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LACTOSE CRYSTALLIZATION: CURRENT ISSUES AND PROMISING ENGINEERING SOLUTIONS

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Abstract: Current technological aspects of lactose crystallization are considered. A promising lactose crystallization method involving simulation seed crystals is reported. Advanced engineering solutions for continuous crystallization using spraying in vacuo and scraped-surface heat exchangers are presented.

Key words: lactose, heterogeneous and homogeneous crystallization, simulation seed crystals, continuous crystallization of lactose, scraped-surface plate-type cooling crystallizer, vacuum spray crystallizer.

INTRODUCTION

Lactose crystallization is among the necessary technological operations in the production of sweetened canned condensed milk. This operation consists of creating appropriate conditions for extensive formation of crystallization centers and for subsequent controllable crystal growth at certain processing parameters and under post-production storage conditions.

The lactose crystallization kinetics can be described in terms of a C = f(t) function, where *C* is the lactose concentration in the solution (%) and *t* is time (s). The corresponding crystallization curve can conventionally be divided into the following three segments: induction period, in which C = const; rapid increase in the concentration with time; slow variation of the concentration at the late stages of the process.

The first period is characterized by the formation of crystal nuclei; the second and third periods, by their growth. A nucleus (crystallization center) is the minimum amount of a new phase that is capable of independently existing [1, 10]. Once stable nuclei have formed in the new phase, they begin to grow. The main processes determining the crystal growth rate are the diffusion of the constituent particles to the surface of the growing crystal and their incorporation in the crystal lattice. In turn, the latter process includes the adsorption of particles by the surface, their migration on the surface, and their incorporation in the lattice as such.

The factors on which the crystal growth rate depends are the solution temperature, stirring intensity, the presence of impurities, degree of supersaturation, viscosity, etc. The effect of a given factor depends on crystallization conditions. For example, the variation of the crystal growth rate with the degree of supersaturation depends on whether the solution is stirred or not. On the whole, the value of supersaturation is so significant that its variation alters the growth mechanism [2–4, 9–11]. A large number of crystal growth theories have been devised to account for the complicated dependence of the growth rate on various factors. However, there is still no unified theory completely describing the multiformity of the crystallization process.

Contributions to classical crystallization theory were made by J. Gibbs, M. Volmer, W. Kossel, I.N. Stranskoi, and R. Kaishev. The theory is based on the thermodynamic conception that an isolated system is absolutely stable when its entropy is invariable [5].

The present-day technologies of production of sweetened condensed dairy products necessarily include the introduction of seed crystals for preventing the consistency defects arising from uncontrolled lactose crystallization. The seeds used in these technologies are microcrystalline lactose with a crystal size of 2-3 ?m, supersaturated solutions or suspensions of lactose containing crystal nuclei, and water-soluble crystalline macromolecular organic compounds mixed with lactose [6, 7].

The seed material is introduced at the enhanced crystallization temperature, which depends on the lactose concentration in the system. At this temperature, the maximum degree of lactose supersaturation is rapidly reached at a minimum increase in the viscosity of the milk. The enhanced crystallization temperature is determined from Hudson's plot shown in Fig. 1.

To determine the enhanced crystallization temperature of lactose, the weight fraction of lactose in the aqueous part of condensed milk (lactose number) is first determined. Next, the intersection point between the vertical line corresponding to this lactose weight fraction and the enhanced crystallization curve is found. This intersection point indicates the mass crystallization temperature in the ordinate axis.



Fig. 1. Plot for determining the enhanced lactose crystallization temperature.

The lactose weight fraction in condensed milk ($L_{\rm pr}$, %) is calculated via the formula

$$L = \frac{L_{\rm n} \times F_{\rm pr}}{F_{\rm n}},\tag{1}$$

where L_n is the lactose weight fraction in normalized milk (%), F_{pr} is the weight fraction of fat in the product (%), and F_n is the weight fraction of fat in normalized milk (%).

The lactose weight fraction in the aqueous part of condensed milk (lactose number, L_{con} , %) is calculated via the formula

$$L_{\rm con} = \frac{100 \times L_{\rm pr}}{L_{\rm pr} + W_{\rm pr}},\tag{2}$$

where L_{pr} is the lactose weight fraction in sweetened condensed milk (%) and W_{pr} is the water weight fraction in sweetened condensed milk (%).



