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Comparative study of the granulometric composition of seed materials used during the lactose crystallization

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Abstract. *The properties of many dairy products with high levels of dry matter and lactose directly depend on the crystallization of lactose, which is a key component. To control these properties and achieve the desired parameters, the process of lactose crystallization is often used. In this paper, data on the granulometric composition of seeding materials used to manage the lactose crystallization process in dairy raw materials are presented. Measurements of particle sizes are performed by laser diffraction on microcrystalline lactose, powdered sugar, and a liquid crystallizer based on sucrose. It was found that the seeding materials have different particle sizes. The liquid crystallizer is distinguished by its greater uniformity and convenience of dosing, but its use does not significantly affect the crystallization rate.*

Keywords: lactose, seed material, crystallization, granulometric composition

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Сравнительное исследование гранулометрического состава затравочных материалов, использующихся при кристаллизации лактозы

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Аннотация. *В молочных продуктах, характеризующихся высокой массовой долей сухих веществ и значительным содержанием лактозы в сухом остатке, свойства продуктов в значительной степени обусловлены степенью кристаллизации и размером кристаллов лактозы, являющейся одним из ключевых компонентов молочного сырья. С целью обеспечения заданных характеристик в производственных процессах зачастую применяется кристаллизация лактозы. Настоящая статья содержит сведения о гранулометрическом составе затравочных материалов, полученные в результате лазерного дифракционного анализа. Представлена теоретическая модель для выбора подходящего материала, а также экспериментальные данные, подтверждающие ее соответствие. В качестве объектов исследования были выбраны мелкокристаллическая лактоза, сахарная пудра и жидкий кристаллообразователь на основе сахарозы. Показано, что затравочные материалы отличаются по размерам частиц, при этом жидкий кристаллообразователь характеризуется наибольшей однородностью и удобством дозирования, хотя его использование не приводит к существенным изменениям в динамике кристаллизации.*

Ключевые слова: лактоза, затравочный материал, кристаллизация, гранулометрический состав

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Introduction. Lactose is one of the main components of dairy raw materials, therefore its phase state, or more precisely the degree of crystallization and the size of the crystals, have a great influence on the properties of many dairy products, especially with a high mass fraction of dry matter and a significant lactose content in the dry residue [1–4].

The type of product determines the required size of lactose crystals and, accordingly, the modes of its crystallization. For example, lactose crystals larger than 15 μm are the cause of the consistency defect of condensed milk with sugar. When producing dry whey and dry permeate, it is generally recommended to crystallize lactose in the condensed product before drying until crystals are obtained, most of which are 50–100 μm in size. This allows obtaining a non-hygroscopic product in which crystalline lactose will predominate [1]. In turn, when producing crystalline lactose, the crystallization process is aimed at obtaining sufficiently large crystals for a more complete separation of the crystalline fraction from molasses.

In industry, the process of lactose crystallization is usually implemented by controlled cooling of the thickened raw material using modes that allow obtaining crystals of the required sizes with the highest possible degree of lactose crystallization.

Thus, when preparing lactose-containing raw materials for spray drying, the cooling regime includes an initial period of rapid cooling to approximately 30 °C, followed by a period of slow cooling to approximately 15 °C [12–16]. Rapid cooling increases the rate of lactose crystal formation, while slow cooling promotes crystal growth [17]. The size of the forming lactose crystals is controlled by targeted adjustment of the cooling temperature regime parameters. In particular, smaller crystals are formed when rapid cooling to lower temperatures is performed [18–20]. With a long-term slow cooling regime, it is possible to obtain larger lactose crystals [21].

In addition to cooling conditions, the lactose crystallization process is also affected by other factors, including viscosity, temperature, pH and composition of the raw material, the presence of minerals, acids and other impurities, etc. [16, 17, 19]. The type of nucleation (primary or secondary) has a significant effect on the crystallization process. Homogeneous nucleation, characterized by spontaneous formation of nuclei, occurs when the temperature of the supersaturated solution decreases [9]. The presence of impurities in the solution containing lactose causes heterogeneous primary nucleation, in which lactose molecules are adsorbed on the surface of the impurities [14]. This process is characterized by reduced free energy required for nucleation

[9]. In addition to non-crystalline impurities, seed materials such as microcrystalline lactose and sucrose can act as centers of heterogeneous nucleation.

