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# Effects of storage conditions on milk powder properties

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#### **ABSTRACT**

Studies on the storage stability of milk powder are currently fragmented and mainly affect only the area of above-zero temperatures. At the same time, there are no studies that consider the load factor when milk powder is stored in bags on a pallet. The purpose of this study was to identify the influence of various factors of industrial storage (temperature, height or layer number, and time) on the change in quality and technological properties of powdered dairy products. We placed skim milk powder (SMP) and whole milk powder (WMP) in 10  $\times$  14  $\times$  2 cm resealable plastic bags on a model stand simulating an industrial layout on pallets. The samples were stored for 18 mo at temperatures  $-30 \pm 1^{\circ}$ C, 6  $\pm$  1°C, and 25  $\pm$  3°C and 40 to 80% relative humidity. Samples from the control (0), 5, and 10 (lower) layers of pallets were selected for analysis on 0, 3, 6, 9, 12, 15, and 18 mo of storage for each of the temperatures. As a result, we did not detect any changes in the storage process for water activity and mass fraction of moisture. The particle size distribution of all the SMP and WMP samples changed over time. The greatest changes were observed in the WMP samples placed on the 10th layer of pallets at  $25 \pm 3$ °C, from 0 to 18 mo of storage, the mean particle size (D[4,3]) increased from 120 to 258 μm (90% of all sample particle sizes ranging from 209 to 559  $\mu$ m). We found significant clumping in the WMP samples (lumps up to 5 cm), correlating with the layer and storage time. The contact angle of the samples increased from  $17^{\circ}$  (SMP) and  $53^{\circ}$  (WMP) to  $40^{\circ}$  and 71°, respectively. The insolubility index and titratable acidity did not change only in the SMP samples stored with no load applied at  $-30 \pm 1^{\circ}$ C and  $6 \pm 1^{\circ}$ C. The heat stability of all samples stored at 25  $\pm$  3°C showed the lowest values. The data obtained allowed us to rank the factors as "layer - time - temperature." Only the temperature of  $25 \pm 3^{\circ}$ C caused critical changes in the product properties. Thus, the possibility of industrial storage of the product for up to 15 mo over the entire temperature range is confirmed.

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#### INTRODUCTION

Milk and other dairy products are important nutrients for people. However, raw milk production is often subject to seasonality, which makes it difficult to ensure the continuity of production (Galstyan et al., 2019; Felfoul et al., 2021; Timlin et al., 2021). In such conditions the solution to this problem is the processing of milk powder, which contains all the necessary nutrients, such as proteins (casein and whey proteins), fat, lactose, fat-soluble, and water-soluble vitamins, as well as minerals (Skanderby et al., 2009; Radaeva et al., 2019). In addition, milk powder is more convenient for transportation and storage compared with raw milk. All of the above served as prerequisites for the widespread distribution of milk powder in the food industry and among the population. The food industry uses milk powder (both as is and as remade reconstituted milk) to produce dairy products (Masum et al., 2020b; Rozhkova and Olentsova, 2020; Cândido de Souza et al., 2021), and it is used in confectionery (Bianchi et al., 2021; Yadav et al., 2021), bakery (Kamali Rousta et al., 2021; Ergashovna et al., 2022), meat (Bianco et al., 2022), and other sectors of the food industry. For the consumer, milk powder is a ready-made food product that can fully replace raw milk in conditions of its shortage. Thus, milk powder contributes to ensuring food security especially in terms of the availability of food products regardless of the region of residence of a citizen, taking into account the established structure and tradition of nutrition (Zhang et al., 2020; Hernández et al., 2021; Rodnina, 2022), which is one of the priorities of state policy.

Milk powder quality and safety properties can be divided into 2 groups: rated values specified in regulations, and extra values introduced to detail various technological properties. The physical, chemical, organoleptic, and microbiological properties are rated (Council of the European Union, 2002, 2013; GOST 33629-2015, 2015; TR TS 033/2013, 2013).

No less important indicators are the extra functional and technological properties of milk powder, such as

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water activity  $(\mathbf{a}_{\mathbf{w}})$ , particle size distribution, caking, rehydration, and heat stability. Water activity is the key factor of milk powder stability as it determines the glass transition temperature  $(\mathbf{T}_{g})$ , which controls, for example, caking, clumping, and lactose crystallization properties (Thomsen et al., 2005; Roos, 2010; Silalai and Roos, 2010; Fournaise et al., 2020). In addition, during storage, the solubility decrease rate, fat oxidation, and nonenzymatic browning (Thomsen et al., 2005; Zhou and Langrish, 2021; Maidannyk et al., 2022) greatly depend on a<sub>w</sub>. The particle size distribution in milk powder determines its appearance (Kim et al., 2005; Han et al., 2021), rehydration characteristics (O'Donoghue et al., 2019; d'Almeida Francisquini et al., 2020), surface reactivity (Carpin et al., 2017; O'Donoghue et al., 2019), and flowability (Kosasih et al., 2016; Ding et al., 2020). Rehydration characteristics describe the milk powder behavior during reconstruction process. Key property is solubility: poorly soluble powders make processing difficult and lead to higher production costs. Solubility also controls the quality of reconstituted milk, namely, the ability of milk powder components to form a stable suspension (Morr et al., 1985; Selomulya and Fang, 2013; Maidannyk et al., 2022). The solubility of spray-dried milk powder exceeds 99%, whereas roller-dried milk powder is about 85% (Tamime, 2009). The heat stability of milk determines the reconstituted milk resistance to heating without significant coagulation or gelation (Singh, 2004; Gilmanov et al., 2020). It becomes especially important in the production of recombined dairy products that are sterilized. If the heat stability is insufficient, the protein in the product coagulates and precipitates during or immediately after sterilization (Lin et al., 2018; Singh et al., 2019; Wu et al., 2022).

These milk powder properties mostly depend on the raw material quality and the processing technology. However, even during storage, negative changes may occur since elevated temperature and relative humidity (RH) stimulates various chemical reactions and physical processes. Milk powder can be stored in a wide range of above-zero temperatures and humidity. Nevertheless, Russian regulatory standards recommend 0 to 10°C storage temperature and RH below 85% (Russian National Inspectorate, 1987). In worldwide practice, the acceptable storage temperature is up to 25°C with RH below 65% (US Dairy Export Council, n.d.). To date, scientists of the world (Kosasih et al., 2016; Smith et al., 2016; Fan et al., 2018; Ho et al., 2019, 2021; Masum et al., 2020a; Phosanam et al., 2020a, 2021; Fialho et al., 2021; Murayama et al., 2021) have studied the effects of elevated, above-zero temperatures and various humidity levels during storage, which in general

allowed us to form an understanding of the negative processes occurring in milk powder storage. However, the below-zero temperature range was not properly studied and only a few papers on this subject have been found. For example, Mistry and Pulgar (1996) noted a decrease in skim milk powder solubility at  $-20^{\circ}$ C on 105 d of storage. Galstyan et al. (2019) studied whole milk powder in the  $-20^{\circ}$ C to  $20^{\circ}$ C temperature range. No important changes in milk powder properties were found except for slight sticking. Obviously, the presented studies are not sufficient to understand the processes occurring in low temperature storage and their impact on the technological and physical properties of milk powder. The most common industrial method of milk powder storage and transportation is placing bags on wooden pallets, 3 in a layer stacked up to 10 layers. This results in another factor that affects the product: the load applied to the bag. The lower the layer, the greater the impact on it. We have not been able to find studies of the load effects during storage on the functional and technological properties of milk powder. In this study we investigated the effect of various temperature conditions of industrial storage on the changes in the functional and technological properties of milk powder produced in Russia. We also intended to confirm and update the data available in the literature and provide recommendations for milk powder storage in an extended temperature range.

### **MATERIALS AND METHODS**

Because no human or animal subjects were used, this analysis did not require approval by an Institutional Animal Care and Use Committee or Institutional Review Board.

#### Experimental Design

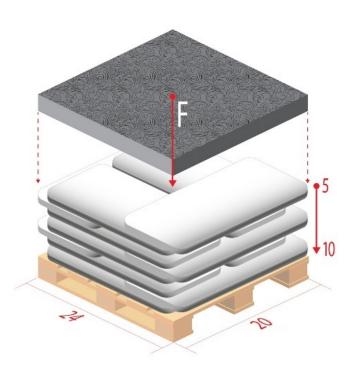
We obtained 64 samples of skim milk powder (SMP) and 47 samples of whole milk powder (WMP) produced in Russia from several different manufacturers between February and October 2019. The average physical and chemical properties of all samples presented by the manufacturers are shown in Table 1. The samples were sealed in resealable plastic airtight bags made of foodgrade high-pressure performance polyethylene (similar to food film liner bags by GOST 19360-74, 1975),  $10 \times 14 \times 2$  cm,  $80 \mu \text{m}$  density,  $0.5 \text{ g/m}^2 \cdot 24 \text{ h}$  water vapor permeability and placed on the storage simulator (Figure 1). We stacked the bags in 5 layers of 3 bags each, imitating the common industrial arrangement on pallets. At the same time, the empty layers were simulated by 2.4 kg weights. The bags were stored for

**Table 1.** Statistical analysis of the milk powder sample properties<sup>1</sup>

		P	Product	
Property	Value	SMP (n = 64)	WMP (n = 47)	
Moisture content, %	Min	3.2	3.3	
	Max	4.7	3.9	
	$Avg. \pm SEM$	$3.9 \pm 0.09$	$3.6 \pm 0.04$	
Fat, %	Min	0.42	26.0	
	Max	0.49	29.0	
	$Avg. \pm SEM$	$0.45 \pm 0.005$	$27.3 \pm 0.25$	
Protein, %	Min	33.1	23.05	
	Max	36.7	27.28	
	$Avg. \pm SEM$	$35.1 \pm 0.2$	$25.1 \pm 0.3$	
Lactose, %	Min	49.6	32.2	
	Max	54.5	38.5	
	$Avg. \pm SEM$	$52.3 \pm 0.35$	$35.3 \pm 0.48$	
Acidity, °T	Min	17.1	17.1	
	Max	19.5	19.3	
	Avg. $\pm$ SEM	$18.3 \pm 0.14$	$18.1 \pm 0.12$	
Insolubility index, mL	~	Less than 0.5	Less than 0.5	

 $<sup>^{1}</sup>$ SMP = skim milk powder; WMP = whole milk powder; n = number of samples, Min = minimum; Max = maximum; Avg. = average.