Fig. 2. Microstructure and classical shape of crystals of (A) α-lactose monohydrate and (B) sucrose.

Figure 2 shows the lactose and sucrose crystal shapes.

Tables 1 and 2 list the values of crystallization efficiency criteria and lactose crystals settling velocity data, respectively.

Number of crystals in 1 mL^3	Average	Consistency of
of sweetened condensed milk,	particle	sweetened
thousands	size, ?m	condensed milk
400-300	≤10	Uniform
300-100	12-15	Slight floury
100–50	16-20	Floury
50-25	21-24	Strong floury
≤25	≥25	Sandy

 Table 1. Crystallization efficiency criteria

The mutarotation phenomenon takes place in lactose solutions. The lactose isomers are in dynamic equilibrium. α -Lactose turns into β -lactose via the tautomeric aldehyde form, which has a carbonyl group. α -Lactose crystallizes under commonly used process conditions, because it is less soluble than the β -form. β -Lactose begins to turn into α -lactose once the latter has

precipitated. This can be explained by the disturbance of the dynamic equilibrium between the isomers.

Table 2. Lactose crystals settling velocity in sweetened condensed whole milk as a function of viscosity and crystal size

Consistency	Viscosity, Pa.s	Crystal size, ?m	Settling velocity,	
Velvety (uniform)	2.0	10	0.0470(5.7)	
	3.0	10	0.0309 (8.6)	
	5.0	10	0.0188 (14.0)	
	10.0	10	0.00941 (28.6)	
	2.0	20	0.189 (1.4)	
Floury	3.0	20	0.124 (2.1)	
(nonuniform)	5.0	20	0.0754 (3.2)	
	10.0	20	0.0377 (7.0)	
Sandy	2.0	40	0.766 (0.33)	
	3.0	40	0.497 (0.53)	
	5.0	40	0.300 (0.86)	
	10.0	40	0.149 (1.8)	

* Sediment formation time in months

This process continues until the solution is completely exhausted. Even if seed crystals have been introduced, it is practically impossible to completely eliminate the supersaturation of the solution and to bring crystallization to completion at the product cooling stage, so lactose crystals continue growing during longterm storage at low temperatures, and this can lead to the formation of large crystals. Recrystallization processes consisting in the growth of larger crystals at the sacrifice of small crystals are also possible here. This recrystallization, which progressively spoils canned dairy products, most often occurs under uncontrolled temperature variations during storage [8, 11].

If the product is additionally heated after the introduction of seed crystals, the crystallization process will be inefficient. This technology is used in the production of boiled sweetened condensed milk.

Having analyzed the literature and internet sources dealing with crystallization, including the crystallization of salts, various alloys, and biological fluids, we considered the possibility of replacing conventional seed materials with alternative crystalline substances. It was found that continuous lactose crystallization methods are promising.

The purpose of this work is to investigate the basic principles of the heterogeneous lactose crystallization technology and to design apparatuses for continuous crystallization using spraying in vacuo and scrapedsurface heat exchangers.

The introduction of this crystallization technology in the dairy industry would shorten the processing time and significantly reduce the working area requirements.

EXPERIMENTAL

The objects of this study were lactose and demineralized whey solutions and sweetened condensed milk. The simulation seed materials were calcium carbonate, titanium dioxide, and silicon dioxide powders with a crystal size of 1–4 ?m. The amount of seeds added was 0.02–0.1% of the product weight. The seed material was introduced at $(33.5 \pm 1.5)^{\circ}$ C. The linear dimensions of the lactose crystals in the samples examined were determined using L.V. Chekulaeva's procedure. The crystal size uniformity coefficient was determined via N.A. Figurovskii's formula.



Fig. 3. Schematic of the improved unit for continuous cooling of sweetened condensed milk: (1) receiver tank, (2) product supply pump, (3) scraped-surface heat exchanger, (4) first cooling section, (5) second cooling section, (6) disc treater, (7) control board, (8) seed tank, (9) dosing pump, (10) resistance thermometer, (11) membrane manometer, (12) jet mixer, and (13) buffer tank.

The improved pilot plant based on a scraped-surface heat exchanger for continuous crystallization of lactose in canned dairy products is schematized in Fig. 3.