The size of the seed material crystals has a significant effect on the morphology and size of the lactose crystals formed in the product. Therefore, when choosing a crystal former, information on the granulometric distribution of the seed material can be one of the determining factors in the formation of crystals with specified size characteristics.

Materials and research methods. The purpose of the study of seed materials is to evaluate its granulometric composition, which allows choosing a crystal former that provides the process of lactose crystallization.

Theoretically, the criterion for selecting the seed material can be substantiated as follows. Let us assume that after introducing seed crystals into the thickened raw material, the process of crystallization of lactose from it occurs only on the surface of these crystals [5].

To develop a mathematical assessment model, we introduce the following notations:

– l , μm — average characteristic size of filling crystals;

– h , μm — average (desired) characteristic size of the final crystals;

– M , kg — mass of raw material - condensed permeate of cheese whey

– m , kg — mass of seed crystals;

– n – number of seed crystals;

– C , % — lactose concentration in raw materials;

– B , % — degree of crystallization of lactose

Research results and their discussion. During the crystallization process, the growth of seed crystals must be carried out in such a way that their final mass is equal to the mass of crystallized lactose, corresponding to its value for a given final cooling temperature of the raw material.

The mass of crystallized lactose $M_л$ can be expressed in two ways (formulas 1 and 2).

$$M_л = M \times C \times B, \quad (1)$$

$$M_л = n \times K \times h^3 - m, \quad (2)$$

where K is a proportionality coefficient depending on the density of lactose and its form factor at the final stage of crystallization.

Similarly for finely crystalline lactose

$$m = n \times k \times l^3, \quad (3)$$

The coefficients K and k differ in magnitude due to the difference in the values of the corresponding form factors. Indeed, for finely crystalline lactose, obtained, for example, using a colloid mill, the shape of the crystals may differ from the shape of the crystals at the finishing stage, as a result of which the ratio between size and volume may differ significantly for them.

Let us now consider the expression

$$\frac{m}{M_л} = \frac{n \times k \times l^3}{n \times K \times h^3 + n \times k \times l^3}, \quad (4)$$

Since by meaning $m \ll M_л$, then formula (4) can be simplified

$$\frac{m}{M_л} = \frac{n \times k \times l^3}{n \times K \times h^3} = \frac{k}{K} \times \left(\frac{l}{h}\right)^3, \quad (5)$$

The ratio $\frac{k}{K}$ depends only on the values of the form factors of lactose crystals at the initial and final parts of the crystallization process.

Using formula (1), expression (5) can be expressed as

$$\frac{m}{M_{л}} = \frac{m}{M \times C \times B} = \frac{k}{K} \times \left(\frac{l}{h}\right)^3, \quad (6)$$

Where

$$\frac{m}{M} = C \times B \times \left(\frac{k}{K}\right) \times \left(\frac{l}{h}\right)^3, \quad (7)$$

In formula (7) the most difficult parameter to determine is the form factor F

$$F = \frac{k}{K} \quad (8)$$

When used systematically as a seed of one type and one batch, the *F value* can be taken as a constant.

Then, using formula 7, the permissible value $l$can be expressed as follows:

$$l = \sqrt[3]{\frac{m \times h^3}{M \times C \times B \times F}}, \quad (9)$$

With a given percentage of introduction of seed material to the raw material (ratio m / M) of 0.01%, concentration of lactose in the raw material (C) of 55.0% and minimum degree of crystallization (B) of at least 80.0%, expression (9) will take the following form:

$$l = \sqrt[3]{\frac{0{,}0001 \times h^3}{0{,}55 \times 0{,}80 \times F}} = \frac{0{,}06 \times h}{\sqrt[3]{F}} \quad (10)$$

If we assume that the seed material used was not milled and is crystalline lactose, then F can be taken as close to one.

Thus, the proposed model allows for a preliminary assessment of the suitability of the seed material for use in the process of producing dairy products with preliminary crystallization of lactose.

For example, to obtain crystallized permeate with the crystal sizes recommended [23], h = *(50 – 100)* μm, the minimum size of seed crystals should be about 3.0 μm, and the maximum 6.0 μm. Accordingly, if the goal is to obtain larger lactose crystals, the maximum value of seed crystals can be larger. Obviously, the size of seed crystals will be especially critical if it is necessary to form small crystals. If the main target parameter is the maximum degree of crystallization, then when choosing a crystal former, the emphasis can be shifted to the functional and technological characteristics: homogeneity, granulometric composition of the product.