18 mo at  $-30 \pm 1^{\circ}$ C,  $6 \pm 1^{\circ}$ C, and  $25 \pm 3^{\circ}$ C and 40 to 80% RH. The reference samples were packed and stored under similar conditions but with no load applied. The sample codes are listed in Table 2. The frequency of measurements was 0, 3, 6, 9, 12, 15, and 18 mo.



**Figure 1.** The visualization of the experiment. Resealable plastic bags with milk powder are packed according to the most common palette arrangement. Dimensions are in centimeters; numbers 5 and 10 represent row number; F = gravity.

#### Moisture Mass Fraction Measurement

The moisture content in the samples was measured by gravimetric method using an ES-4610 drying cabinet (Ecros, Russia). According to this method, we dried 3 to 4 g of milk powder sample at  $102 \pm 2^{\circ}\text{C}$  for 2 h to a constant weight. To verify that the weight was constant, we dried the samples for another hour until the decrease in mass after the first and second drying did not exceed 0.0005 g.

#### Water Activity Measurement

Water activity was measured with a HygroLab 3 moisture analyzer (Rotronic, Switzerland) at 20°C according to ISO 18787:2017 (ISO, 2017).

#### Particle Size Distribution Measurement

The particle size distribution of milk powder samples was identified by laser diffraction with an LS 13 320 XR particle size analyzer (Beckman Coulter, USA) equipped with the Dry Powder System module. All measurements were carried out 3 times. The results of the studies were recorded and processed using the ADAPT software (v. 1.3.337; Bench).

#### Wetting Angle Measurement

The wetting angle  $(\theta)$  of the milk powder samples was measured by the sessile drop method with a Drop Shape Analyzer DSA25 (Krüss, Hamburg). A thin layer of the samples was applied to a double-sided adhesive

 Pable 2. Sample code names; naming convention is [milk type] [row number] [storage temperature in degrees Celsius]

		$-30\pm1^{\circ}\mathrm{C}^{1}$			$6 \pm 1^{\circ} \mathrm{C}^1$			$25\pm3^{\circ}\mathrm{C}^{1}$	
tem	$Control^2$	Layer $5^2$	Layer $10^2$	$Control^2$	$Control^2$ Layer $5^2$ Layer $10^2$	Layer $10^2$	$Control^2$	Layer $5^2$	Layer $10^2$
Skim milk (SM) powder Codename	SM0-30	SM5-30	SM10-30	SM0+6	SM5+6	SM10+6	SM0+25	SM5+25	SM10+25
whole milk (WM) powder Codename	WM0-30	WM5-30	WM10-30	WM0+6	$^{ m VM5+6}$	WM10+6	WM0+25	WM5+25	WM10+25
Storage temperature.									

tape ( $\theta=103^\circ$ ) glued to a slide. Excess milk powder was removed by tapping on the slide (Puri et al., 2010). A drop of distilled water  $20\pm1^\circ\mathrm{C}$  was added to a powder layer. The drop was photographed and measured using the Krüss Advance Drop Shape software (v. 1.12.2.06901, Krüss Gmbh). To estimate the average  $\theta$  value, the drop on the surface of milk powder was held for 3 min for WMP or 1 min for SMP. The reason for this is that it takes longer to stabilize the water droplet on the WMP surface. The data given herein are the average values of the obtained results of studies of milk powder samples.

# Reconstitution of Milk Powder Samples

We dissolved 12.5 g of whole milk powder or 9 g of skim milk powder in 87.5 and 91 g of distilled water at  $40 \pm 2$ °C, respectively. The sample was then incubated for 15 to 20 min at 18°C to 25°C.

# **Titratable Acidity**

Titratable acidity was measured with the method of potentiometric titration using an inoLab pH/Cond Level 1 pH meter (Wissenschaftlich-Technische Werkstatten GmbH [WTW], Germany) with a WTW Sen-Tix 81 glass combination electrode. We mixed 10 mL of reconstituted milk with 20 mL of distilled water, stirred and titrated with 0.1 N sodium hydroxide solution until the equivalence point (pH = 8.9) was reached. The acidity was expressed as the lactic acid share (%) using Equation [1] for conversion.

$$\label{eq:lactic_acid_share} \text{(\%)} = \frac{\text{volume of titrant} \times N \times 90}{\text{weight of sample} \times 1,000} \times 100,$$
 [1]

where N is the normality of titrant, and 90 is the equivalent for lactic acid. All the samples were analyzed twice. The limit of permissible error of measurement is 0.0072% of lactic acid content.

# Heat Stability Measurement

The heat stability of the reconstituted milk powder samples was determined by a thermal breakdown test using a UKT-150 heat stability monitor with a test tube rocker (BioPischeMash Engineering Center, Russia) equipped with a LOIP LT-316a circulation thermostat (Lab Equipment and Devices, Russia) (Vafin et al., 2021). To estimate the heat stability, we compared the exposure time in minutes. The experiment was as follows: 3 mL of reconstituted milk was introduced into

a molybdenum glass test tube, corked and placed in a cassette holder. The test samples were kept in a thermostat at  $140 \pm 1$ °C. The time was marked upon seeing the first signs of coagulation (precipitation or protein flakes appeared).

# Milk Powder Insolubility Index Measurement

The insolubility index was determined as follows: 10 mL of the reconstituted milk powder was introduced into graduated centrifuge-grade test tubes. After centrifugation (ZLM 1–12 centrifuge, Technocom Scientific and Production Association, Russia) for 5 min at 162  $\times$  g and 20°C, the supernatant was drained, and the tubes were refilled with water. Then the sample was centrifuged again for 5 min, and the precipitate volume was measured using the scale on the test tube. To express the insolubility index in milliliters, the solubility value was multiplied by 5. If the precipitate volume was less than 0.1 mL, it was registered as 0.05 mL. The discrepancy between the 2 concurrent measurements did not exceed 0.1 mL.

# Statistical Analysis

We applied the Kruskal-Wallis test (Ostertagová et al., 2014); 1-, 2-, and 3-way post hoc ANOVA (Rasch and Verdooren, 2020); and the Tukey's range test (McHugh, 2011) for the statistical analysis of the experimental data and statistical hypothesis testing. Unless otherwise specified, the significance level was assumed to be 0.05. The maximum log-likelihood method was used to match the particle size distribution to the Weibull distribution (Lucas et al., 2020). We applied the Wolfram Mathematica software (Wolfram Research, Inc.).

#### **RESULTS**

### Milk Powder Moisture Content and Water Activity

Figure 2 shows moisture content and  $a_w$  in milk powder. The average moisture content in the samples over 18 mo in any storage conditions was 3.77% to 4.08% (SMP), and 3.42% to 3.76% (WMP). The  $a_w$  value for SMP did not exceed 0.315 and 0.278 for WMP. Regression analysis of the results revealed no significant factors except for the free term (Student's criterion,  $\alpha = 0.05$ ), so the values did not change over time.

# Milk Powder Clumping

The WMP samples formed clumps during storage (Figure 3). The number and size of the clumps

depend on external conditions: temperature, location, and duration of storage. Sample WM10+25 showed the greatest changes at the 18th month of storage, where the average clump size reached 5 to 6 cm and a considerable effort was required to disperse them. The WMP samples stored in layer 10 at 6  $\pm$  1°C and -30  $\pm$  1°C formed significantly smaller clumps (P=0.024, up to 1 cm at 6  $\pm$  1°C and 2 to 3 cm at -30  $\pm$  1°C) and dispersed with little manual force. The SMP samples showed no significant changes over the entire storage period.

# Milk Powder Particle Size Distribution

The mean particle size ( $\mathbf{D}[4,3]$ ) at the 18th month of storage did not exceed for WM0+6 133.2 µm, for WM5+6 148.8 µm, and for WM10+6 159.0 µm. A decrease in storage temperature to  $-30 \pm 1^{\circ}\mathrm{C}$  increased D[4,3] to 181.7 µm only in WM10–30 samples. Storage at 25 ± 3°C significantly intensified the particle agglomeration: WM0+25: 161.8 µm; WM5+25: 192.5 µm; and WM10+25: 258.5 µm (all P < 0.01). At the 18th month of storage, for WM10+25, 10% of the particles larger than the indicated size in micrometers (**D90**) did not exceed original sizes by more than 166%. The WM0+25, WM5+25, and WM10+25 samples featured the largest agglomeration.

The SMP sample particle sizes differ greatly from that of WMP samples. The reference sample (SM0+6) particle size was 77.8 µm at the beginning of the storage period. All of the studied SMP samples featured an agglomeration rate similar to that of the WMP samples. In particular, after 18 mo of storage, the mean particle size increased by 6.15% and 9.55% for samples SM0+6, SM5+6, and SM10+6 and SM0-30, SM5-30, and SM10-30, respectively. The largest change in the particle size distribution was observed in samples SM0+25, SM5+25, and SM10+25: D90 were up to 12.4% greater than the original particle size. The Tukey's post hoc range test identified some differences in the particle size distributions of the samples stored in layers 5 and 10 (Table 3). All of the milk powder samples showed a statistically significant difference in their particle size distribution after storage (largest P = 0.039).

