). *Official methods of analysis*. Association of Official Analytical Chemists, Rockville, MD, USA.

Bhusari, S.N., Muzaffar, K., and Kumar, P. (2014). Effect of carrier agents on physical and microstructural properties of spray dried tamarind pulp powder. *Powder Technology*, 266: 354–364. https://doi.org/10.1016/j.powtec.2014.06.038.

Fischer, C. and Kleinschmidt, T. (2021). Valorisation of sweet whey by fermentation with mixed yoghurt starter cultures with focus on galactooligosaccharide synthesis. *International Dairy Journal*, 119: 105068. https://doi.org/10.1016/j.idairyj.2021.105068.

Ghanimah, M.A. (2018). Functional and technological aspects of whey powder and whey protein products. *International Journal of Dairy Technology*, 71(2): 454–459. https://doi.org/10.1111/1471-0307.12436.

Jafari, S.M., Masoudi, S., and Bahrami, A. (2019). A Taguchi approach production of spray-dried whey powder enriched with nanoencapsulated vitamin D_3. *Drying Technology*, 37(16): 2059–2071. https://doi.org/10.1080/07373937.2018.1552598.

Jiang, N., Dev Kumar, G., Chen, J., Mishra, A., and Mis Solval, K. (2020). Comparison of concurrent and mixed-flow spray drying on viability, growth kinetics and biofilm formation of *Lactobacillus rhamnosus* GG microencapsulated with fish gelatin and maltodextrin. *LWT – Food Science and Technology*, 124: 109200. https://doi.org/10.1016/j.lwt.2020.109200.

Kheroufi, A., Brassesco, M.E., Campos, D.A., Mouzai, A., Boughellouta, H., and Pintado, M.E. (2022). Whey protein-derived peptides: the impact of chicken pepsin hydrolysis upon whey proteins concentrate on their biological and technological properties. *International Dairy Journal*, 134: 105442. https://doi.org/10.1016/j.idairyj.2022.105442.

Manickavasagan, A., Thangavel, K., Dev, S.R.S., Delfiya, D.S.A., Nambi, E., Orsat, V., and Raghavan, G.S.V. (2015). Physicochemical characteristics of date powder produced in a pilot-scale spray dryer. *Drying Technology*, 33(9): 1114–1123. https://doi.org/10.1080/07373937.2015.1014045.

Reddy, R.S., Ramachandra, C.T., Hiregoudar, S., Nidoni, U., Ram, J., and Kammar, M. (2014). Influence of processing conditions on functional and reconstitution properties of nadeem made from Osmanabadi goat milk by spray drying. *Small Ruminant Research*, 119(1): 130–137. https://doi.org/10.1016/j.smallrumres.2014.01.013.

Saha, D., Nanda, S.K., and Yadav, D.N. (2019). Optimization of spray drying process parameters for production of groundnut milk powder. *Powder Technology*, 355: 417–424. https://doi.org/10.1016/j.powtec.2019.07.066.

Samsu, Z.A. and Zahir, A.Z.M. (2020). Production of oil palm milk powder by spray drying technique. *Materials Today. Proceedings*, 31(Part1): 306–312. https://doi.org/10.1016/j.matpr.2020.06.015.

Schuck, P., Dolivet, A., and Jeantet, R. (2012). *Analytical methods for food and dairy powders*. Wiley, Hoboken, NJ, p. 239.

Selvamuthukumaran, M. (2019). *Spray drying of dairy and cereal food products. Handbook on spray drying applications for food industries*. CRC Press, pp. 69–86. https://doi.org/10.1201/9780429055133-3.

Seth, D., Mishra, H.N., and Deka, S.C. (2017). Functional and reconstitution properties of spray-dried sweetened yogurt powder as influenced by processing conditions. *International Journal of Food Properties*, 20(7): 1603–1611. https://doi.org/10.1080/10942912.2016.1214965.

Wang, W., Dufour, C., and Zhou, W. (2015). Impacts of spray-drying conditions on the physicochemical properties of soy sauce powders using maltodextrin as auxiliary drying carrier. *CyTA – Journal of Food*, 13(4): 548–555. https://doi.org/10.1080/19476337.2015.1014430.

Westergaad, V. (2010). *Milk powder technology evaporation and spray drying*, 5th ed. GEA Niro, Copenhagen, p. 337.

Yadav, J.S.S., Yan, S., Pilli, S., Kumar, L., Tyagi, R.D., and Surampalli, R.Y. (2015). Cheese whey. A potential resource to transform into bioprotein, functional/nutritional proteins and bioactive peptides. *Biotechnology Advances*, 33(6): 756–774. https://doi.org/10.1016/j.biotechadv.2015.07.002.

Zhu, X., Yuan, X., Zhang, Y., Liu, H., Wang, J., and Sun, B. (2022). The global concern of food security during the COVID-19 pandemic. Impacts and perspectives on food security. *Food Chemistry*, 370: 130830. https://doi.org/10.1016/j.foodchem.2021.130830.

Žolnere, K. and Ciproviča, I. (2019). The study of physical properties of spray dried whey and milk permeates lactose. *Agronomy Research*, 17(S2): 1501–1510. https://doi.org/10.15159/AR.19.054.

Zouari, A., Perrone, Í.T., Schuck, P., Gaucheron, F., Dolivet, A., Attia, H., and Ayadi, M.A. (2019). Effect of outlet drying temperature and milk fat content on the physicochemical characteristics of spray-dried camel milk powder. *Drying Technology*, 37(13): 1615–1624. https://doi.org/10.1080/07373937.2018.1526189.