The plant consists of the following elements: receiver tank 1; positive displacement pump 2 for product supply, fitted with an IG5-Rus frequency .changer (LG Corp.); two-section scraped-surface heat exchanger 3 with a disc treater 6 equipped with an IG5-Rus frequency .changer (LG Corp.); jacketed seed tank 8 with an impeller; dosing pump 9; jet mixer 12; resistance thermometers 10; membrane manometers 11; control board 7; stirred buffer tank 13.

The unit was operated in the following way. After vacuum evaporation, the condensed lactose-containing product, whose temperature was 55-60°C, entered the receiver tank, from which it was pumped into the first section of the scraped-surface plate-type heat cooler, where it was cooled to the lactose mass crystallization temperature. Next, the product entered the disc treater that consisted of a system of rotating and fixed discs. Passing through the gaps between the discs, the product was subjected to intensive mechanical treatment. A dispersion of microcrystalline lactose from the seed preparation tank was injected with the dosing pump through the jet mixer into the product stream entering the disc treater. In the jet mixer, the injected liquid stream was preliminarily broken up by the product stream passing through the mixer. Next, the seedcontaining dispersion droplets were further reduced and uniformly incorporated in the product in the disc treater. This ensured the uniform distribution of the seed crystals over the entire volume of the product. Since the actuator of the disc treater was equipped with a frequency changer, it was possible to vary the intensity of mechanical treatment to alter the crystal size.

The product that left the disc treater was directed to a holder and then to the second section of the cooler, where it was cooled to room temperature (18–20°C). Prior to be canned, the product was stirred in the buffer tank.

The improved pilot plant based on vacuum spray crystallization is schematized in Fig. 4.



Fig. 4. Setup for investigating the continuous vacuum crystallization process: (1) vacuum chamber, (2) product tank, (3) screw pump, (4) liquid ring vacuum pump, (5) control board, (6) spray nozzle, (7) movable temperature sensor, (8) two lamps, (9) inspection window, (10) air dome with a pressure-vacuum gage, (11) standard vacuum gage, (12) resistance thermometer, (13) sampler, (14) vacuum relief valve, (15) electric heater, (16) flowmeter, (17) measuring vessel, and (18) crate with beakers.

This setup includes a 0.075-m^3 vacuum chamber 1 with a spray nozzle 6 mounted at its top. The lid of the vacuum chamber has two lamps 8, a vacuum gage 11, a well for the movable temperature sensor 7, a pipe with a vacuum relief valve 14, and an air suction pipe. In the upper part of the sidewall, there are two inspection windows for watching the spraying process and photographing. The bottom of the vacuum chamber has an emptying pipe, a temperature sensor 12, and a sampler 13. There is a crate with beakers 18 inside the vacuum chamber for determination of the irrigation density. The setup includes an OVN-4 screw pump 3 fitted with an IG5-Rus frequency changer (LG Corp.), an MEX-50 liquid ring vacuum pump 4 (Italy), a control board 5, and a 0.006-m³ product tank 2 with an electric heater 15 operable in automatic and manual modes, a resistance thermometer 12, and a float-type flowmeter 16. The capacity of the screw pump is measured using a measuring vessel 17. Temperature is measured with TSP-0879-01 resistance thermometers with a TRM-2 microprocessor-based temperature controller.

The unit was operated in the following way. The initial product from tank 2 was delivered by the screw pump 3 at a preset rate through the spray nozzle with a given geometry into the vacuum chamber pumped down to a preset depression. The sprayers were geometrically similar conical nozzles with an outlet orifice diameter of $d_{\rm c} = 0.82$? 10^{-3} -2.15 ? 10^{-3} m. Experiments were carried out on distilled water, a 50% lactose solution, and condensed whey with a dry matter content of 50±1% at 60-80°C and on sweetened condensed milk with a water content of 26.5% at 50-90°C. The following parameters were monitored during the experiment: product tank temperature, vacuum chamber temperature at fixed distances from the nozzle, throughput capacity (by a volumetric method), pressure before the nozzle, and depression in the chamber.

The product samples obtained in these experiments were packed in tin cans (400 g of product in each) and were stored for 120 days at $6-10^{\circ}$ C. The lactose crystal size was determined in the freshly prepared product, in the product that had been stored for 48 h, and then at 15-day intervals.