The particle size distribution curves for the SMP and WMP samples showed that all the control samples, as well as samples WM5–30 and WM5+6, corresponded to the Weibull distribution throughout the entire storage period. The WMP samples stored on the bottom layer and SM10+25 did not follow this distribution after 3 mo of storage, whereas for SM5–10, SM10+6, SM5–30, and SM10–30, it was after 6 mo. In general, the particle size distribution curves shifted right and down as the storage time increased. The greatest changes to the

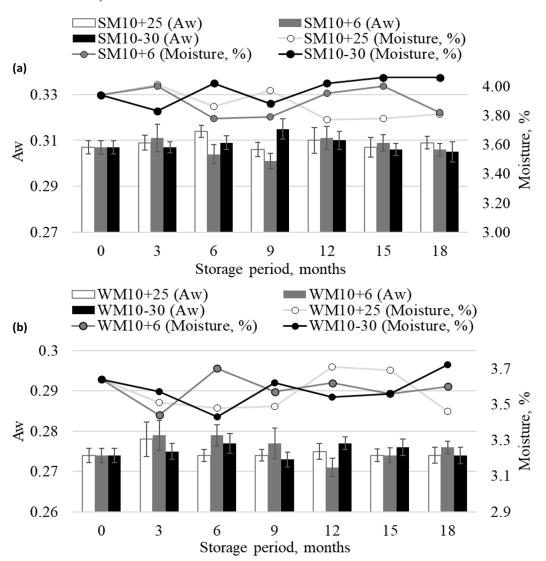


Figure 2. Water activity  $(a_w)$  and moisture mass fraction in the skim milk powder (a) and whole milk powder (b) samples stored in layer 10 for 18 mo. Naming convention is [milk type][row number][storage temperature in degrees Celsius], where WM = whole milk and SM = skim milk. Values depicted are mean  $\pm$  SD.

distribution curves were observed in the WMP samples stored on the bottom layer at all the given temperatures (Figure 4) and in all the WMP samples stored at  $25\pm3^{\circ}\mathrm{C}$ . Thus, in WM10+6 and WM10–30 samples, the curve peak shifted right and down from 128  $\mu m$  (0 mo) to 140  $\mu m$  and 169  $\mu m$  (18 mo), respectively. After 3 mo of storage, the curves featured another small peak at the 429  $\mu m$  particle size. The peaks in WM0+25, WM5+25, and WM10+25 samples shifted over time from 128  $\mu m$  (0 mo) to 169, 185, and 204  $\mu m$  (18 mo), respectively. The WM10+25 sample featured an additional small peak at 684  $\mu m$  after 12 mo of storage. This indicates a particle size increase, presumably due to their agglomeration.

The 3-way ANOVA was used to determine the effect of storage temperature on particle size distribution, where the categorical variable of the series (0, 5, 10), storage time, and temperature served as factors. The SMP and WMP samples were tested separately. Storage at  $25 \pm 3^{\circ}$ C significantly affected the particle size distribution in all WMP samples (largest P = 0.003, Figure 5a). Only the samples stored on the bottom layers showed statistically significant changes in the particle size distribution at  $-30 \pm 1^{\circ}$ C and  $6 \pm 1^{\circ}$ C (P = 0.041, P = 0.013, respectively). As to SMP samples, a statistically significant effect of temperature was found only for SM5+25 and SM10+25 samples starting from the 9th month of storage (largest P = 0.38, Figure 5b).



Figure 3. Photos of the skim milk powder (SMP) and whole milk powder (WMP) samples after 18 mo of storage.

To summarize, we found a significant effect of the combination of temperature and load on the WMP particle size distribution changes during storage. For SMP samples, SM10+25 featured great particle size distribution changes. The rest of the samples showed minimal changes not affecting the dissolution rate. It should be noted that temperature increase to  $25 \pm 3^{\circ}\mathrm{C}$  was the primary negative factor for all samples.

# Wetting Angle

Table 4 presents the  $\theta$  values measured over the 18 mo of storage. The initial mean  $\theta$  value for SMP samples was  $16.9 \pm 2.0^{\circ}$ , and for WMP samples it was  $53.3 \pm 1.9^{\circ}$ . The maximum increase of the mean  $\theta$  was recorded in the samples stored in layer 10 at  $25 \pm 3$ °C. For SMP samples, it was  $39.9 \pm 1.4^{\circ}$ . In WMP samples, the mean  $\theta$  increased to 71.3  $\pm$  2.1°. Subsequent statistical analysis of the mean  $\theta$  values showed a significant mismatch between the 3 WMP sample groups stored at  $-30 \pm 1^{\circ}\text{C}$ ,  $6 \pm 1^{\circ}\text{C}$ , and  $25 \pm 3^{\circ}\text{C}$  (P = 0.008) and 2 SMP samples stored at  $-30 \pm 1^{\circ}$ C,  $6 \pm 1^{\circ}$ C, and  $25 \pm 1^{\circ}$ C  $3^{\circ}$ C (P = 0.022; 2-way ANOVA and post hoc Tukey's range test). At the same time, the ANOVA did not indicate any relationship between the layer number and the  $\theta$ . Figure 6 shows the variations of the  $\theta$  in the reference sample (mo 0) and after 18 mo for various storage conditions.

# **Titratable Acidity**

In accordance with Guidelines MUK 4.2.1847-04 (developed by The Ministry of Health of the Russian Federation), with a declared shelf-life of more than 30 d, the safety factor must correspond to 1.2. The duration of the study was increased to 21.6 mo. The results are shown in Figure 7. The initial acidity of the samples was 0.154% and 0.156% in terms of lactic acid content for SMP and WMP samples, respectively. For WMP samples, significant changes were observed after 12 mo of storage for WM10+25 sample (P = 0.038), which amounted to 0.165% lactic acid. After 21.6 mo of storage, only WM0+6 and WM0-30 samples showed no significant changes. The maximum titratable acidity was measured after 21.6 mo of storage in WM10+25 sample: 0.172% lactic acid content. For SMP samples, significant titratable acidity changes (P < 0.05) were observed after 15 mo of storage. The largest changes occurred at 25  $\pm$  3°C.

### Solubility

Solubility is an important functional property of milk powder. It directly affects the production costs and the finished product quality. Milk powder is soluble when the insolubility index is less than 1 mL (99%). It is known that solubility mostly depends on the composi-

Table 3. Particle size distribution of whole milk powder (WMP) and skim milk powder (SMP) samples stored on different pallet layers (layers 5 and 10)<sup>1</sup>

