COMPARISON OF THE SORPTION PROPERTIES OF MILK POWDER WITH LACTOSE AND WITHOUT LACTOSE

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Abstract. The powdered milk market is determined by consumer demand. Studies have shown that up to 37% of Polish society has problems with lactose digestion. In Poland, approximately 1.5% of infants and children and 30-37% of adults suffer due to a lack of β-D-galactosidase. Only lactose-free milk may replace mother's milk in infants with this disorder. Knowledge of powdered milk properties is the basis for ensuring safety at each stage of a product's life, which is of particular importance for infants – a very sensitive group of consumers. The objective of the paper was a comparative assessment of the sorption properties of full-fat lactose-free powdered milk (MB) and a standard variety (ML) of the same brand. It was assumed that modified powdered milk without lactose would substantially influence both the functional properties and utility characteristics of powdered milk. The experiment was carried out with *Enfamil Premium 1* infant milk (abbreviated as ML) and *Enfamil 0-Lac* (abbreviated as MB) purchased in a pharmacy located in Gdynia. The study included the following components: measurements of water content and water activity, mapping adsorption isotherms at 20°C and 30°C, mathematical description of adsorption properties with GAB model, estimation of specific surface area and of dimensions and capacity of capillaries in the tested material. The examined products differed in their chemical composition, including water content. A different chemical composition substantially impacted the sorption properties of the investigated powdered milk varieties. The replacement of lactose with glucose syrup in the MB product eliminated the crystallisation phenomenon and determined the integrity of isotherms throughout the entire water activity range. The tested products also differed in microstructural surface parameters which determined the powdered milk utility characteristics affecting susceptibility to rehydration and shelf-life.

Keywords: adsorption isotherms, monolayer, specific adsorption surface, water activity, Guggenheim, Anderson and De Boer (GAB) equation

INTRODUCTION

In humans, good health status and proper functioning of the body mainly depend on lifestyle which includes also optimal nutrition with food of adequate quality (Trafalska and Grzybowska 2004). Therefore, people suffering from lac-

tose intolerance are forced to eliminate milk and milk products from their diet or to replace them with different products (Ziarno 2006). The necessity of consuming modified milk products is difficult due to a problem with their availability or risks connected with possible unexpected effects after consumption of a product with unnatural properties. However, the prerequisite of food, especially health-promoting products, must be consumer safety (Czapski 2012).

Lactose intolerance is a well-studied food intolerance. It may be of a primary and genetically determined nature or secondary, acquired and caused by, for instance, gastritis and enteritis. Epidemiological data indicate that lactose intolerance affects 1.5% of infants and children and 30-37% of the adult Polish population (Ziarno 2006, Danków *et al.* 2009). In infants, this problem is particularly important as it presents substantial feeding limitations. In response to the existing needs, lactose-free powdered milk with desired health properties, categorised as functional food, has been introduced on the market (Trafalska and Grzybowska 2004). Lactose-free powdered milk may replace mother's milk for infants suffering from this pathological condition provided this product is safe at each stage of life, which is of particular importance for this very sensitive group of consumers.

Developing new food products, especially those with health properties, should include a comprehensive approach to the quality of such products. Quality should be perceived holistically, considering the fact that a change in one of its attributes may alter the others. A desired health effect, such as the elimination of an intolerance factor, should not deteriorate utility characteristics associated with, for example, an increase in sensitivity to storage conditions leading to reduced shelf-life. Only the achievement of all of the above prerequisites may be regarded as health-promoting activities in the context of food technology innovation.

The objective of the study was a comparative assessment of the sorption properties of full-fat, lactose-free powdered milk (MB) and a standard variety (ML). It was assumed that a modification of powdered milk involving the elimination of lactose would significantly impact both the functional properties and the utility characteristics of powdered milk, determined by their sorption capacity.

MATERIALS AND METHODS

The experiment was carried out with *Enfamil Premium 1* infant milk (abbreviated as ML) and *Enfamil 0-Lac* (abbreviated as MB), that were produced in the Netherlands by Mead Johanson B.V. Material for the study, which was purchased at a pharmacy located in Gdynia, was diverse in terms of chemical composition (Tab. 1).