RESULTS AND DISCUSSION Simulation seed materials

It was established that the minimum necessary dosage and particle size of a seed material depend crucially on what the seed material is and when it is introduced. Preliminary crystallization efficiency data (obtained after 48-h-long storage) as a function of the quantity of seeds introduced are presented in Fig. 5.

It follows from the data presented in Fig. 5 that the crystallization efficiency depends significantly on the kind and amount of simulation seed material. The highest efficiency (96%) was achieved with 0.2% SiO₂, and the lowest efficiency was observed for 0.01% TiO₂. A decrease in the dosage of a simulation seed material exerts an adverse effect irrespective of what the material is. Preliminary studies demonstrated that, when the amount of seed material is 0.05% or below, up to 38% of the samples undergo no crystallization. Note that the efficiency of the process does not depend significantly

on whether 0.1 or 0.2% seed material is added. For this reason, subsequent studies were carried out for a seed material dose of 0.1%.



Fig. 5. Crystallization efficiency as a function of the kind and amount of seed material introduced.

The resulting samples were stored for 90 days at 6–10°C. The crystal size of lactose in the samples was determined at 15-day intervals. The results of these measurements are presented in Fig. 6.



Fig. 6. Crystallization efficiency during storage as a function of the kind and amount of seed material introduced.

Silicon dioxide proved to be the most effective seed material: with a SiO_2 dose of 0.1% relative to the product weight, the average crystal size of lactose did not exceed 6.4 ?m on the 90th day. A seed dose was considered to be effective when its increase did not exert any significant effect on lactose crystallization.

It was found that use of a simulation seed material does not change the classical shape of lactose crystals (Fig. 7).

On the whole, the seed efficiency decreases in the following order: $SiO_2 > TiO_2 > CaCO_3$.

These data suggest that use of alternative seed materials in lactose crystallization from saturated solutions needs to be further studied.



Fig. 7. Lactose crystals with simulation nuclei.

Continuous crystallization in a scraped-surface heat exchanger

The experiments carried out in this study can be divided into two groups. In the first group, the seed materials was a suspension of lactose powder in a vegetable oil (combined seed material); in the second group, the seed material was the finished product that had been subjected to crystallization.

Below, we will consider the most important results of these experiments. The first group of experiments included two variants.

First variant. The seed material as a suspension of lactose powder in a vegetable oil (0.12% of the product weight) from tank 8 was continuously introduced with the dosing pump 9 into the product stream through the special-purpose jet mixer 12 downstream of the first section of the cooling crystallizer (Fig. 3) at the lactose mass crystallization temperature. Next, the product was directed to the disc treater, where it was subjected to intensive hydrodynamic treatment (n = 1000 rpm). As a result, the seed material and fat were uniformly distributed throughout the product volume and the fat was finely dispersed. The product processed in this way entered the holder, which was a tube segment 50 mm in diameter connecting the disc treater with the second section. The residence time of the product in the holder was 4 s at a preset crystallizer throughput capacity of 300 kg/h.

In the second section, the product was cooled to 20° C, the temperature prescribed by the technical regulations.

The seed introduction temperature was varied between 30 and 35°C in 1°C steps. The product was sampled after the second cooling stage. The size distribution of lactose crystals in the samples was determined via a standard procedure.



Fig. 8. Dependence of the extent of crystallization of lactose on the seed introduction temperature.

Figure 8 shows how the extent of crystallization of lactose in the product depends on the seed introduction

temperature. It is clear from the plot that the number of the resulting crystals depends on the mass crystallization temperature and reaches its maximum value of 1420 thousands per cubic millimeter (average crystal size of 4.5-5.0 ?m) at 34° C.

Second variant. In order to evaluate the effect of hydrodynamic treatment intensity on the crystal size distribution of lactose, we carried out a series of experiments at different rotational speeds of the working elements of the disc treater. Sweetened condensed milk at 55°C was fed into the first section of continuous cooling crystallizer, where it cooled to 34°C. At this temperature, the combined seed material was introduced into the product. After the introduction of the seed material, the product was subjected to intensive mechanical treatment in the disc treater. The rotational speed of the working elements of the disc treater was varied from one series of experiments to another and was 200, 600, or 1000 rpm. The product leaving the disc treater was directed through the holder to the second section of the continuous cooling crystallizer, where it was cooled to 20°C.