			1	`		,		,		7			/		
		om 0	00	3 1	3 mo	6 1	6 mo	om 6	no	12	12 mo	15	15 mo	18 mo	mo
Item	$Code name^2 D[4,3]$	D[4,3]	D90	D[4,3]	D90	D[4,3]	D90	D[4,3]	D90	D[4,3]	D90	D[4,3]	D90	D[4,3]	D90
Storage at $6 \pm 1^{\circ}$ C															
	SM0+6	77.8	129.3	77.9	129.5	78.5	$130.5^{\mathrm{A}}_{\mathrm{B}}$	78.9	$130.8^{\rm A}_{\rm P}$	79.4	$131.5^{A}_{B}$	79.8	$131.6^{\mathrm{A}}_{\mathrm{B}}$	82.5	$136.0^{\rm A}_{ m B}$
	$_{9+2MS}$	77.8°	129.3	78.8	130.1	$79.5^{a}$	$129.8^{\text{b}*}$	$79.5^{a}$	$128.7^{5*}$	$79.0^{a}$	$127.0^{5*}$	$80.0^{a}$	$128.6^{5*}$	$83.0^{a}$	$134.4^{\text{b*}}$
	SM10+6	$77.8^{0}$	129.3	$78.6^{\rm p}$	130.1	$79.3^{a}$	$129.8^{5*}$	$79.1^{a}$	$128.0^{5*}$	$79.3^{3}$	$127.7^{5*}$	$79.2^{a}$	$127.8^{5*}$	$80.8^{a}$	$131.5^{b*}$
Storage at $-30 \pm 1^{\circ}$ C															
	SM0-30	77.8	129.3	8.77	129.6	78.4	$130.8^{\mathrm{A}}$	79.2	$131.9^{A}$	7.62	$132.9^{A}$	79.2	$132.1^{A}$	78.3	$130.7^{A}$
	SM5-30	$77.8^{\circ}$	129.3	$79.6^{\mathrm{p}}$	130.1	$79.5^{\rm b}$	$130.1^{B*}$	$79.5^{\mathrm{b}}$	$129.0^{\text{B}*}$	$79.0^{\rm p}$	$127.3^{B*}$	$80.0^{\rm p}$	$129.0^{B*}$	$83.2^{a}$	$135.4^{B*}$
	SM10-30	$77.8^{\circ}$	129.3	$78.6^{\circ}$	130.1	$79.3^{\rm b}$	$127.7^{B*}$	$79.2^{\rm b}$	$127.8^{B*}$	$81.7^{\mathrm{a}}$	$133.3^{B*}$	$84.1^{a}$	$137.7^{B*}$	$85.2^{a}$	$138.0^{B*}$
Storage at $25 \pm 3^{\circ}$ C															
	SM0+25	8.77	129.3	8.77	$129.3^{A}$	79.1	$131.9^{A}$	79.4	$132.5^{A}$	78.3	$130.4^{\mathrm{A}}$	79.5	$132.7^{A}$	79.1	$131.9^{A}$
	SM5+25	$77.8^{\rm b}$	129.3	$79.1^{\mathrm{b}}$	$131.9^{A}$	78.3	$130.4^{\mathrm{A}}$	79.4	$132.5^{A}$	80.9	$134.8^{\rm A}$	$82.3^{a}$	$137.0^{\text{B}*}$	$83.3^{a}$	$138.8^{B*}$
	SM10 + 25	$77.8^{\circ}$	129.3	$79.1^{\mathrm{b}}$	$130.8^{B*}$	$80.9^{\rm p}$	$134.8^{B*}$	83.0	$138.0^{B*}$	$85.4^{\rm a}$	$140.2^{B*}$	$86.8^{a}$	$143.8^{\text{C}*}$	$87.8^{a}$	$145.4^{\text{C}*}$
Storage at $6 \pm 1^{\circ}$ C															
	MM0+6	$120.3^{\rm b}$	209.8	$120.2^{\rm b}$	$210.2^{A}$	$122.5^{\rm b}$	$214.9^{A}$	127.4	$224.8^{A}$	$130.7^{\rm a}$	$231.7^{\mathrm{A}}$	$132.3^{a}$	$235.5^{\mathrm{A}}$	$133.2^{a}$	$237.1^{\mathrm{A}}$
	MM5+6	$120.3^{c}$	209.8	$124.3^{\rm b}$	$217.3^{\mathrm{A}}$	$127.4^{\rm b}$	$222.8^{\mathrm{A}}$	$131.2^{\rm b}$	$230.0_{2}$	$139.1^{\rm b}$	$243.0^{\rm B}$	$145.0^{a}$	$255.8^{\rm E}$	$148.8^{a}$	$262.2^{\mathrm{B}}_{\widetilde{\mathfrak{g}}}$
	WM10+6	$120.3^{c}$	209.8	$133.5^{\mathrm{b}}$	$234.8^{B*}$	141.6	$248.2^{B*}$	142.7	$252.3^{B*}$	147.8	$262.4^{B*}$	$154.3^{a}$	$275.8^{\circ}$ *	$159.0^{\rm a}$	$287.0^{\circ}$
Storage at $-30 \pm 1$ °C															
	WM0-30	$120.3^{\rm b}$	209.8	$120.2^{\rm b}$	$210.4^{A}$	$122.5^{b}$	$214.9^{A}$	127.4	$224.4^{A}$	$129.6^{a}$	$230.0^{A}$	$131.3^{a}$	$233.1^{A}$	$132.5^{a}$	$234.5^{A}$
	WM5-30	$120.3^{\rm b}$	209.8	$124.3^{\rm b}$	$217.3^{\mathrm{A}}$	$127.7^{\rm b}$	$223.8^{\mathrm{A}}$	131.8	$232.6^{ m A}$	$136.6^{a}$	$242.7^{A}$	$139.5^{a}$	$249.3^{ m A}$	$142.7^{a}$	$255.7^{\mathrm{A}}$
	WM10-30	$120.3^{c}$	209.8	$141.6^{\rm b}$	$248.2^{B*}$	$146.7^{\rm b}$	$259.0^{8*}$	$154.8^{\rm b}$	$278.4^{\text{B}*}$	$167.8^{\rm a}$	$309.0^{18}$ *	$175.3^{a}$	$323.2^{5*}$	$181.7^{a}$	$339.1^{B*}$
Storage at $25 \pm 3^{\circ}$ C				-	٠		٠								
	WM0+25	$120.3^{\circ}$	209.8	$127.2^{\rm b}$	$222.7^{A}$	$133.0^{\rm b}$	$232.9^{A}$	$140.5^{\rm b}$	$244.5^{A}$	$152.0^{\rm a}$	$265.2^{\rm A}_{ m B}$	$156.0^{\rm a}$	$273.7^{A}$	$161.8^{\rm a}$	$282.6^{\rm A}$
	WM5+25	$120.3^{\circ}$	209.8	138.8	$241.1_{B.}$	$146.5^{\circ}$	$256.0^{*}$	$161.3^{\circ}$	$279.7^{\text{p*}}$	$176.5^{\circ}$	$310.6^{-*}$	$185.0^{\circ}$	$325.3^{-*}$	$192.5^{\circ}$	$341.6^{-*}$
	WM10 + 25	$120.3^{\circ}$	209.8	$155.2^{\mathrm{d}}$	$272.5^{6*}$	$183.4^{\mathrm{c}}$	$344.6^{8*}$	$203.1^{\rm b}$	$382.9^{\circ}*$	$231.5^{a}$	$462.1^{\circ*}$	$245.3^{a}$	$512.7^{\circ*}$	$258.5^{a}$	$559.8^{\circ}$

A-C Different uppercase superscripts in columns denote statistical difference between the samples stored at different pallet layers (one-way ANOVA and post hoc Tukey's test); P \*\*\*Different lowercase superscripts in rows denote statistical difference between the particle size distributions during storage (Kruskal-Wallis test; groups denoted with different letters are significantly different from each other); P < 0.05.

'D[4,3] column: mean particle size, μm; D90 column: 10% of the particles are larger than the indicated size, μm.

'Naming convention is [milk type] [row number] [storage temperature in degrees Celsius].

\*Significant agglomeration (the size distribution no longer matches the Weibull distribution); P < 0.05

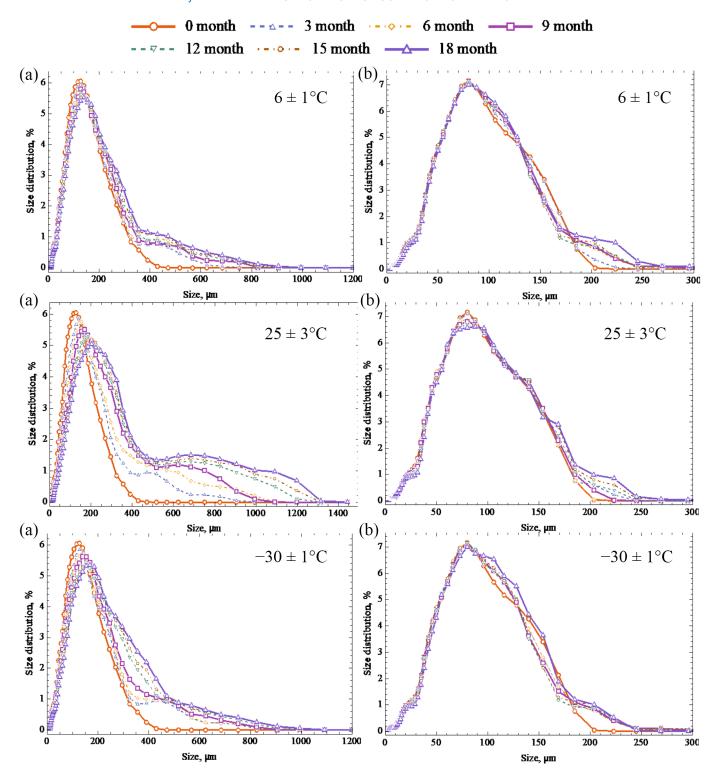


Figure 4. Particle size distribution in whole milk powder (WMP; a) and skim milk powder (SMP; b) samples in layer 10 at different temperatures during 18 mo of storage. The figures show the particle agglomeration and formation of particle clusters 500  $\mu$ m for WMP and 200  $\mu$ m for SMP samples.

**Table 4.** Average wetting angle values (mean  $\pm$  SD) for skim milk powder (SMP) and whole milk powder (WMP) samples at 18 mo of storage<sup>1</sup>

SMP		WMP	
Sample	Average wetting angle	Sample	Average wetting angle
SM0	$16.9 \pm 2.0$	WM0	$53.3 \pm 1.9$
SM0-30	$23.4 \pm 1.8$	WM0-30	$57.1 \pm 2.0$
SM5-30	$22.8 \pm 1.9$	WM5-30	$60.2 \pm 2.2$
SM10-30	$23.8 \pm 2.1$	WM10-30	$61.8 \pm 1.8$
SM0+6	$19.8 \pm 2.4$	WM0+6	$58.8 \pm 2.2$
SM5+6	$21.7 \pm 2.1$	WM5+6	$59.6 \pm 2.0$
SM10+6	$22.8 \pm 1.6$	WM10+6	$60.6 \pm 2.4$
SM0+25	$25.8 \pm 1.5$	WM0+25	$66.1 \pm 1.7$
SM5+25	$35.4 \pm 0.9$	WM5 + 25	$69.0 \pm 2.4$
SM10+25	$39.9 \pm 1.4$	WM10+25	$71.3 \pm 2.1$

<sup>1</sup>SM0 and WM0: SMP and WMP samples at mo 0 for reference. Naming convention is [milk type][row number] [storage temperature in degrees Celsius], where WM = whole milk and SM = skim milk.

tion, especially on the nature and structure of proteins (Felfoul et al., 2021).

Figure 8 shows the dynamics of the solubility of WMP and SMP measured during 21.6 mo of storage. The figure shows that both products have an identical insolubility increase pattern. The insolubility index of freshly prepared WMP and SMP was less than 0.5 mL. At the same time, WMP solubility did not actually change during the first 3 mo of storage, regardless of storage conditions. From the sixth month of storage, we found significant changes in samples WM0+25, WM5+25, and WM10+25 (largest P=0.044). Samples WM10+25 and WM5+25 after 15 and 21.6 mo of storage exceeded 1 mL, which is the upper value of the normalized limit. The smallest changes for WMP samples were observed in the samples stored at  $6 \pm 1^{\circ}$ C and  $-30 \pm 1^{\circ}$ C. The SMP samples showed a different

pattern of solubility change with less scattering over a particular period. From the 15th month of storage, critical solubility changes were observed in samples SM10–30 and SM10+25. Only samples SM0+6 and SM5+6 had acceptable solubility after 21.6 months of storage, according to regulatory documentation (1 mL is the upper value of the insolubility index; if exceeded, milk powder is rejected).