Table 1. Composition of the examined powdered milk varieties

Enfamil O-Lac	Glucose syrup, calcium phosphate, sodium citrate, magnesium phosphate, inositol, manganese sulphate, riboflavin, potassium iodide, sodium molybdate, chromium chloride
Ingredients contained in both products	Whey protein concentrate (milk-derived), vegetable oils (palm oil, coconut oil, soy oil, high-oleic sunflower oil), calcium carbonate, sodium L-ascorbate, choline chloride, emulsifier (soy lecithin), potassium chloride, oils derived from single-cell organisms (ARA from Mortierella alpina, DHA from Crypthecodinium cohnii), potassium citrate, sodium selenite, D-biotin, thiamine hydrochloride, pyridoxine hydrochloride, folic acid, iron (II) sulphate, taurine, L-carnitine, zinc sulphate, DL-alpha-tocopherol acetate, phytomenadione, antioxidant (ascorbyl palmitate), retinyl palmitate, nicotinamide, cholecalciferol, calcium D-pantothenate, copper sulphate
Enfamil Premium 1	Lactose, calcium hydroxide, magnesium chloride, calcium chloride, citrate-5-monophosphate, uridine-5-monophosphate, adenosine-5-monophosphate, guasine-5-monophosphate, magnesium sulphate

Source: based on the data submitted by the manufacturer and specified on the outer packaging

The measurements of water content were taken by drying a sample of approximately 2 grams in weighing vessels of approximately 35 mm in diameter at 100°C until a fixed mass was obtained. The content of water in grams was calculated per 100 g of dry matter (DM).

The water activity was evaluated in AquaLab (Series 3, TE model, Decagon Devices, Inc., Pullman, WA, USA) with an accuracy of ± 0.003 at 293.15 K.

The results of measuring the water content and the water activity were the average of three parallel repetitions.

The adsorption isotherms were determined with the static-desiccator method using saturated solutions of the corresponding salts. The scope of studies included water activity from 0.07 to 0.98. The temperature was 293.15 K (20°C) and 303.15 K (30°C). The milk powder samples designated for the determination of adsorption isotherms were placed in 35 mm diameter weighing vessels in order to uniformly cover the whole surface of a vessel. Then the samples were placed in a desiccator containing P_2O_5 as a drying compound, at room temperature for 3 weeks in order to minimise their humidity to about 2%. The samples were weighed to an accuracy of 0.0001 g and placed in a desiccator with a saturated solution. In this way, only the adsorption process was studied. Thymol was placed in the desiccators with water activity >0.7 in order to protect samples against the growth of microorganisms. The time required to achieve dynamic equilibrium between the test samples and the environment amounted to 30 days. The equilibrium water content was calculated

based on the initial mass of product and the changes of water content. The water activity of the samples after incubation and achieved dynamic equilibrium was assayed with AquaLab. Each of the experimentally determined points of the ad-sorption isotherm was the average of three parallel repetitions.

Then estimation was made of parameters of the GAB equation in the form:

$$v = \frac{v_m C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)} \qquad (1)$$

where:

a_w – water activity (–);

v – equilibrium water content (g H_2O (100 g d.m.)$^{-1}$);

v_m – water content in the monolayer (g H_2O (100 g d.m.)$^{-1}$);

C – Guggenheim energy constant;

K – constant correcting properties of multilayer molecules with relation to the liquid phase (Paderewski 1999, Figura and Teixeira 2007).

The identification was carried out on the basis of non-linear regression with a Monte Carlo algorithm. Minimisation of residual sum of squares was adopted as the target function (Ocieczek and Kostek 2009). The calculations were performed using MS Excel 2003. The errors of determined equation parameters were detected with the SolverAid macro command.

The adsorbent specific surface was calculated based on the following equation:

$$a_{sp} = \omega \frac{v_m}{M} N \qquad (2)$$

where:

a_{sp} – sorption specific surface (m^2 (g d.m.)$^{-1}$);

N – Avogadro number (6.023·10^{23} molecules mol^{-1});

M – water molecular weight (18 g mol^{-1});

ω – water setting surface (1.05·10^{-19} m^2 molecule^{-1}) (Paderewski 1999).

The size and volume of capillaries of the material under test were determined in the capillary condensation area by the Kelvin equation, assuming their cylindrical shape:

$$\ln a_w = -\frac{2\sigma V}{r_k RT} \qquad (3)$$

where:

σ – liquid surface tension at temp. T (N m^{-1});

r_k – capillary radius (nm);

R – gas constant (kJ (mol K)$^{-1}$)
T – temperature of process (K);
V – molar volume of adsorbate (m^3 mol^{-1}) (Paderewski 1999, Figura and Teixeira 2007).

