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INFLUENCE OF CRYSTALLIZING AGENT ON SUGAR QUALITY

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ABSTRACT

Background. One of the important processes in sugar production is massecuite boiling. The intensification of this process is possible by reducing the boiling time and thus improving the quality of the finished product. This study examines the methods of massecuite boiling by using different types of crystal formers: powdered sugar, a standard factory suspension, and the crystal former "Ester K 01", as well as a comparison of their direct effects on the quality of the finished product.

Materials and methods. All laboratory tests of crystal-forming agents were carried out using syrup taken from sugar factories. Laser diffractometry was used to determine the particle size (dispersion) of the crystal-forming agents. Prepared syrup samples were used to obtain the massecuite, which was boiled in a laboratory vacuum evaporator in conditions maximally adapted to production. Sugar was obtained by separating the massecuite in a laboratory vacuum filtration. All the studies of qualitative and quantitative indicators of products and intermediates were performed using standard methods.

Results. The particle size distribution was determined and the curves of the differential size distribution of sugar fineness were given. The most uniform particle distribution was noted using "Ester K 01" crystal former. The duration of massecuite boiling was studied and a comparative characteristic of the quality indicators was provided for the obtained massecuite and massecuite syrup in accordance with the type of crystal former used. The highest sucrose content was observed in the sugar sample obtained using the "Ester K 01" crystallizing agent. This sample also had the lowest ash content. Microphotographs of the sugar crystals were provided with fixation of their size after 5, 10, and 20 minutes of adding various types of crystallizing agent, and the morphology of the sugar crystals in the finished massecuite was studied. The most equal crystals were produced using an "Ester K 01" crystallizing agent and a standard factory suspension in comparison to massecuite made with icing sugar. The granulometric composition of sugar crystals obtained with different crystal formers was also compared in this research. The crystal former "Ester K 01" was studied in production and laboratory conditions, and the crystallizing agents' comparative characteristics were also given.

Conclusion. In conclusion, the usage of the crystallizing agent "Ester K 01" in the fully automated process conditions for producing massecuite would be recommended by us. It will allow more uniform granulometric composition of sugar to be obtained.

Keywords: crystallization center, crystallizing agent, crystallization, crystal size distribution, massecuite, sugar

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INTRODUCTION

One of the key processing steps in sugar manufacturing is massecuite production. Usually, it consists of a set of technological processes, namely, evaporation (concentration) of the syrup solution to increase the coefficient of saturation $K_p = 1.3$, the formation of sucrose crystallization centers, and crystal growth (Martins et al., 2005; Van der Poel et al., 1998).

During growth, the crystals were washed with a layer of saturated intercrystal solution (boundary layer). Excess sucrose molecules from this layer quickly passed (released) onto the crystals surface and the solution became saturated, with the concentration of sucrose *Cn*. However, at a certain distance from the crystallization centers, the solution remained saturated with a concentration of *Cp*. Due to the difference of concentrations *Cp-Cn*, sucrose diffuses through the solution boundary layer and approaches the crystals and sucrose molecules which are embedded in the crystal lattice (phase transition). Therefore, the crystal growth rate is determined by the sucrose diffusion rate and the phase transition rate at the phase separation boundary. After that, both existing and spontaneous formation of new crystallization centers takes place in the solution. With a decrease in the supersaturation coefficient ($K_p \leq 1.2$), the crystallization rate decreases, new crystallization centers cease to form, but the pre-existing crystals build up. The productivity of the vacuum unit decreases slightly and, as a result, the duration of the process of boiling massecuite increases (Rózsa et al., 2016; Rózsa et al., 2017a; Rózsa et al., 2017b).

However, there is the challenge of intensifying the process of massecuite boiling, in particular, by reducing the boiling time duration, reducing the cost of thermal energy, and reducing the loss of sucrose from thermal decomposition, and thus improving the quality of the finished product (Hernández et al., 2014; Mazaeda et al., 2014; Skoryk et al., 2016).

The best way to solve the problems listed below is by using a modern crystallizing agent which is needed for the process (Rózsa, 2008; Zinab et al., 2015).

For a long time, powdered sugar was used to establish crystallization centers in boiling massecuite in sugar manufacturing. However, the usage of powdered sugar has several disadvantages. Among the main ones are the following: uneven linear dimensions of crystals, adhesion of sugar crystals, the formation of a significant number of drusen, deterioration in sugar quality due to increased color and ash content, the complexity of the centrifugation process, and the deterioration of the granulometric composition of sugar (Skoryk, 2004).