# **Heat Stability**

The heat stability of reconstituted milk is an important factor in its further processing and determines the properties of the finished product. This indicator, in fact, is integral and is formed on the basis of chemical and physical properties of the substance. As part of the research, we studied the heat stability variations in

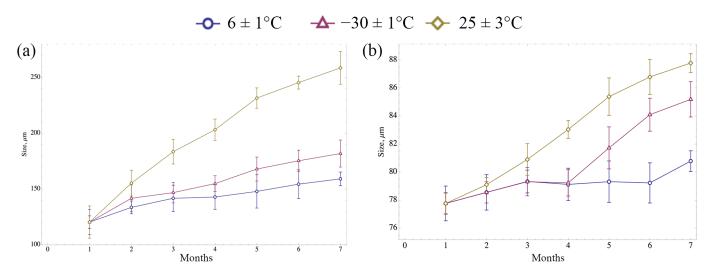


Figure 5. Effect of the storage temperature on the particle size distribution in whole milk powder (a) and skim milk powder (b) samples. Values depicted are mean  $\pm$  SD.

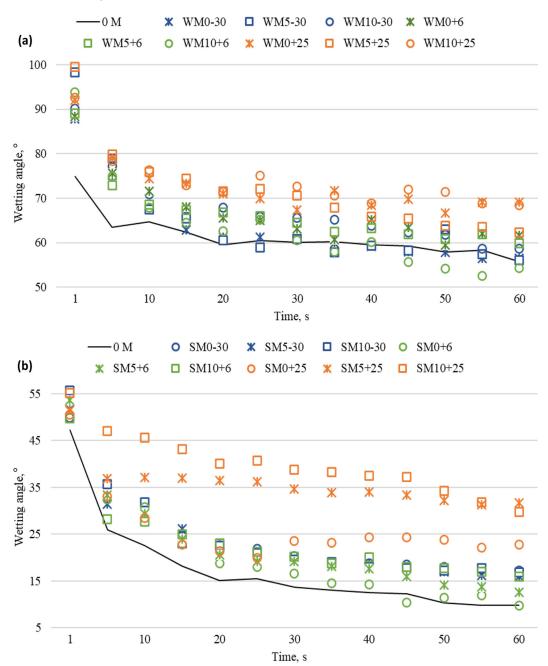


Figure 6. Changes of wetting angle in whole milk powder (a) and skim milk powder (b) samples after 18 mo of storage. Naming convention is [milk type][row number][storage temperature in degrees Celsius], where WM = whole milk, and SM = skim milk. 0 M = wettability angle in whole milk powder and skim milk powder samples before storage.

WMP and SMP samples during storage (Figure 9). The heat stabilities of WMP and SMP samples decreased in the same pattern. The mean values at the start of the study were 41.6 min for WMP samples and 52.2 min for SMP samples. The samples stored at  $25\pm3^{\circ}\mathrm{C}$  showed the lowest heat stability. The range of the heat stability values after 21.6 mo of storage was 12.1 to 15.3 for WMP samples and 16.9 to 19.7 for SMP samples.

In the  $6 \pm 1^{\circ}$ C and  $-30 \pm 1^{\circ}$ C temperature range, the heat stability change rate was reduced.

# **DISCUSSION**

Industrial storage of milk powder is an important post-technological stage in ensuring the quality of the finished product. At the same time, the above-zero

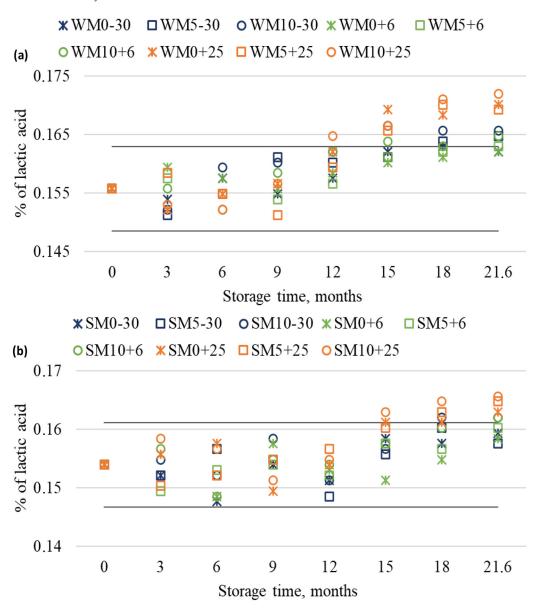


Figure 7. Titratable acidity in whole milk powder (a) and skim milk powder (b) samples. The horizontal lines indicate the acceptable error range. Naming convention is [milk type][row number][storage temperature in degrees Celsius], where WM = whole milk and SM = skim milk.

temperature ranges during storage are well studied in contrast to below-zero ones. Most studies focus on the effects of temperature and humidity on the functional and technological properties of milk powder and do not take into account the possible impact of loads applied to milk power during industrial storage. In this regard, the purpose of this study is to determine the effect of industrial storage at various temperature conditions, including low below-zero and high above-zero ones, on the change in  $a_{\rm w}$ , particle size distribution, caking, rehydration, and heat stability of milk powder produced in Russia.

In this study we used SMP and WMP samples obtained from various Russian manufacturers. The samples were sealed and stored on a storage simulator ("mini-pallet") for 18 mo at  $-30 \pm 1$ °C,  $6 \pm 1$ °C, and  $25 \pm 3$ °C and 40 to 80% RH to simulate the actual industrial storage conditions. The samples were taken and analyzed every 3 mo.

The moisture content and  $a_w$  are associated with the main changes in milk powder. The moisture content in the samples did not exceed the recommended 5% (Fournaise et al., 2020; GOST 33629-2015) at the start of the experiment. The  $a_w$  values were consistent with

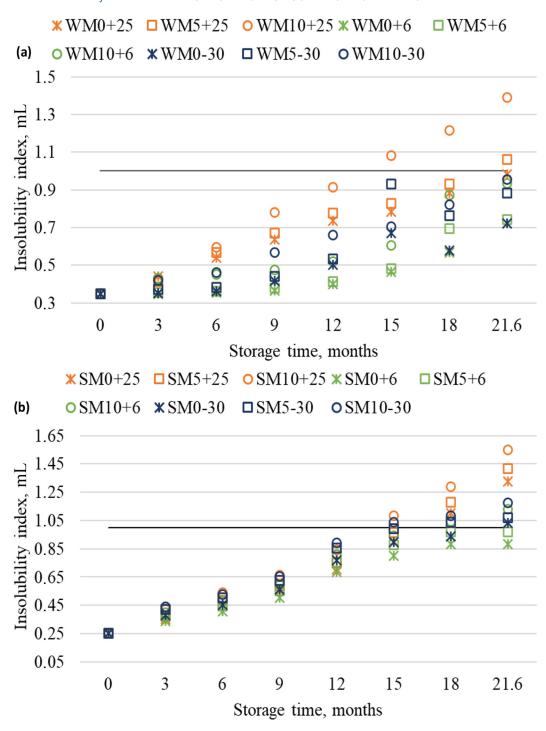


Figure 8. Insolubility index for whole milk powder (a) and skim milk powder (b) samples. Naming convention is [milk type][row number] [storage temperature in degrees Celsius], where WM = whole milk and SM = skim milk.

the results reported by other researchers (Galstyan et al., 2016; Pugliese et al., 2017; Fournaise et al., 2020). These values did not change over the 18 mo of storage. However, Ellahi et al. (2011) reported an increase in the moisture content in WMP samples stored in poly-

ethylene (PE) bags for 120 d at 15 and 40°C. However, Yang et al. (2022) did not report any significant  $a_{\rm w}$  change after 180 d of SMP and WMP storage in resealable plastic PE bags at about 22°C. We believe there were no significant changes in moisture content and  $a_{\rm w}$ 

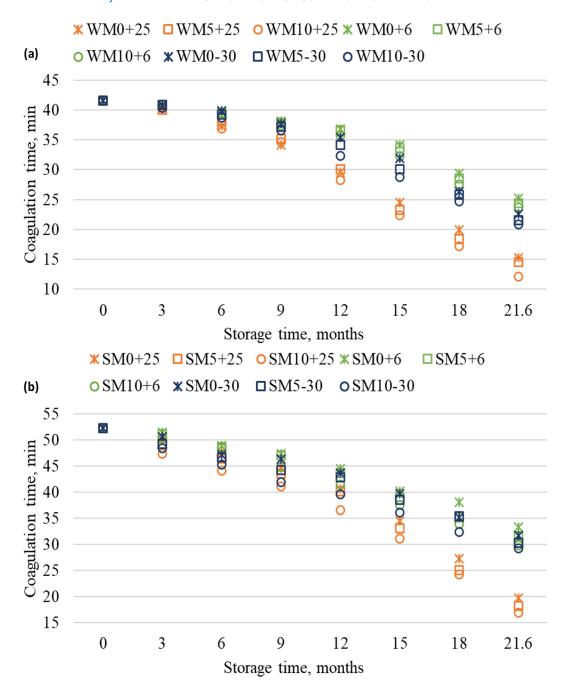


Figure 9. Heat stability of the reconstituted whole milk powder (a) and skim milk powder (b) samples versus storage conditions. Naming convention is [milk type][row number][storage temperature in degrees Celsius], where WM = whole milk and SM = skim milk.

due to a closed, sealed environment without ambient air humidity fluctuations.