DISCUSSION

The content of water in the tested products, measured after opening the individual packaging units, ranged from 2.7514 g H$_2$O (100 g DM)$^{-1}$ (ML) to 3.0140 g H$_2$O (100 g DM)$^{-1}$ (MB). The water activity in these products ranged between 0.183 (MB) and 0.186 (ML). The interrelation of water content and its activity indicated that the fraction of free water in ML was more loosely bound with the matrix of the product than the fraction of free water in MB. It may be assumed that the reason for the stronger bond of water with the MB matrix was the replacement of lactose with glucose syrup which may contain from 11% (after acidic hydrolysis) to 97% (after enzymatic hydrolysis) of glucose.

Low levels of water activity in both products stored in airtight packaging ensure their microbiological safety and substantially long shelf-life. Stapelfeldt *et al.* (1997) indicated that the optimal range of water activity for long-stored powdered milk should be between 0.11 and 0.23, depending on its type. In the case of such low a_w, oxidation of lipids is very slow and similar to the processes of non-enzymatic browning. The enzymatic activity starts at $a_w = 0.3$. The growth of microorganisms becomes possible above a threshold at $a_w = 0.6$ (Pałacha 2008).

The isotherms of water vapour adsorption for both tested powdered milk samples had a type II course according to BET classification (Fig. 1 and 2). This shape clearly indicated that a monomolecular layer was formed during adsorption phenomenon (a_w below 0.30) and it was gradually super-built in a process of multilayer adsorption ($0.30 < a_w < 0.65$) that led, in some areas of the matrix, to filling the capillaries and capillary condensation (a_w above 0.65).

An initial assessment of shape and location of water vapour adsorption isotherms for the tested powdered milk varieties confirmed their complex and diversified chemical composition (Pałacha and Sitkiewicz 2010), the occurrence of crystallisation in the ML product and the capillary nature of the surface of particles in the tested powdered milk samples that determined the occurrence of capillary condensation with a mechanism comparable to condensation.

The isotherms for ML determined at 20°C and 30°C in an area that approximated the second zone of sorption curve inflexion (0.4-0.5) were discontinuous. A possible reason for the discontinuity was a phase transformation of lactose as one of the most important milk constituents. Storage stability and its derivative, i.e. the nutritional quality of powdered milk, largely depend on the physical state

of lactose which may be amorphous or crystalline. As a result of the rapid remov-
al of water from milk during drying, fully-hydrated lactose in an amorphous state
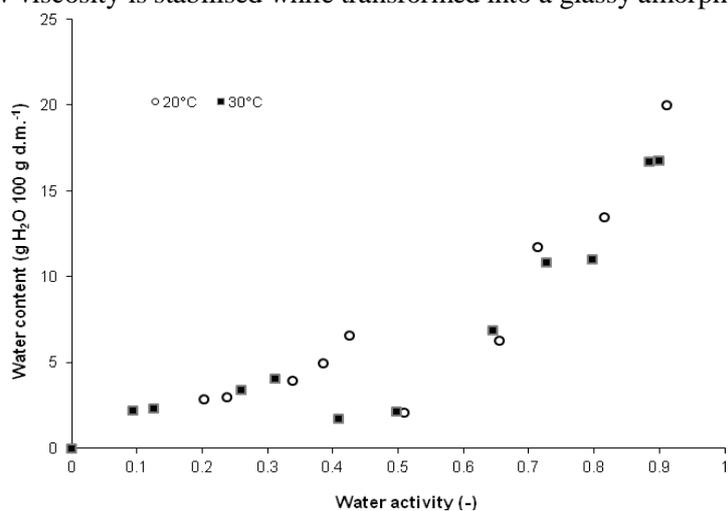and of low viscosity is stabilised while transformed into a glassy amorphous state.