For proving the efficiency of the method suggested here, we cooled the product in the conventional way. In this experiment, the product temperature decreased from 55 to 20°C in 40 min. The linear dimension of 100 lactose crystals was measured in the samples, and the average value was then determined (Table 3).

Table 3.	Crystal	size	data	for	lactose
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		Continuous crystallization					
Parameter		after the disc			after the second		
	Conventional	treater for			cooling section		
	conventional	different		for different			
	ci ystamzation	rotational		rotational			
		speeds, rpm			speeds, rpm		
		200	600	1000	200	600	1000
Average lactose crystal size, ?m	6.35	7.72	6.26	5.03	5.84	4.83	4.18
Size uniformity coefficient	0.65	0.63	0.71	0.76	0.83	0.87	0.91

From the cumulative size distribution curves (Fig. 9), we derived the size uniformity coefficient.

Based on the data presented in Table 3, we plotted the dependence of the average lactose crystal diameter on the rotational speed of the working elements of the disc treater (Fig. 10). It can be seen from the plot that the average lactose crystal diameter definitely depends on the rotational speed of the working elements: as the rotational speed is increased, the lactose crystal diameter at the apparatus outlet decreases on the average by 30%. This seems to be a significant factor for enhancing the quality and storability of the finished product.

The second series of experiments was aimed at organizing a continuous process for lactose crystallization in the sweetened condensed milk without use of lactose powder. The seed material in these experiments was the finished product that had been subjected to crystallization. This variant of the process is prescribed by the RF Regulations TPI GOST R 53436-001.



Fig. 9. Cumulative crystal size distribution function for lactose.



Fig. 10. Average lactose crystal diameter as a function of the rotational speed of the working elements of the disc treater: crystals (1) after the disc treater and (2) after the second section of the cooling crystallizer.

In conventional (batch) crystallization, it is allowable to replace lactose powder with an earlier obtained finished product having good organoleptic properties (1.5–2.0% of the weight of the product being processed).

The design of our pilot plant allows this replacement to be made. In this case, a small amount of combined seed material that is necessary only for starting the plant is prepared in the seed tank. After the entire seed material is consumed, part (1.5-2.0%) of the finished product is continuously supplied to the seed tank via a special-purpose pipeline. This part of the product is injected into the product stream with a dosing pump though a special-purpose mixer. Next, the product enters the disc treater, which ensures perfect mixing and exerts strong hydrodynamic action on the product, thus intensifying crystal nucleation and growth. After passing through the holder, the product cools to the final temperature prescribed by the technical regulations in the second section of the scraped-surface plate-type heat exchanger, which is employed as the lactose cooling crystallizer. Note that the seed material in the plant startup period can be the finished product with good velvety consistency from an earlier batch. This approach

excludes lactose powder from the technology, making this variant of the crystallization process very advantageous.

For experimental confirmation of the possibility of carrying out continuous lactose crystallization with part of the finished product used as the seed material and for optimizing the processing conditions, we performed the second series of experiments, in which part of the finished product as the seed material was returned via a special-purpose pipeline into the seed tank and was then injected with the dosing pump into the product stream cooled to the lactose mass crystallization temperature. The experiments in which part of the finished product was recycled in this way yielded good results: all samples examined had over 1 million lactose crystals per cubic millimeter of the finished product, and the average crystal size was 4-5 ?m. This ensured the uniform velvety consistency of the product. Note the very favorable crystal size distribution: the first group of crystals (below 10 ?m) included 98-99% of the total number of crystals, and the crystallization uniformity coefficient was 0.87-0.92. For the sake of comparison, we carried out experiments on the same product using conventional (batch) method of the lactose crystallization.

The continuous crystallization experiments were performed at different seed introduction temperatures $(30-35^{\circ}C)$. The product was sampled sing four samplers: (I) sampler placed downstream of the jet mixer before the inlet of the disc treater, (II) sampler placed before the holder, (III) sampler placed before the second section of the crystallizer, and (IV) finished-product sampler mounted at the outlet of the apparatus.

Figure 11 illustrates the kinetics of the continuous crystallization of lactose at different seed introduction temperatures. The total residence time of the product in the apparatus was 30 s, which consisted of the following periods: 8 s, the first section of the cooling crystallizer; 2 s, disc treater; 4 s, holder; 16 s, the second section of the crystallizer.