At the moment, most sugar plants are using new methods of establishing crystallization centers, in particular the introduction of suspensions of ductile or hard ductile crystallizers (Zinabet al., 2015).

The aim of this study was to compare the effectiveness of the action of crystal-forming agents obtained in various ways during the process of sugar crystallization. For this research, we used a suspension, which was obtained in a sugar factory by grinding sugar in a mill and its further stabilization in isopropyl alcohol, and a crystallizing agent for the sugar industry “Ester K 01” with producer’s food safety certificate FSSC 22000 (Anisimova et al., 2017). This is a suspension of homogeneous sugar crystals with a size of about 10–12 microns obtained by precipitation from a supersaturated solution and stabilized by a specially selected food-grade surface active agent. According to the producer’s instruction, this allows it to maintain the integrity of the crystals’ shape and ensures the uniformity of their sizes. To obtain more objective results, powdered sugar obtained in a factory by grinding sugar crystals was used as a control sample in our research.

MATERIALS AND METHODS

All laboratory tests of crystal-forming agents were carried out using syrup sourced from Ukrainian sugar factories.

The quality parameters of the syrup samples were held to the general requirements of the industry (Table 1).

Laser diffractometry was used to determine the particle size (dispersion) of the crystal-forming agents. The measurements were carried out on a SALD-201V diffraction laser analyzer (SHIMADZU, Japan). A semiconductor laser with a wavelength of 670 nm was used as a light source. The processing of correlation functions was carried out using software Wing-SALD II-201V V3.3 (SHIMADZU, Japan).

The samples were prepared for measurement as follows: before measurement, each sample of the crystallizing agent was shaken manually, then the samples

Table 1. Quality parameters of sugar syrup

Parameter	Meaning
Dry matter, %	72
Sucrose content, %	66.8
Purity, %	92.8
Colour, ICUMSA unit	768
Calcium salts, %	0.036
Ash content, %	0.142
The concentration of hydrogen ions (pH_{20})	6.8

were mixed with a magnetic stirrer for 10 min and then placed in an ultrasonic stirrer for 10 min. In a well-mixed sample, measurements were taken. Isopropyl alcohol was used in an amount of 100 ml as a solvent.

Microscopy of massecuite samples was carried out using the microscope GENETIC PRO MONO (A; Delta Optical, Poland) in accordance with the working instructions (Faria et al., 2003; Mazzobre et al., 2003). Micrographs were recorded with a digital camera MC-MOS 5100 5.1 MP USB 2.0 (SIGETA, Ukraine). Microphotography handling was done using the software ToupView V3.7.

Prepared syrup samples were used to obtain massecuite, which was boiled in a laboratory vacuum evaporator, created on the basis of the typical evaporator RE-2000A (Shanghai Yarond Biochemistry Instrument Factory, China) in conditions maximally adapted to production (Fig. 1). The evaporator consisted of the following elements: vacuum boiling flask (1), faucet for pumping syrup (2), refrigerator (3), condensate receiving flask (4), vacuum gauge (5), gearbox for regulating the depth of vacuum (6), vacuum pump (7) and heating bath (8).

The massecuite samples being tested were boiled under a vacuum. First, the syrup was evaporated for 20–30 minutes under a pressure of $P = –60$ kPa. The pressure was then decreased to –90 kPa. The temperature of the boiling process was maintained within 69–72°C.

During the first 20 minutes of boiling, the syrup began to contain dry matter = 80–82%, which under the above conditions corresponds to a coefficient of oversaturation $C_o \geq 1.2–1.3$.

Fig. 1. Vacuum evaporator for massecuite boiling

On reaching the oversaturation coefficient, the crystallizing agents were added in the following proportions: icing sugar (at the rate of 100 g per 40 tons of syrup), factory suspension (at the rate of 15 g per 40 tons of syrup), and “Ester K 01” crystal former (at the rate of 4 ml per 40 tons of syrup).

Next, the massecuite was boiled and the sugar crystals were grown during two-time syrup pumping, which was necessary to reduce the coefficient of saturation and to avoid the formation of a large number of new crystallization centers, as well as the growth of existing crystals.

