

STUDY OF TECHNOLOGICAL PROPERTIES OF COMMERCIAL SWEET WHEY AND WHEY PERMEATE POWDERS

Alexey Lodygin¹, Ivan Evdokimov¹, Irina Kulikova^{1*}, Alexander Gridin¹, Kirill Sorokin¹

¹Faculty of Food Engineering and Biotechnology, North-Caucasus Federal University, Pushkin 1, 355017 Stavropol, Russian Federation

*e-mail: kik-st@yandex.ru

Abstract

Sweet whey and whey/milk permeates are raw materials that are formed during milk processing on high protein products: cheese, quark, and dairy protein concentrates. The main feature of whey and permeate dry substance composition is prevalent lactose content. It allows to use of these raw materials for different fermentation processes. On the other hand, high lactose content and availability of nitrogen compounds and other nutrients cause instability of liquid whey and permeate during storage and transportation. That is why sometimes usage of whey and permeate spray-dried powders is preferable. Our researchers aim to compare the main characteristics of whey and permeate powders as potential raw materials for different products, including fermented dairy products.

Duplicate commercially packaged samples of partially demineralized sweet whey and whey permeate powders by local dairy manufacturers have been used for research. The samples' chemical composition (lactose, protein, ash, fat) and physical parameters (bulk density, insolubility index, and wettability) have been determined by GOST and ISO methods. The particle size distribution in the samples has been measured by laser diffraction analysis. The research has been carried out in the scientific laboratory of "Food and Industrial Biotechnology" of North Caucasus Federal University.

The results have shown that the samples' component composition is compliant with typical commercial whey and permeate powders. Other samples' characteristics (bulk density, insolubility index, particle size distributions) are very close. The main

differences have been observed in wettability and water-absorbing of the powders, especially whey demineralized permeate. It could result from the lower lactose crystallization of the powder. Its feature should be factored into when the powders are used.

Thus, the results confirmed that lactose has a great influence on the physicochemical and structural properties of whey and permeate powder, as it's present in the product as a continuous phase and can absorb moisture from the atmosphere during storage easily. It could be a reason for the forming of highly viscous liquid, clumping, and caking of the product. These features should be taken into account when the powders are chosen as raw materials for different applications.

Key words: Sweet whey powder, Permeate powder, Bulk density, Insolubility index, Particle size distributions.

1. Introduction

Today the role of dairy by-product processing is increasing significantly. It's caused by the necessity to solve environmental problems and improve the economic performance of milk processing by manufacturing added-value products.

Sweet whey is an important by-product of hard, semi-hard, and soft cheese production. Whey has a pH of 5.9 - 6.6, and contents with about: 50% of total milk solids, including lactose, milk protein, minerals, all water-soluble, and part of fat-soluble vitamins, that are presented in milk (Table 1) [12].

Table 1. Average dry matter composition of sweet whey of sweet whey and sweet whey permeate [12]

Composition	Sweet whey		Sweet whey permeate	
	Average	Span	Average	Span
Total protein, % w/w (in DS)	0.41	0.16 - 0.58	-	-
Non-protein nitrogen, % (in DS)	0.41	0.16 - 0.58	0.69	0.48 - 1.38
Fat, % w/w (in DS)	3.46	0.94 - 5.26	-	-
Lactates, % w/w (in DS)	2.07	0.00 - 4.01	3.45	no data
Lactose, % w/w (in DS)	75.0	66.0 - 83.0	84.0	74.0 - 91.0
Ash, % w/w (in DS)	8.45	5.00 - 11.4	9.38	8.26 - 10.7
Dry solids (DS) of the liquid product, %	6.2	5.16 - 7.00	4.85	3.4 - 5.8

Typically, the protein fraction is 20% of whey dry matter and includes about 50% of β -lactoglobulin, 25% of α -lactalbumin, and 25% of other proteins [14]. Carbohydrates are the biggest part of whey dry matter, or about 70%. They are represented by the disaccharide lactose mainly (up to 90%), lactose hydrolysis products (glucose and galactose), and some other minor oligosaccharides. Whey minerals include the cations of potassium, sodium, calcium, magnesium, and iron, and anions of citrates, phosphates, chlorides, and some trace elements [18].