We observed SMP and WMP particle size distribution changes during storage, and the presence of clumps was characteristic only of WMP samples. It should be noted that clumping in amorphous powders with a moisture content of less than 5% is undesirable as further agglomeration causes sticking and caking with

the formation of poorly soluble agglomerates (Pugliese et al., 2017; Dag et al., 2019; Kruchinin et al., 2020; Sert and Mercan, 2021). All this hampers the subsequent milk powder processing. The kinetics of product structure change (particle size distribution and clumping) is influenced by many factors, the main ones being the particle structure, ambient temperature and RH during storage, and the applied mechanical impact

(Aguilera et al., 1995). It is generally accepted that structural changes in milk powder occur as the humidity increases, which corresponds to a decrease in the  $T_g$  of milk powder below the storage temperature. At temperatures above T<sub>g</sub>, the powder is transitioned from an amorphous to a glass state, which greatly increases water mobility and promotes the formation of liquid and solid interparticle bridges (Roos, 2010; Phosanam et al., 2020b; Juarez-Enriquez et al., 2022). However, in our study, the moisture content did not rise, and therefore we can claim that the load applied to milk powder significantly boosts clumping and changes the particle size distribution. At the same time, the results obtained allow us to associate changes in the product structure with the mass fraction of fat. The number and size of clumps increase with the increasing mass fraction of fat. This probably occurs due to the formation of free (surface) fat, which increases the cohesion of the particles (Kim et al., 2009), thereby contributing to lower flowability of the powder.

The  $\theta$  between the surfaces of the powder and the water drop is an indicator of milk powder wettability. As a rule, smaller  $\theta$  values correspond to higher wettability, whereas larger  $\theta$  values correspond to lower wettability (Kim et al., 2002; Angelopoulou et al., 2021). The results of our study are consistent with WMP data reported by Angelopoulou et al. (2021), whereas previous studies by O'Sullivan et al. (2017) presented different, higher values for SMP. We assume that the discrepancy in the average  $\theta$  values for SMP is most likely due to a different sample preparation procedure, which consisted in the formation of cylindrical pellets. This method is able to significantly modify the structure of milk powder, thereby affecting the average  $\theta$ . Nevertheless, our results confirm that milk powder particles with a fat (hydrophobic component) on their surface show lower wettability and higher  $\theta$  than particles with a hygroscopic surface and lower  $\theta$  (Kim et al., 2002). Previous works have considered  $\theta$  values simultaneously, without studying its dynamics during storage. We found that the greatest change in  $\theta$  for WMP occurred during storage at 25  $\pm$  3°C. This indirectly confirms the possibility of free fat formation in the mass of the product and predicts a decrease in the efficiency of its dissolution (Kim et al., 2002). The SMP sample data are the most interesting. We observed an increase of  $\theta$  at high above-zero storage temperatures, which is intensified by the storage location depending on the pallet layer. This can probably be explained by the fact that SMP particle surfaces may be covered by as much 25% fat and by the lack of a homogenization operation in SMP manufacturing process. It leads to the release of residual fat during storage at high temperatures and protein transformations.

Titratable acidity is a characteristic of a product indicating the amount of acid residues in it. High acidity values indicate low product quality. The initial acidity of the samples was 0.154% and 0.156% in terms of lactic acid content for SMP and WMP samples, respectively. Its increase during storage mostly depended on the temperature and milk powder type. In this regard, we can state that the reactions are initiated and intensified with the complication of the system and an increase in temperature. A similar change in milk powder properties during storage was noted by Ellahi et al. (2011), who attributed the changes to the possible growth of psychrophilic and thermophilic microorganisms. However, Chudy et al. (2015) attributed the increase in acidity to hydrolysis, the formation of free fatty acids, oxidation of fats, and the reaction of glucose with proteins and phospholipids. Since the permissible range of titratable acidity specified in the Russian regulations is from 0.126\% to 0.189\% of lactic acid in milk powder (GOST 33629-2015, 2015), the changes we observed were not critical. According to the Codex Alimentarius CXS 207-1999 (1999), the upper range of titratable acidity corresponds to 0.162\% of lactic acid. A probable main factor is initial acidity. We can assume that as it decreases, the acidity increase rate in storage will be significantly lower or remain at the same level.

Solubility is an important functional property of milk powder. It directly affects the production costs and the finished product quality. Milk powder is soluble when the insolubility index is less than 1 mL (99%). It is known that solubility mostly depends on the composition, especially on the nature and structure of proteins (Felfoul et al., 2021). The results of our study showed that significant changes in milk powder solubility were observed after 15 mo of storage, which correlates with previously obtained data (Ellahi et al., 2011; Galstyan et al., 2016). We introduced an additional factor for detailing the influence of the pallet layer number, with the help of which it was found that the greatest change occurred in samples SM10+25 and WM10+25. We attribute this change to the loss of native properties of proteins, which corresponds to the general concept of biomacropolymer aging proposed by Gorbatova (2021).

One of the important technological properties of milk powder is heat stability. As part of the research, the dynamics of heat stability of WMP and SMP samples in storage were determined. The results of the study showed that WMP and SMP samples had a similar pattern of index decline. Given the available data on the decrease of solubility, a similar heat stability decrease confirms the protein phase degradation (Sharma et al., 2012; Singh, 2004; Radaeva et al., 2019).

### **CONCLUSIONS**

Milk powder is a long shelf-life product with high nutritional value, used in various branches of the food industry. Taking into account the geographical and climatic conditions of Russia, milk powder storage conditions vary from below-zero to above-zero temperatures. We could not find any data on milk powder storage at below-zero temperatures. There are no data on the influence of the bag location on the pallet. The data obtained by us confirmed the possibility of storing milk powder at  $-30 \pm 1^{\circ}$ C and  $25 \pm 3^{\circ}$ C for 18 mo without any weight load applied. At the same time, the industrial storage simulation revealed that at -30± 1°C the location of the product on the pallet is not essential. At  $25 \pm 3$ °C, the location and mass fraction of fat significantly affect milk powder quality. In particular, the most pronounced negative changes were associated for SMP with solubility, heat stability, and titratable acidity. The first signs of product quality loss were observed after 15 mo of storage. For WMP, the particle size distribution, solubility, heat stability, and titratable acidity were mostly affected. At the same time, the change rate confirms the possibility of storing the product for up to 15 mo without considerable loss of quality.

# **ACKNOWLEDGMENTS**

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### **REFERENCES**

- Aguilera, J. M., J. M. del Valle, and M. Karel. 1995. Caking phenomena in amorphous food powders. Trends Food Sci. Technol. 6:149–155. https://doi.org/10.1016/S0924-2244(00)89023-8.
- Angelopoulou, D., V. Meunier, L. Forny, G. Niederreiter, S. Palzer, and A. D. Salman. 2021. Particle surface design for enhanced reconstitution of fat-based food powders. Powder Technol. 393:397–404. https://doi.org/10.1016/j.powtec.2021.07.054.
- Bianchi, F. R., L. Moreschi, M. Gallo, E. Vesce, and A. Del Borghi. 2021. Environmental analysis along the supply chain of dark, milk and white chocolate: A life cycle comparison. Int. J. Life Cycle Assess. 26:807–821. https://doi.org/10.1007/s11367-020-01817-6.
- Assess. 26:807–821. https://doi.org/10.1007/s11367-020-01817-6. Bianco, M., C. D. Calvano, G. Ventura, I. Losito, and T. R. I. Cataldi. 2022. Determination of hidden milk allergens in meat-based food-stuffs by liquid chromatography coupled to electrospray ionization and high-resolution tandem mass spectrometry. Food Control 131:108443. https://doi.org/10.1016/j.foodcont.2021.108443.
- Cândido de Souza, W. F., C. R. Souza do Amaral, and P. D. Lima da Silva Bernardino. 2021. The addition of skim milk powder and dairy cream influences the physicochemical properties and the sensory acceptance of concentrated Greek-style yogurt. Int. J. Gastron. Food Sci. 24:100349. https://doi.org/10.1016/j.ijgfs.2021 .100349.