Fig. 1. Water vapour adsorption isotherms for lactose-containing powdered milk (ML) determined
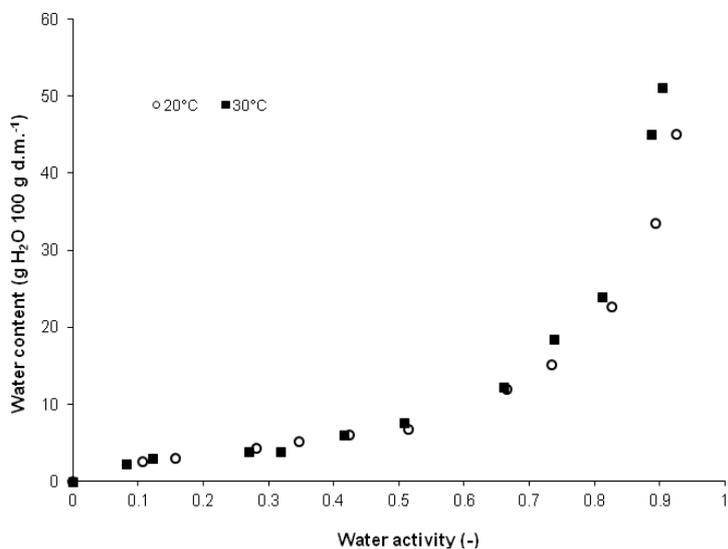at 20° and 30°C

Fig. 2. Water vapour adsorption isotherms for lactose-free powdered milk (MB) determined at 20°
and 30°C

This state is unstable at temperatures below the temperature of glass transition (T_g). Hydration of a product or exceeding the T_g temperature is a factor that favours glass transition and crystallisation of lactose. Under such conditions, a glassy state transforms into a viscous-elastic state, which is also called rubber-like, which results in a substantial increase in the mobility of lactose particles and leads to its gradual crystallisation (Olkowski *et al.* 2012). The occurrence of crystalline lactose results in a balance between water content and its activity being set at a new level, which is reflected in a disruption of the continuity of a sorption isotherm. The phase transformation of lactose causes substantial changes in the nutritional quality of powdered milk due to an increase in the oxidation rate of other milk constituents, for instance milk fat, non-enzymatic browning and a reduced capacity for rehydration (Fitzpatrick *et al.* 2007). As a result of lactose crystallisation, water is released, which favours lactose hydrolysis to glucose and galactose.

The empirical data were used to determine the parameters of the GAB equation (v_m, C, K) and a_w values corresponding to v_m. The magnitude of errors with which the parameters of the GAB equation were estimated was calculated using Hesse's matrix. This matrix is a square matrix of the second partial derivatives of a function with actual values that are double-differentiated at a certain point of a domain. The inverse of Hesse's matrix approximates the variance/co-variance matrix of estimator parameters. It thus indicates an inverse relation between the second rank derivative for a parameter and its standard error. If a change in the slope of a function around its minimum is very large, the second rank derivative will also be large. The estimator of a parameter will be, however, quite steady in a sense that the minimum is clearly identifiable. If the second rank derivative approximates zero, a change in the slope around the minimum equals zero, which implies that a parameter can be carried over in any direction without any greater impact on the loss function. Therefore, the standard error of the parameter will be very high. The recorded results are presented in Table 2.

According to Lewicki (1997), the GAB equation describes experimental data well when $0.24 < K \leq 1$ and $5.67 < C \leq \infty$. The values of K and C indicate that the conditions were met. It may be assumed that the values of parameters generated with the applied calculation procedures did not lose a physical sense, which was emphasised by Pérez-Alonso *et al.* (2006). Hence, the calculation procedure was continued and v_m values were determined together with corresponding a_w values.

The magnitude of standard errors with which the individual parameters of GAB equation were determined had relatively low values compared to the values of the parameters themselves (Tab. 2).

The capacity of the monolayer serves to determine the sorption capacity of adsorbents and is used as an indicator of the availability of polar positions for water vapour. It is determined by the amount of constituents rich in polar positions and their physical status (Mathlouthi 2001). The determined v_m values

ranged between 2.66±0.50 and 4.58±0.15 g H_2O per 100 g of a product's DM (ML 30°C and MB 20°C, respectively) and were within the range established by Karel (1975) for different food products. MB had a higher monolayer capacity. The differences between MB and ML in the extent of v_m expansion were probably caused by substantial differences in the chemical composition of these products. In ML, lactose is a source of sugar in an amorphous state. Even a slight absorption of water results in increased mobility of amorphous sugar particles. This allows for its transition from an amorphous state into a crystalline state and reduces its physical and utility characteristics and the powdered milk shelf-life. These changes are reflected in the sorption curves for ML powdered milk depicted in Figure 1; they present the discontinuity of the isotherm caused by the transition of lactose from an amorphous into a crystalline state.