Whey is a high nutrient value by-product that could be used in many applications. For example, whey has long been used in the production of a lot of food products: baked products, confectionaries, desserts, ice cream, and others. On the other hand, sweet whey could be processed on protein ingredients: whey protein concentrates and isolates (WPC and WPI). As ultrafiltration removes proteins, permeate dry matter (Table 1) consists of lactose (above 85%) and other low-molecular compounds: minerals, non-protein nitrogen, etc.

Permeate is a high nutrient value by-product as well and it could replace sweet whey in many applications. For example, permeate is a suitable carbohydrate substrate for different fermentation processes [11]. However, high lactose content and availability of nitrogen compounds and other nutrients could cause instability of liquid whey and permeate during storage and transportation. That is why the usage of whey and permeate spray-dried powders is preferable.

The quality of whey and permeate powders is determined by several parameters including the component composition, nutritional value, and of course its safety. Characteristics that determine the design and operating modes of technological equipment for transportation, packaging, dosing, and other technological operations required when using a particular powder: particles' size and porosity, bulk density, fluidity, wettability, dispersibility, solubility, hygroscopicity, thermal stability, etc. are equally important [16]. The specified parameters are the

most significant characteristics of the ingredients. For example, the insolubility index characterizes the measure of the final state to which the powder components can be introduced into a solution or stable suspension. Poorly soluble powders cause processing difficulties and lead to significant recovery losses. Bulk density is also an economically and functionally important characteristic since it determines the requirements for materials and the choice of equipment for the powders' processing.

Several indicators should be considered when choosing technological modes for the processing of dry powders additionally. Hygroscopicity is important in determining the sensitivity of a dry product to fluctuations in humidity and temperature during storage and processing. Wettability, dispersibility, and immersion of particles are mandatory quality characteristics of instant powders. Flowability, which shows how freely the powder particles move relative to each other, is of determinative importance for the design of pneumatic transport, the device for filling and emptying hoppers, dosing, mixing, and conditioning powders [13].

Our study aims to compare the main characteristics of whey and permeate powders as potential raw materials for different products, including fermented dairy products.

2. Materials and Methods

The research has been carried out in the scientific laboratory of "Food and Industrial Biotechnology" of North Caucasus Federal University. Duplicate commercially packaged samples of partially demineralized sweet whey and whey permeate powders by local dairy manufacturers have been used for research.

The samples' composition, lactose, protein, fat, and ash concentrations have been measured using GOST methods [3, 5, and 6]. Total moisture was determined by Karl-Fischer titration using the GOST method [1], and free moisture content by using the GOST method [2].

The samples' physical parameters, bulk density, insolubility index, and wettability have been determined by different GOST methods [4, 7, and 9].

The particle size distribution in the samples has been measured by laser diffraction analysis according to Smoczyński [17].

Lactose crystallinity (LC, %) was calculated according to the formula described by O'Donoghue *et al.*, [13]:

$$LC = \frac{BWL * 19}{L} \times 100$$

Where: BWL is the Bound Water Content in the lactose (g /kg) and L is the Lactose content (g /kg).

The BWL was calculated according to the following formula:

$$BWL = TW - FW - (0.005 \times WPC) - (0.0155 \times MSSC)$$

Where: TW is total water content (g /kg), FW is free water content (g /kg), WPC is whey protein content (g/kg), and MSSC is milk salt solution content (g/kg).

The capability of the powders to humidity absorption has been estimated by the modified method described by [8]. The vials with powder samples (~5.0 g) are placed in a desiccator at 85% RH. The samples have been equilibrated and weighed at regular intervals until a constant weight has been observed. Humidity absorption (HA) is calculated by using the following formula:

$$HA = \frac{w_2 - w_0}{w_1} \times 100$$

Where: w_0 is vial weight (g), w_1 is sample weight (g), and w_2 is the weight of the vial after equilibration (g).

The Hausner ratio (HR) and The Carr index have been determined to describe the flow properties of powder samples. Hausner ratio is calculated by dividing the

tapped density by the loose poured bulk density [15]. The Carr index is defined as:

$$CI = (\rho_t - \rho_b) / \rho_t * 100$$

Where: ρ_t is the tapped bulk density (100-taps), and ρ_b is the loose bulk density.

All analyses were carried out in at least duplicate.