- Carpin, M., H. Bertelsen, A. Dalberg, J. K. Bech, J. Risbo, P. Schuck, and R. Jeantet. 2017. How does particle size influence caking in lactose powder? J. Food Eng. 209:61–67. https://doi.org/10.1016/j.jfoodeng.2017.04.006.
- Chudy, S., J. Pikul, M. Rudzinska, and A. Makowska. 2015. The effect of storage on physicochemical properties of spray-dried milk, egg and milk-egg mixture. Acta Agrophysica 22:17–26.
- Council of the European Union. 2002. Council Directive 2001/114/ EC of 20 December 2001 relating to certain partly or wholly dehydrated preserved milk for human consumption. Off. J. Eur. Commun. 7:19–23.
- Council of the European Union. 2013. Regulation (EU) No 1021/2013 of the European Parliament and of the Council of 9 October 2013 amending Directives 1999/4/EC and 2000/36/EC of the European Parliament and of the Council and Council Directives 2001/111/EC, 2001/113/EC and 2001/114/EC as regards the powers to be conferred on the Commission. Official Journal of the European Union.
- CXS 207-1999. 1999. Standard For Milk Powders and Cream Powder CXS 207. Codex Alimentarius.
- d'Almeida Francisquini, J., E. Martins, I. R. Toledo Renhe, L. F. Cappa de Oliveira, R. Stephani, Í. T. Perrone, and A. Fernandes de Carvalho. 2020. Particle size distribution applied to milk powder rehydration. Quim. Nova 43:226–230. https://doi.org/10.21577/0100-4042.20170466.
- Dag, D., R. K. Singh, and F. Kong. 2019. Dielectric properties, effect of geometry, and quality changes of whole, nonfat milk powder and their mixtures associated with radio frequency heating. J. Food Eng. 261:40–50. https://doi.org/10.1016/j.jfoodeng.2019.04.017.
- Ding, H., W. Yu, I. Boiarkina, N. Depree, and B. R. Young. 2020. Effects of morphology on the dispersibility of instant whole milk powder. J. Food Eng. 276:109841. https://doi.org/10.1016/j .jfoodeng.2019.109841.
- Ellahi, M. Y., M. Nadeem, A. Rashid, and A. M. Arif. 2011. Pysicochemical and sensory characteristics of whole milk powder stored at different temperatures. Carpathian J. Food Sci. Technol. 3:47– 53.
- Ergashovna, Y. I., R. Otabek, and H. Ba. 2022. Enrichment of bread products on the basis of milk whey. Galaxy International Interdisciplinary Research Journal 10:257–261.
- Fan, F., M. Liu, P. Shi, X. Xu, W. Lu, Z. Wang, and M. Du. 2018. Protein cross-linking and the Maillard reaction decrease the solubility of milk protein concentrates. Food Sci. Nutr. 6:1196–1203. https://doi.org/10.1002/fsn3.657.
- Felfoul, I., J. Burgain, C. Perroud, C. Gaiani, J. Scher, H. Attia, and J. Petit. 2021. Impact of spray-drying conditions on flow properties of skim dromedary and cow's milk powders using the FT4 powder rheometer. J. Food Process. Preserv. 45. https://doi.org/10.1111/jfpp.15566.
- Fialho, T. L., L. G. L. Nascimento, A. Moreau, G. Delaplace, E. Martins, Í. Tuler Perrone, A. F. Carvalho, and P. P. de Sá Peixoto Junior. 2021. Sugar type matters in spray drying II: Glycation effects on physicochemical characteristics of aged lactose-hydrolyzed milk powder. Food Structure 30:100215. https://doi.org/10.1016/j.foostr.2021.100215.
- Fournaise, T., J. Burgain, C. Perroud, J. Scher, C. Gaiani, and J. Petit. 2020. Impact of formulation on reconstitution and flowability of spray-dried milk powders. Powder Technol. 372:107–116. https://doi.org/10.1016/j.powtec.2020.05.085.
- Galstyan, A. G., A. N. Petrov, E. E. Illarionova, V. K. Semipyatniy, S. N. Turovskaya, A. E. Ryabova, S. A. Khurshudyan, R. R. Vafin, and I. A. Radaeva. 2019. Effects of critical fluctuations of storage temperature on the quality of dry dairy product. J. Dairy Sci. 102:10779–10789. https://doi.org/10.3168/jds.2019-17229.
- Galstyan, A. G., A. N. Petrov, I. A. Radaeva, S. N. Turovskaya, V. V. Chervetsov, E. E. Illarionova, and V. K. Semipyatnyi. 2016. Dairy and Canning Food Production. Theory and Applications [in Russian]. Fedotov D.A. Publishing House.
- Gilmanov, K. K., V. K. Semipyatniy, A. V. Bigaeva, R. R. Vafin, and S. N. Turovskaya. 2020. New determination method for the ratio of the relative proportions of æ-casein alleles in milk powder. Food

- Processing: Techniques and Technology 50:525–535. https://doi.org/10.21603/2074-9414-2020-3-525-535.
- Gorbatova, K. K. 2021. Biochemistry of Milk and Dairy Products. 5th ed. GIORD.
- GOST 19360-74. 1975. Russian National Standard. Film liner-bags. Generic specifications [in Russian] Official edition, IPK Izdatelstvo Standartov, Moscow.
- GOST 33629-2015. 2015. Russian National Standard. Canned milk. Powder Milk. Specifications (as amended) [in Russian] Official edition. Standartinform, Moscow.
- Han, J., J. Fitzpatrick, K. Cronin, and S. Miao. 2021. Investigation of the influence of powder characteristics on the breakage of dairy powders. Food Res. Int. 150:110775. https://doi.org/10.1016/j .foodres.2021.110775.
- Hernández, P., A. Carmona, M. S. Tapia, and S. Rivas. 2021. Dismantling of institutionalization and state policies as guarantors of food security in Venezuela: Food safety implications. Front. Sustain. Food Syst. 5:623603. https://doi.org/10.3389/fsufs.2021.623603.
- Ho, T. M., S. Chan, A. J. E. Yago, R. Shravya, B. R. Bhandari, and N. Bansal. 2019. Changes in physicochemical properties of spraydried camel milk powder over accelerated storage. Food Chem. 295:224–233. https://doi.org/10.1016/j.foodchem.2019.05.122.
- Ho, T. M., T. T. Ton, C. Gaiani, B. R. Bhandari, and N. Bansal. 2021. Changes in surface chemical composition relating to rehydration properties of spray-dried camel milk powder during accelerated storage. Food Chem. 361:130136. https://doi.org/10.1016/j .foodchem.2021.130136.
- ISO. 2017. ISO 18787:2017(en) Foodstuffs—Determination of water activity.
- Juarez-Enriquez, E., G. I. Olivas, P. B. Zamudio-Flores, S. Perez-Vega, I. Salmeron, E. Ortega-Rivas, and D. R. Sepulveda. 2022. A review on the influence of water on food powder flowability. J. Food Process Eng. 45. https://doi.org/10.1111/jfpe.14031.
- Kamali Rousta, L., S. Bodbodak, M. Nejatian, A. P. Ghandehari Yazdi, Z. Rafiee, J. Xiao, and S. M. Jafari. 2021. Use of encapsulation technology to enrich and fortify bakery, pasta, and cereal-based products. Trends Food Sci. Technol. 118:688–710. https://doi.org/10.1016/j.tifs.2021.10.029.
- Kim, E. H. J., X. D. Chen, and D. Pearce. 2002. Surface characterization of four industrial spray-dried dairy powders in relation to chemical composition, structure and wetting property. Colloids Surf. B Biointerfaces 26:197–212. https://doi.org/10.1016/S0927-7765(01)00334-4.
- Kim, E. H. J., X. D. Chen, and D. Pearce. 2009. Surface composition of industrial spray-dried milk powders. 2. Effects of spray drying conditions on the surface composition. J. Food Eng. 94:169–181. https://doi.org/10.1016/j.jfoodeng.2008.10.020.
- Kim, E. H. J., X. D. Chen, and D. Pearce. 2005. Effect of surface composition on the flowability of industrial spray-dried dairy powders. Colloids Surf. B Biointerfaces 46:182–187. https://doi.org/10 .1016/j.colsurfb.2005.11.005.
- Kosasih, L., B. Bhandari, S. Prakash, N. Bansal, and C. Gaiani. 2016. Effect of whole milk concentrate carbonation on functional, physicochemical and structural properties of the resultant spray dried powder during storage. J. Food Eng. 179:68–77. https://doi.org/10.1016/j.jfoodeng.2016.02.005.
- Kruchinin, A. G., E. E. Illarionova, A. V. Bigaeva, and S. N. Turovska-ya. 2020. The role of dry milk technological properties in forming the quality of food systems. Krasnoyarsk State Agricultural University Bulletin 8:166–173. https://doi.org/10.36718/1819-4036-2020-8-166-173. [in Russian]
- Lin, Y., A. L. Kelly, J. A. O'Mahony, and T. P. Guinee. 2018. Effect of heat treatment, evaporation and spray drying during skim milk powder manufacture on the compositional and processing characteristics of reconstituted skim milk and concentrate. Int. Dairy J. 78:53-64. https://doi.org/10.1016/j.idairyj.2017.10.007.
- Lucas, J., M. Ralaivao, B. N. Estevinho, and F. Rocha. 2020. A new approach for the microencapsulation of curcumin by a spray drying method, in order to value food products. Powder Technol. 362:428–435. https://doi.org/10.1016/j.powtec.2019.11.095.