Table 2. Parameters of the GAB equation for the tested powdered milk varieties and a_w corresponding to v_m

Product	*SSD*	Parameters			
		v_m	a_w	*C*	*K*
		20°C			
ML	26.49±1.82	3.86±1.38	0.335	5.74±7.84	0.88±0.07
		30°C			
	18.54±1.52	2.66±0.50	0.216	14.70±22.87	0.96±0.03
		20°C			
MB	1.69±0.46	4.58±0.15	0.303	5.77±1.16	0.97±0.00
		30°C			
	3.99±0.71	4.37±0.17	0.267	7.15±2.29	1.02±0.00

Keys:
ML – lactose-containing milk,
MB – lactose-free milk,
SSD – sum of square deviations,
v_m – water content in the monolayer (g H_2O (100 g DM)$^{-1}$),
a_w – water activity (-),
C – Guggenheim energy constant,
K – constant which corrects the properties of multi-layer particles compared to a liquid phase.

The increase of sorption temperature resulted in a reduction of the capacity of the mononuclear layer in both products. Although this phenomenon should be attributed to an exothermic nature of sorption, an increase in temperature by 10°C reduced the capacity of v_m more in ML than in MB. A significant role is attributed to glucose syrup found in the composition of powdered milk. This ingredient was

a substitute for lactose as a factor that triggers milk intolerance; it determines the sweet taste of the product and affects the physical and sorption properties of powdered milk.

Glucose syrup is a purified and concentrated solution (up to 80%) of glucose (dextrose) and maltodextrin in water. It is usually produced by means of enzymatic hydrolysis of starch which is a polymer composed of glucose. It has a mildly sweet taste, high osmotic pressure and good temperature stability. Glucose syrup can be stored for a prolonged period of time at an ambient temperature without concern for crystallisation, which determines the stability of physical properties and, consequently, the nutritional values of powdered milk.

The values of a_w corresponding to v_m confirmed that water activity was a function not only of the content of water but also of the interactions between the surface and water, condensation of water vapour in capillaries, and concentration and type of water-soluble substances (Czerniawski and Michniewicz 1998) that depend on the chemical composition and physical structure of a solid matrix.

The values of K index ranging from 0.84 to 1.00 confirm that proteins played a predominant role in the development of sorption properties in the tested products. According to Pérez-Alonso *et al.* (2006), K values between 0.717 and 0.893 are typical of a product with a substantial fraction of fibre.

The constant C with values over 5.67 indicate that the sorption isotherms for the examined products should be categorised as type II curves.

Each porous material can be characterised with values describing its microstructure. These parameters include sorption specific surface, total capacity of capillaries and radius of capillaries that are filled when capillary condensation is initiated. A high sorption specific surface ($a_{sp} > 500$ m^2 g^{-1}) indicates the presence of narrow pores (there is an inverse relation between specific surfaces and radii of pores), whereas a low value of sorption specific surface ($a_{sp} < 10$ m^2 g^{-1}) is typical of macroporous solids (Paderewski 1999). According to IUPAC classification, pores from 2 to 50 nm are mesopores. Products with a high degree of comminution and high porosity are strongly hygroscopic and thus adsorption may be very intense.

Based on Kelvin's equation, the microstructure of particles of the tested powdered milk varieties was evaluated by determining the total capacity of capillaries at $a_w = 0.70$ and identifying the radius of capillaries that are filled during the initial phase of condensation. The sorption specific surface was then calculated based on v_m.

The recorded results (Tab. 3) indicate that following the initiation of capillary condensation in MB, mesocapillaries were filled, while in ML these were microcapillaries. The examined products also differed in sorption specific surface that was reduced as a result of increasing temperature in both cases. The MB product had a substantially higher total capacity of capillaries in comparison with ML, which was

probably caused by a higher number of capillaries (as indicated by a large specific surface) with a greater radius.

The type of raw materials was the most probable source of diversity in the microstructure surface of the tested powdered milk varieties (Tab. 3) and, consequently, the quantitative and qualitative diversity of the chemical composition and the applied technological process. Hence, purposely changed nutritional values and a clear diversification of sorption properties were a result of such modifications.