The objects of the study were seed materials used in studying the crystallization process under industrial conditions. Fine-crystalline lactose or fine-crystalline milk sugar are finely ground lactose crystals. This is a sweetish white, free-flowing powder without odor. According to GOST 33567-2015 "Milk sugar. Specifications", 70.0% of lactose crystals in the product should be (3 - 4) μm in size, the maximum size of individual crystals can reach 10 μm. Powdered sugar, according to GOST 33222-2015 "White sugar. Specifications", is finely ground sucrose crystals, the size of 95.0% of which crystals should not exceed 200 μm. This is a sweet white, free-flowing powder without odor, highly soluble in water. The liquid crystal former according to TU U 15.8-22942814-025:2006 is a suspension of homogeneous sucrose crystals in a liquid phase, stabilized by a food surfactant [6].

For a comparative assessment of crystal formers, taking into account the mathematical model developed above, a comparison was made of the granulometric composition of fine-crystalline lactose from two Russian manufacturers, Russian-made powdered sugar, and the imported liquid crystal former “ESTER-K”. A diagram of the comparative analysis of the granulometric composition according to the method [23] is shown in Figure 1.

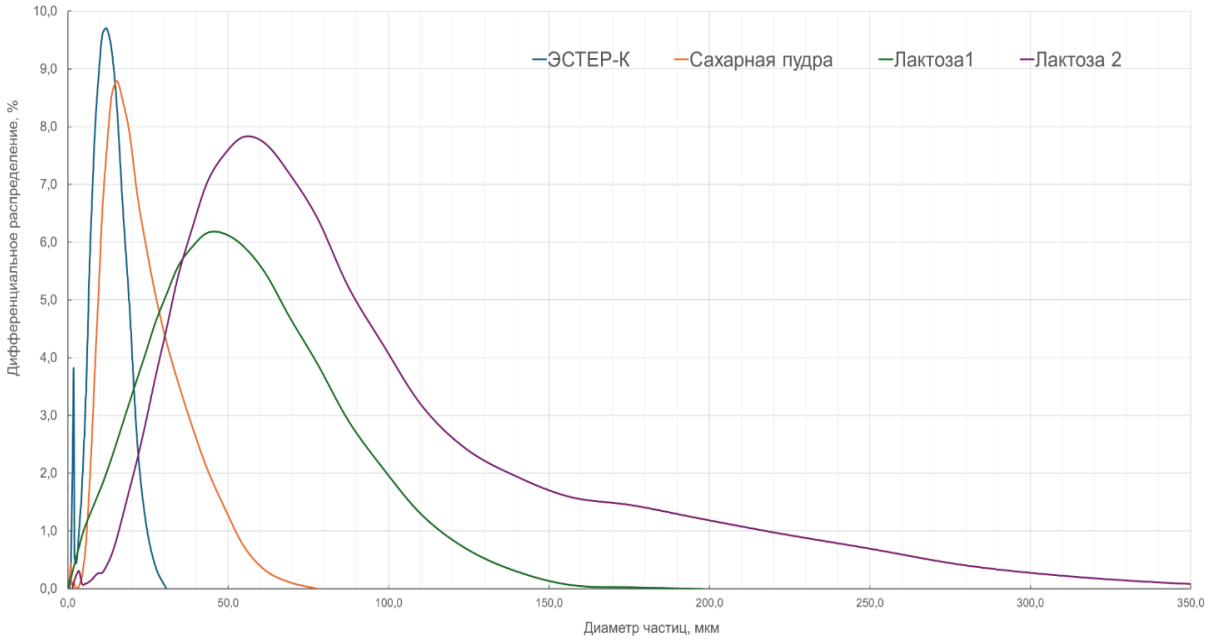

Figure 1 – Diagrams of differential distribution of seed material particles by size

According to the results of laser diffraction analysis (Fig. 1), the particle size distribution in all samples of crystal formers can be described as asymmetric, since the average diameter D_{50} and modal D_{mode} for all samples differ significantly ($p>0.05$). Thus, for fine-crystalline lactose sample 1, these parameters are: D_{50} = (31.37±0.23) µm, D_{mode} = (43.51±0.17) µm, for fine-crystalline lactose sample 2 - D_{50} = (50.87±0.28) µm, diameter – D_{mode} = (54.92±0.15) µm, for powdered sugar - D_{50} = (15.14±0.05) µm, diameter – D_{mode} = (13.58±0.13) µm, for liquid crystal former - D_{50} = (9.86±0.08) µm, diameter – D_{mode} = (10.77±0.12) µm.