- Maidannyk, V. A., D. J. McSweeney, S. Montgomery, V. L. Cenini, B. M. G. O'hagan, L. Gallagher, S. Miao, and N. A. McCarthy. 2022. The effect of high protein powder structure on hydration, glass transition, water sorption, and thermomechanical properties. Foods 11:292. https://doi.org/10.3390/foods11030292.
- Masum, A. K. M., J. Chandrapala, T. Huppertz, B. Adhikari, and B. Zisu. 2020a. Influence of drying temperatures and storage parameters on the physicochemical properties of spray-dried infant milk formula powders. Int. Dairy J. 105:104696. https://doi.org/ 10.1016/j.idairyj.2020.104696.
- Masum, A. K. M., J. Chandrapala, T. Huppertz, B. Adhikari, and B. Zisu. 2020b. Effect of storage conditions on the physicochemical properties of infant milk formula powders containing different lactose-to-maltodextrin ratios. Food Chem. 319:126591. https:// doi.org/10.1016/j.foodchem.2020.126591.
- McHugh, M. L. 2011. Multiple comparison analysis testing in ANOVA. Biochem. Med. (Zagreb) 21:203–209. https://doi.org/10.11613/BM.2011.029.
- Mistry, V. V., and J. B. Pulgar. 1996. Physical and storage properties of high milk protein powder. Int. Dairy J. 6:195–203. https://doi.org/10.1016/0958-6946(95)00002-X.
- Morr, C. V., B. German, J. E. Kinsella, J. M. Regenstein, J. P. V. Buren, A. Kilara, B. A. Lewis, and M. E. Mangino. 1985. A collaborative study to develop a standardized food protein solubility procedure. J. Food Sci. 50:1715–1718. https://doi.org/10.1111/j.1365-2621.1985.tb10572.x.
- Murayama, D., Y. Zhu, and S. Ikeda. 2021. Correlations between the solubility and surface characteristics of milk protein concentrate powder particles. J. Dairy Sci. 104:3916–3926. https://doi.org/10 .3168/jds.2020-18311.
- O'Donoghue, L. T., M. K. Haque, D. Kennedy, F. R. Laffir, S. A. Hogan, J. A. O'Mahony, and E. G. Murphy. 2019. Influence of particle size on the physicochemical properties and stickiness of dairy powders. Int. Dairy J. 98:54–63. https://doi.org/10.1016/j.idairyj.2019.07.002.
- O'Sullivan, J. J., C. Schmidmeier, K. P. Drapala, J. A. O'Mahony, and A. L. Kelly. 2017. Monitoring of pilot-scale induction processes for dairy powders using inline and offline approaches. J. Food Eng. 197:9–16. https://doi.org/10.1016/j.jfoodeng.2016.10.023.
- Ostertagová, E., O. Ostertag, and J. Kováč. 2014. Methodology and application of the Kruskal-Wallis test. Appl. Mech. Mater. 611:115–120. https://doi.org/10.4028/www.scientific.net/AMM .611.115.
- Phosanam, A., J. Chandrapala, T. Huppertz, B. Adhikari, and B. Zisu. 2020a. Changes in physicochemical and surface characteristics in milk protein powders during storage. Dry. Technol. 40:638–652. https://doi.org/10.1080/07373937.2020.1824189.
- Phosanam, A., J. Chandrapala, T. Huppertz, B. Adhikari, and B. Zisu. 2020b. Effect of storage conditions on physicochemical and microstructural properties of skim and whole milk powders. Powder Technol. 372:394–403. https://doi.org/10.1016/j.powtec.2020.06.020.
- Phosanam, A., J. Chandrapala, T. Huppertz, B. Adhikari, and B. Zisu. 2021. Changes in physicochemical and surface characteristics in model infant milk formula powder (IMF) during storage. Dry. Technol. 39:2119–2129. https://doi.org/10.1080/07373937.2020 1755978
- Pugliese, A., G. Cabassi, E. Chiavaro, M. Paciulli, E. Carini, and G. Mucchetti. 2017. Physical characterization of whole and skim dried milk powders. J. Food Sci. Technol. 54:3433–3442. https://doi.org/10.1007/s13197-017-2795-1.
- Puri, V., A. K. Dantuluri, M. Kumar, N. Karar, and A. K. Bansal. 2010. Wettability and surface chemistry of crystalline and amorphous forms of a poorly water soluble drug. Eur. J. Pharm. Sci. 40:84-93. https://doi.org/10.1016/j.ejps.2010.03.003.
- Radaeva, I. A., E. E. Illarionova, S. N. Turovskaya, A. E. Ryabova, and A. G. Galstyan. 2019. Domestic dried milk quality assurance principles. Food Ind. 9:54–57. https://doi.org/10.24411/0235-2486-2019-10145. [in Russian]

- Rasch, D., and R. Verdooren. 2020. Determination of Minimum and maximum experimental size in one-, two- and three-way ANOVA with fixed and mixed models by R. J. Stat. Theory Pract. 14:57. https://doi.org/10.1007/s42519-020-00088-6.
- Rodnina, N. V. 2022. On food security of the Northern and Arctic regions of Russia (using the example of the Republic of Sakha (Yakutia). IOP Conf. Ser. Earth Environ. Sci. 988:042052. https://doi.org/10.1088/1755-1315/988/4/042052.
- Roos, Y. H. 2010. Crystallization, collapse, and glass transition in low-water food systems. Pages 335–351 in Water Properties in Food, Health, Pharmaceutical and Biological Systems: ISOPOW 10. Wiley-Blackwell. https://doi.org/10.1002/9780470958193.ch25.
- Rozhkova, A., and J. Olentsova. 2020. Development of New Technological Solutions for the Dairy Industry. E3S Web of Conferences 161:01086. https://doi.org/10.1051/e3sconf/202016101086.
- Russian National Inspectorate. 1987. Terms of Sale, Temperature and other Storage Conditions for Basic Retail Food [in Russian].
- Selomulya, C., and Y. Fang. 2013. Food powder rehydration. Pages 379–408 in Handbook of Food Powders: Processes and Properties. Woodhead Publishing. https://doi.org/10.1533/9780857098672.2 .379.
- Sert, D., and E. Mercan. 2021. Assessment of powder flow, functional and microbiological characteristics of ozone-treated skim milk powder. Int. Dairy J. 121:105121. https://doi.org/10.1016/j.idairyj .2021.105121.
- Sharma, A., A. H. Jana, and R. S. Chavan. 2012. Functionality of milk powders and milk-based powders for end use applications-A review. Compr. Rev. Food Sci. Food Saf. 11:518–528. https://doi .org/10.1111/j.1541-4337.2012.00199.x.Silalai, N., and Y. H. Roos. 2010. Roles of water and solids composi-
- Silalai, N., and Y. H. Roos. 2010. Roles of water and solids composition in the control of glass transition and stickiness of milk powders. J. Food Sci. 75:E285–E296. https://doi.org/10.1111/j.1750-3841.2010.01652.x.
- Singh, H. 2004. Heat stability of milk. Int. J. Dairy Technol. 57:111-119. https://doi.org/10.1111/j.1471-0307.2004.00143.x.
- Singh, J., S. Prakash, B. Bhandari, and N. Bansal. 2019. Comparison of ultra high temperature (UHT) stability of high protein milk dispersions prepared from milk protein concentrate (MPC) and conventional low heat skimmed milk powder (SMP). J. Food Eng. 246:86–94. https://doi.org/10.1016/j.jfoodeng.2018.11.003.
- Skanderby, M., V. Westergaard, A. Partridge, and D. D. Muir. 2009. Dried milk products. Pages 180–234 in Dairy Powders and Concentrated Products. A. Y. Tamime, ed. Blackwell Publishing Ltd. https://doi.org/10.1002/9781444322729.ch5.
- Smith, T. J., R. E. Campbell, Y. Jo, and M. A. Drake. 2016. Flavor and stability of milk proteins. J. Dairy Sci. 99:4325–4346. https://doi.org/10.3168/jds.2016-10847.

- Tamime, A. Y. 2009. Dairy Powders and Concentrated Products. Blackwell Publishing Ltd. https://doi.org/10.1002/9781444322729.
- Thomsen, M. K., L. Lauridsen, L. H. Skibsted, and J. Risbo. 2005. Temperature effect on lactose crystallization, Maillard reactions, and lipid oxidation in whole milk powder. J. Agric. Food Chem. 53:7082-7090. https://doi.org/10.1021/jf050862p.
- Timlin, M., J. T. Tobin, A. Brodkorb, E. G. Murphy, P. Dillon, D. Hennessy, M. O'donovan, K. M. Pierce, and T. F. O'callaghan. 2021. The impact of seasonality in pasture-based production systems on milk composition and functionality. Foods 10:607. https://doi.org/10.3390/foods10030607.
- TR TS 033/2013. 2013. Custom Union Regulation "Safety of Milk and Dairy Products." Gosstandart.
- US Dairy Export Council. n.d. Shelf Life and Packaging | Dairy for Global Nutrition. Accessed Jun. 6, 2022. https://www.dairyglobalnutrition.org/safety-and-quality/shelf-life-and-packaging.
- Vafin, R. R., I. A. Radaeva, A. G. Kruchinin, E. E. Illarionova, A. V. Bigaeva, S. N. Turovskaya, G. A. Belozerov, K. K. Gilmanov, and E. A. Yurova. 2021. K-casein polymorphism effect on technological properties of dried milk. Foods and Raw Materials 9:95–105. https://doi.org/10.21603/2308-4057-2021-1-95-105.
- Wu, J., S. Chen, L. Nyiransabimana, E. J. M. Van Damme, B. De Meulenaer, and P. Van der Meeren. 2022. Improved heat stability of recombined filled evaporated milk emulsions by wet heat pretreatment of skim milk powder dispersions at different pH values. Lebensm. Wiss. Technol. 154:112739. https://doi.org/10.1016/j .lwt.2021.112739.
- Yadav, N., A. Kumari, A. K. Chauhan, and T. Verma. 2021. Development of functional candy with banana, ginger and skim milk powder as a source of phenolics and antioxidants. Curr. Res. Nutr. Food Sci. 9:855–865. https://doi.org/10.12944/CRNFSJ.9.3.13.
- Yang, Y., A. S. Sekhon, A. Singh, P. Unger, and M. Michael. 2022. Survival and thermal resistance of *Listeria monocytogenes* in dry and hydrated nonfat dry milk and whole milk powder during extended storage. Int. Dairy J. 129:105338. https://doi.org/10.1016/ j.idairyj.2022.105338.
- Zhang, L. X., F. Koroma, M. L. Fofana, A. O. Barry, S. Diallo, J. Lamilé Songbono, R. Stokes-Walters, R. D. Klemm, S. Nordhagen, and P. J. Winch. 2020. Food security in artisanal mining communities: An exploration of rural markets in Northern Guinea. Foods 9:479. https://doi.org/10.3390/foods9040479.
- Zhou, Z., and T. Langrish. 2021. A review of Maillard reactions in spray dryers. J. Food Eng. 305:110615. https://doi.org/10.1016/j .jfoodeng.2021.110615.