Statistical analysis was carried out by subjecting data sets to one-way ANOVA with a least significant difference (LSD) test using SPSS for Windows Regression Models statistical analysis package. A level of confidence of $P \leq 0.05$ was used.

3. Results and Discussion

Samples' chemical composition is shown in Table 2. Predictably, the whey powder consists of the lowest lactose (78.3%) and the highest total protein 11.52% and demineralized permeate powder consists of the highest lactose (92.3%) and the lowest total protein (3.08%). Considering the results described in Kravstov [12], it's likely that the figure of the protein content of permeate powders represents low-molecular peptides and non-protein nitrogen content as a true protein should not observed in the permeate powder.

The fat content of the sweet whey powder sample (11.52%) is typical for the same product. Fat traces' presence in the permeate powders could be related to the precision of the method that has been used for the fat determination.

It is to be noticed that the ash content of all samples is lower than typical values. The samples of demineralized permeate powder consist of 0.51% ash in DS that compliances with the demineralization level of more than 90%. The second permeate and whey powder samples consist of 5.83%, and 4.72% ash in DS that compliances with the demineralization level about 25% [6]. The reason for the lower ash level of

Table 2. Powder composition

Parameters	Powder samples					
	Whey powder		Permeate powder		Demineralized permeate powder	
	Average	Randoms	Average	Randoms	Average	Randoms
Total protein content, mass%	11.02	(± 0.22)	3.86	(± 0.22)	3.08	(± 0.22)
Fat content, mass%	0.2	(± 0.15)	Less 0.10	(± 0.15)	Less 0.10	(± 0.15)
Lactose content, mass%	77.3	(± 0.80)	88.5	(± 0.80)	92.3	(± 0.80)
Ash content, mass%	4.53	(± 0.04)	4.6	(± 0.04)	0.54	(± 0.04)
Free water content, %	3.33	(± 0.10)	2.62	(± 0.10)	1.68	(± 0.10)
Total water content, %	6.26	(± 0.16)	6.56	(± 0.16)	4.68	(± 0.16)
Dry matter content, %	96.67	(± 0.50)	97.38	(± 0.50)	98.32	(± 0.50)
Lactose crystallization level*, %	72.02	(± 0.04)	83.02	(± 0.01)	67.73	(± 0.02)

Legend: *Calculated value.

the whey and permeate powders could be using of the nanofiltration process to concentrate the ones before evaporation and spray drying.

The results have shown that most of the lactose in the samples is in the crystalline form of α -lactose monohydrate (Table 3). It evidences that whey and permeate powder production includes the crystallization step before spray drying, in which the majority of amorphous lactose present is converted into the more stable, crystalline form. However, permeate powder with a demineralization level of 90% contained more non-crystalline lactose compared with whey and whey permeate powders.

The particle size distribution of the powders is shown in Figure 1.

In all tested powders, the most of particles are between 250 and 63 μm . However, in the whey powder, most powder particles are between 180 and 100 μm , compared with permeate samples, which mostly contained particles in the range from 150 to 75 μm .

According to O'Donoghue *et al.*, [13], particle size can affect powder moisture absorption as water is on the particle surface mainly. As a result, the smaller particles have a relatively larger exchange surface for water absorption.

The tested samples have very close particle size distribution. However, permeates have better moisture absorption and wettability, in which, higher values have been obtained for demineralized permeate powder, that predominantly due to the more amorphous lactose content (46.9%) of the powder.

It has been found that the whey powder is characterized by the lowest bulk loose density and tapped density (Table 3). The bulk density of permeate powders is similar and ranges from 0.759 to 0.791 g/cm^3 . The highest percentage decrement in density after tapping has been found in demineralized permeate powder.

Table 3 presents the values of Hausner's ratio and Carr's index for the tested powders, compared with the classification proposed by Jinapong *et al.*, [10].