Table 3. Microstructural characteristics of the tested powdered milk

Product	Sorption specific surface ($m^2 g^{-1}$ DM)		Total capacity of capillaries (mm^3 100 g^{-1} DM)		Size of capillaries at a_w= 0.70 (nm)	
	20°C	30°C	20°C	30°C	20°C	30°C
ML	135.7	93.32	57.48	54.85	1.51	1.23
MB	161.9	153.4	112.8	139.4	2.21	2.29

Keys:
ML – lactose-containing milk, MB – lactose-free milk.

CONCLUSIONS

1. The content of water in the tested products ranged from 2.75 g H_2O (100 g DM)$^{-1}$ (ML) to 3.01 g H_2O (100 g DM)$^{-1}$ (MB), protecting them against microbial growth and limiting, to a large extent, enzymatic and non-enzymatic transformations.

2. The sorption curves for ML and MB powdered milk samples were categorised as type II according to the classification by Brunauer and co-workers. The isotherms for lactose-containing milk (ML) were discontinuous as a result of lactose transition from an amorphous into a crystalline state, whereas the isotherms for lactose-free milk (MB) were continuous across the whole a_w range.

3. Lactose-free powdered milk with mesocapillary pores that are filled following the initiation of condensation had a higher monolayer capacity.

4. A substantial extension of the monolayer protects powdered milk against rapid spoiling which results from absorption of a specific volume of water. Considering the aforementioned, lactose-free milk had sorption properties that determined better storage stability in comparison with lactose-containing powdered milk.

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PORÓWNANIE WŁAŚCIWOŚCI SORPCYJNYCH PROSZKÓW MLECZNYCH LAKTOZOWYCH I BEZLAKTOZOWYCH

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Streszczenie. Rynek mleka w proszku uwarunkowany jest potrzebami odbiorców. Wyniki badań wskazują, że 37% polskiego społeczeństwa ma problem z trawieniem laktozy. Z powodu deficytu enzymu β-D-galaktozydazy cierpi w Polsce około 1,5% niemowląt i dzieci oraz 30-37% dorosłych. Jedynie bezlaktozowe mleko w proszku może zastąpić mleko matki niemowlętom z tym schorzeniem. Znajomość właściwości mleka w proszku jest podstawą zapewnienia jego bezpieczeństwa na każdym etapie życia produktu, co ma szczególne znaczenie dla bardzo wrażliwej grupy konsumentów jakimi są niemowlęta. Celem pracy była porównawcza ocena właściwości sorpcyjnych pełnego mleka w proszku bezlaktozowago (MB) i standardowego (ML) tej samej marki. Przyjęto założenie, że modyfikacja proszku mlecznego poprzez wyeliminowanie laktozy wpłynie znacząco nie tylko na właściwości funkcjonalne ale również użytkowe proszku mlecznego. Materiał badawczy stanowiły: *Enfamil Premium 1* mleko początkowe (oznaczone jako ML) oraz *Enfamil 0-lac* (oznaczone jako MB), które zakupiono w aptece na terenie Gdyni. Metody badań obejmowały oznaczenie zawartości wody i aktywności wody, wyznaczenie izoterm adsorpcji w temperaturze 20 i 30°C, matematyczny opis właściwości adsorpcyjnych z wykorzystaniem modelu GAB, oszacowanie powierzchni właściwej oraz rozmiarów i objętości kapilar badanego materiału. Badane produkty różniły się składem chemicznym, w tym zawartością wody. Różnorodny skład chemiczny miał znaczący wpływ na właściwości sorpcyjne badanych proszków mlecznych. Zastąpienie laktozy syropem glukozowym w produkcie MB spowodowało wyeliminowanie zjawiska krystalizacji i zdeterminowało ciągłość przebiegu izoterm adsorpcji w całym zakresie aktywności wody. Badane produkty różniły się także mikrostrukturalnymi parametrami powierzchni, które decydowały o właściwościach użytkowych proszków mlecznych, wpływających na ich podatność na rehydrację i trwałość przechowalniczą.

Słowa kluczowe: izotermy sorpcji, monowarstwa, powierzchnia właściwa sorpcji, aktywność wody, równanie Guggenheima, Andersona i De Boera (GAB)