As the seed material is injected into the product stream and as the the product is subsequently processed in the disc treater (2 s), rapid nucleation of lactose crystals takes place to the extent of 0 to 600-700 thousands per cubic millimeter; after the holder, the number of crystals increases to 1000-1200 thousands per cubic millimeter; at the apparatus outlet, it is as large as 1300 thousands per cubic millimeter. The average crystal diameter increase simultaneously from 3.7 ?m at the beginning of the process to 5.4 ?m at the outlet of the apparatus.

Lactose crystallization in the continuous cooling crystallizer begins at point B, which corresponds to the point at which the seed material is introduced into the product stream. The segment AB of the curves indicates the induction period in lactose crystallization. In the curve segment BC, which corresponds to processing in the disc treater, intensive lactose crystallization takes place (dashed straight line slightly deviating from the vertical). The segment CD indicates the end of product processing in the disc treater and is characterized by a further rapid increase in the number of lactose crystals and by a distinct branching of the kinetic curve into the curves corresponding to different seed introduction temperatures. The segment DE corresponds to product passage through the holder and is characterized by a further increase in the number of lactose crystals. The segment EF corresponds to the cooling of the product from the seed introduction temperature to the final temperature specified in the technical regulations in the second section of the crystallizer. In this period, the rate of increase of the number of crystals decreases and the curves tend to their maximum level. This indicates the end of crystallization at the given supersaturation.



Fig. 11. Lactose crystallization kinetics at seed introduction temperatures of (1) 30, (2) 31, (3) 32, (4) 33, (5) 34, and (6) 35° C.

The data plotted in Fig. 11 also suggest that the optimal seed introduction temperature is 34°C, at which the most extensive crystallization is observed.



Fig. 12. Histogram of the size distribution of lactose crystals.

Figure 12 presents the histogram of the size distribution of lactose crystals. It is clear that, in continuous lactose crystallization in the sweetened condensed milk, the greater part of the crystals (98%) belongs to the first size group (≤ 10 ?m) and there are no lactose crystals belonging to the third group 16 to 30 ?m). The processing of a reference sample by the conventional method yielded the following crystal size

distribution: first group, 75%; second group, 22%; third group, 3%. This size distribution is obviously worse.

Continuous crystallization using product spraying in vacuo

Using a model of the product (aqueous solution of lactose with a dry matter content of 50%), we optimized the spray nozzle geometry and selected a nozzle for the vacuum crystallizer (Fig. 13).



Fig. 13. Schenatic of the spray nozzle of the vacuum crystallizer.

We elucidated the dependences of the spray angle on the product inlet pressure at a fixed depression in the vacuum chamber and on the product inlet temperature (Figs. 14, 15).



Fig. 14. Spray angle as a function of inlet pressure at 60°C.



Fig. 15. Spray angle as a function of inlet pressure at 80°C.

It was experimentally confirmed that it is possible to generate a large number of lactose crystals in the lactose-containing product by spraying the latter in a chamber at a residual pressure of 550-700 Pa and a temperature of 60-90°C, which is well above the boiling point at this pressure.

It was also proved experimentally that the final product temperature depends on the initial temperature at a constant product inlet temperature and a constant depression in the vacuum chamber (Fig. 16). It was elucidated how the final product temperature depends on the injection pressure (Fig. 17).



Fig.16. Final product temperature as a function of the initial product temperature.

CONCLUSIONS

This study of the continuous crystallization of lactose in sweetened condensed milk suggests the following conclusions:

(1) The heterogeneous crystallization of lactose is promising for production of sweetened condensed dairy products. (2) Experiments proved the theoretical prediction that continuous crystallization can be carried out in an apparatus based on scraped-surface plate-type heat exchanger.

(3) Two variants of seed introduction at the optimal mass crystallization temperature of lactose were considered. Both are efficient and can be industrially used in the operation of a continuous cooling crystallizer.



Fig. 17. Final product temperature as a function of the pressure before the nozzle.

(4) Use of the disc treater in combination with the continuous cooling crystallizer ensures uniform distribution of the seed material over the entire product volume. Moreover, the hydrodynamic action of the disc treater on the product intensifies the crystallization process and favors the formation of smaller crystals.

(5) Vacuum spray crystallization is a promising method for processing lactose-containing feedstocks.

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