Table 3. Functional characteristics of tested powders

Parameters	Powder samples					
	Whey powder		Permeate powder		Demineralized permeate powder	
	Average	Randoms	Average	Randoms	Average	Randoms
Insolubility index, cm^3	0.05	(± 0.01)	0.05	(± 0.01)	0.05	(± 0.01)
Wettability, sec	40.00	(± 0.15)	15.00	(± 0.32)	6.00	(± 0.05)
Water absorption, %	13.50	(± 0.17)	19.30	(± 0.32)	27.00	(± 0.14)
Bulk density, g/cm^3	0.534	($\pm 7.8\%$)	0.791	($\pm 7.8\%$)	0.759	($\pm 7.8\%$)
Bulk density (100-taps), g/cm^3	0.660	($\pm 7.8\%$)	0.939	($\pm 7.8\%$)	0.911	($\pm 7.8\%$)
Bulk density (625-taps), g/cm^3	0.697	($\pm 7.8\%$)	0.969	($\pm 7.8\%$)	0.982	($\pm 7.8\%$)
Hausner ratios* (100-taps)	1.2359	(± 0.021)	1.1871	(± 0.015)	1.200	(± 0.010)
Carr's Index* (100-taps)	19.09	(± 0.01)	15.76	(± 0.00)	16.69	(± 0.03)

Legend: *Calculated value.

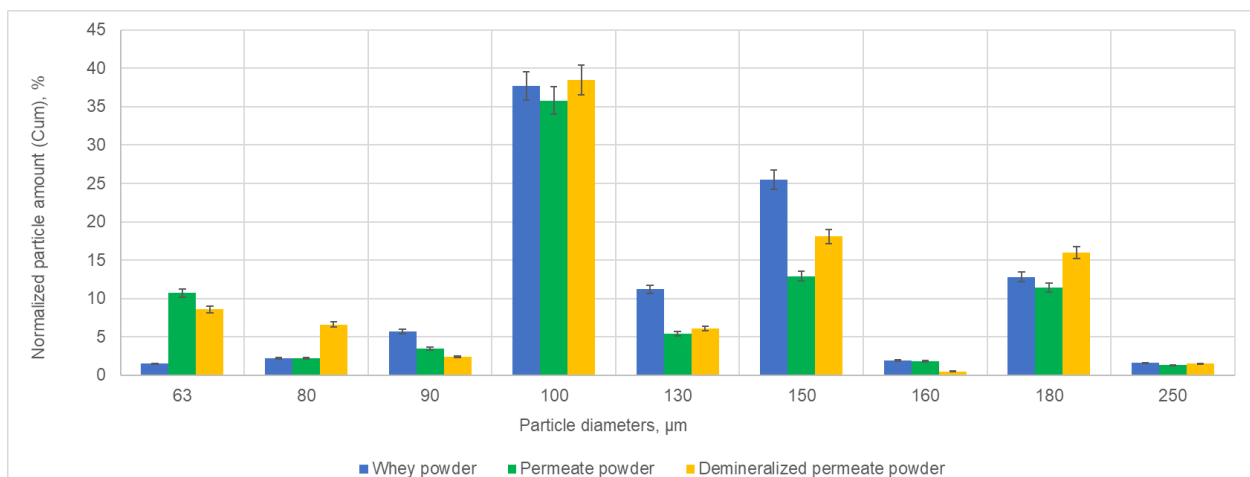


Figure 1. Particles' size distribution of the powders

Based on this classification it has been found that all tested powders are characterized by intermediate cohesiveness and good flowability.

Generally, our results have confirmed the conclusion by other authors as O'Donoghue *et al.*, [13], and Sharma *et al.*, [16], about the effect of powder composition and lactose crystallization on powder properties.

Acknowledgment

The study was funded by a grant from the Ministry of Science and Higher Education of the Russian Federation "Study of the mechanisms of interaction of lactic acid microorganisms, lactose-fermenting yeast and biologically active substances during microencapsulation of various fractions of microbiota" by Decree of the Government of the Russian Federation No. 220 in the form of a subsidy from the federal budget for state support of scientific research conducted under the guidance of leading scientists in Russian educational institutions of higher education, scientific institutions and state scientific centers of the Russian Federation (IX turn), Agreement No. 075-15-2022-1129 from 01.07.2022.

4. Conclusions

- The results have shown that the samples' component composition is compliant with typical commercial whey and permeate powders. Other samples' characteristics (bulk density, insolubility index, particle size distributions) are very close. The main differences have been observed in wettability and water-absorbing of the powders, especially whey demineralized permeate. It could result from the lower lactose crystallization of the powder.