For a more visual comparison, the diagram (Fig. 2) shows the characteristic particle sizes of each seed material: diameters characteristic of 90%, 50.0% and 10.0% differential particle distribution.

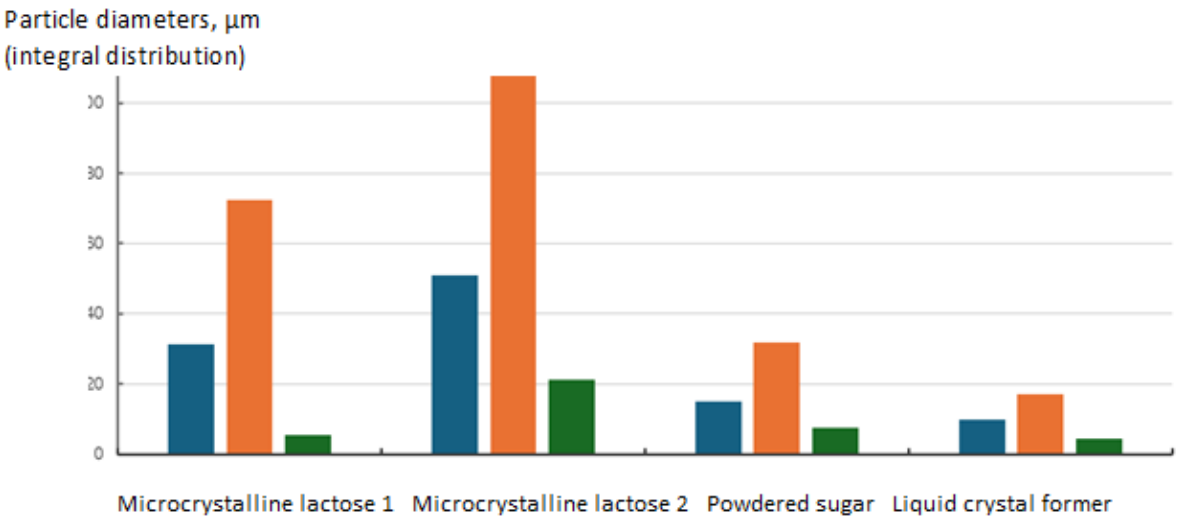

Figure 2 – Characteristic particle sizes of seed materials

The presented data show that the seed materials are quite heterogeneous in size. For example, D_{90} in the fine-crystalline lactose samples 2 exceeded the target sizes of lactose crystals recommended for the technologies of concentrated permeate and concentrated whey [1] . In sample

1, the particles approached 100.0 μm. At the same time, the sizes of most particles in these samples (Fig. 1) shifted to the region of (40.0 – 70.0) μm, and the proportion of particles with a size of up to 10.0 μm in fine-crystalline lactose sample 2 was about 3.0%. For comparison, this indicator for fine-crystalline lactose sample 1 was about 19.0%, and for the liquid crystal former about 48.0%. The smallest sizes of seed crystals (Fig. 1, Table 1) were in the liquid crystal former.

It can be noted that the results of a comparative assessment of the use of all three types of seed materials in the technology of dry permeate of spray drying [6] showed that the replacement of the crystal former did not lead to significant differences in the dynamics of the lactose crystallization process. This did not allow us to draw certain conclusions on the recommendations for the preferred use of one or another crystal former, but technically the use of a liquid crystal former was more convenient, since the liquid form allows better control of the dosage and introduction of the seed.

Conclusion. The analysis of the obtained data allows us to conclude that the most effective seed material for the nucleation of small lactose crystals is a liquid crystal former. However, even with the target task of obtaining large lactose crystals, the use of a liquid crystal former may be preferable due to the ease of administration and dosing accuracy. Due to the lack of domestic production of this seed material, the development of its analogue using lactose-containing raw materials seems to be a promising direction.

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