- Thus, the results confirmed that lactose has a great influence on the physicochemical and structural properties of whey and permeate powder, as it's present in the product as a continuous phase and can absorb moisture from the atmosphere during storage easily. It could be a reason for the forming of highly viscous liquid, clumping, and caking of the product. These features should be considered when the powders are chosen as raw materials for different applications.

5. References

- [1] EASC. (2014). *GOST 12779-2014: Lactose. Determination of water content. Karl Fischer method* (in Russian). <URL:https://docs.cntd.ru/document/1200112307. Accessed 25 June 2023.
- [2] EASC. (2009). *GOST 29246: Dry canned milk. Methods for determination of moisture* (in Russian). <URL:https://internet-law.ru/gosts/gost/38497/. Accessed 25 June 2023.
- [3] EASC. (2009). *GOST 29247-91: Canned milk. Methods for determination of fat* (in Russian). <URL:https://internet-law.ru/gosts/gost/10275/. Accessed 25 June 2023.
- [4] EASC. (1995). *GOST 30305.4-95: Dry dairy products. The procedure of measurement of solubility index* (in Russian). <URL:https://online.zakon.kz/Document/?doc_id=30548734. Accessed 25 June 2023.
- [5] EASC. (2018). *GOST 34454-2018: Dairy products. Determination of protein content by the Kjeldahl method* (in Russian). <URL:https://files.stroyinf.ru/Data/697/69710.pdf. Accessed 25 June 2023.
- [6] EASC. (2016). *GOST R 56833-2015: Demineralized dairy whey. Specifications* (in Russian). <URL:https://docs.cntd.ru/document/1200129055. Accessed 25 June 2023.
- [7] EASC. (2010). *GOST R ISO 8967-2010: Dried milk and dried milk products. Determination of bulk density* (in Russian). <URL:https://meganorm.ru/Data/504/50440.pdf. Accessed 25 June 2023.
- [8] Griffin J., Grady J. (2006). *Textbook of pharmaceutical medicine*. Blackwell Publishing, New Jersey, USA.
- [9] ISO. (2014). *ISO/TS 17758:2014: Instant dried milk - Determination of the dispersibility and wettability*. ISO, Geneva, Switzerland.
- [10] Jinapong N., Supphantharika M., Jamnong P. (2008). *Production of instant soymilk powders by ultrafiltration, spray drying, and fluidized bed agglomeration*. Journal of Food Engineering, 84, pp. 194-205.
- [11] Khramtsov V. A. (2019). *Innovative Solutions in Milk Whey Production* (in Russian). Food Processing Techniques and Technology, 48, pp. 5-15.
- [12] Kravtsov V. (2021). *Development of lactose-containing raw material purification process* (in Russian). PhD thesis, NCSU, Stavropol, Russia.
- [13] O'Donoghue L., Haque K., Kennedy D., Laffir F., Hogan S., O'Mahony J., Murphy E. (2019). *Influence of particle size on the physicochemical properties and stickiness of dairy powders*. International Dairy Journal, 98. <URL:https://doi.org/10.1016/j.idairyj.2019.07.002. Accessed 20 June 2023.
- [14] Ostroumova T. A., Shitov A. P. (2009). *Influence of seasonal changes in milk on the formation of soft cheeses* (in Russian). Food Processing Techniques and Technology, (2), pp. 60-63.
- [15] Saw Horng Y., Davies C., Paterson A., Jones J. (2015). *Correlation between Powder Flow Properties Measured by Shear Testing and Hausner Ratio*. Procedia Engineering, 102, pp. 218-225.
- [16] Sharma A., Jana A., Chavan R. (2012). *Functionality of Milk Powders and Milk-Based Powders for End Use Applications - A Review*. Comprehensive Reviews in Food Science and Food Safety, 11. 10.1111/j.1541-4337.2012.00199.x <URL:https://doi.org/10.1111/j.1541-4337.2012.00199.x. Accessed 20 June 2023.
- [17] Smoczyński M. (2020). *Fractal analysis of the microstructure of milk powders produced at various temperatures*. J. Food Sci. Technol., 57, pp. 2303-2309.
- [18] Tepel A. (2012). *Chemistry and physics of milk* (in Russian). Profession, Moscow, Russia.