The sugar was obtained by separating the massecuite in a laboratory vacuum filtration unit consisting of a Bunsen flask, a Buchner nozzle with a filtration baffle, and a membrane vacuum pump N035AN.18 (KNF, Germany). Next, the obtained sugar samples were washed with 70% ethyl alcohol solution. Following this, the sugar was dried on a sieve in a laboratory dryer CH СНОЛ 58/350 (TermoLab, Ukraine) in a temperature 45–50°C.

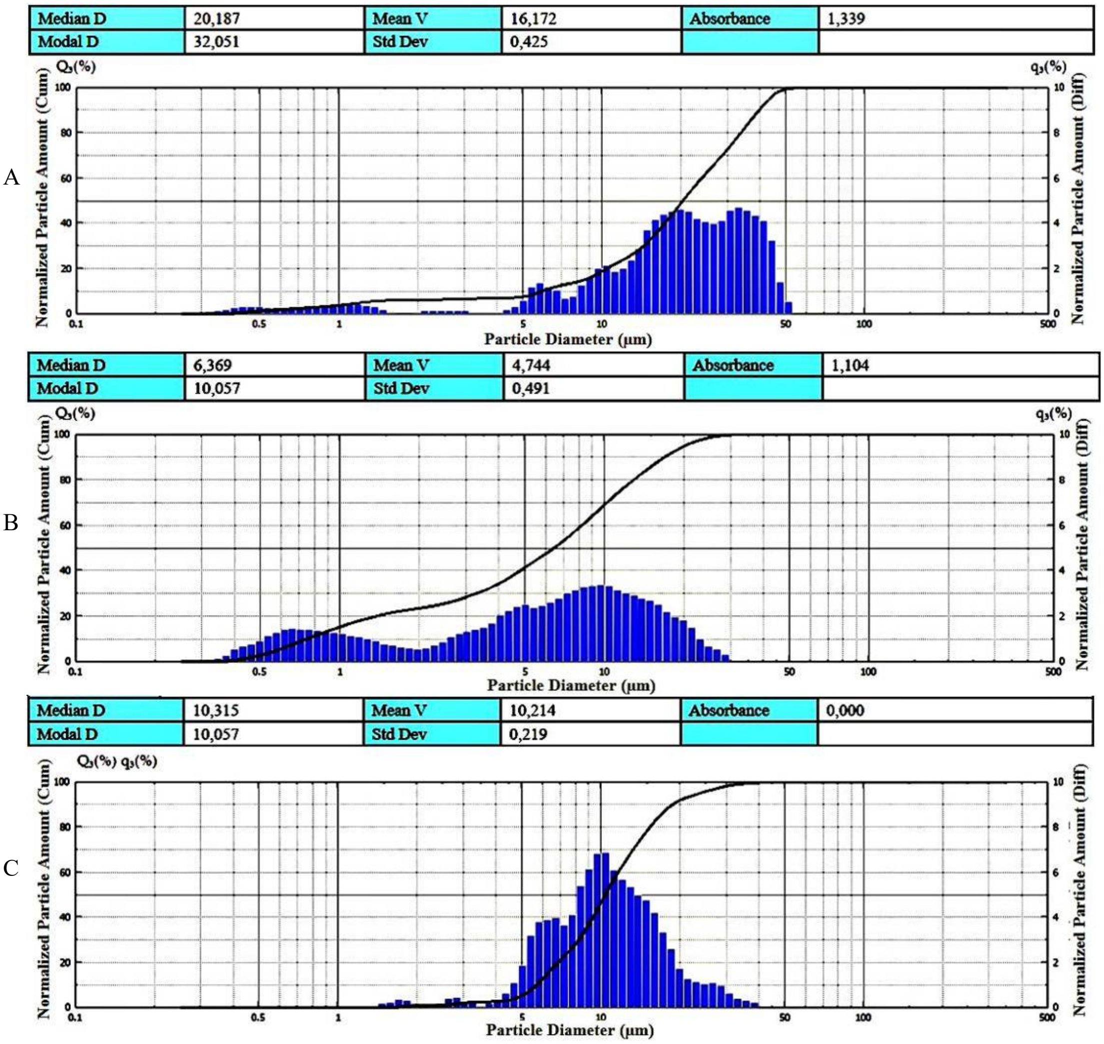
All studies of qualitative and quantitative indicators of products and intermediates were performed using standard methods (ICUMSA..., 2007). In the course of the laboratory research, the following instruments were used: for determining the solids content – refractometer Abbemat (Anton Paar, Austria), sucrose content – saccharimeter MCP 5300/5500 Sucromat

(Anton Paar, Austria), colour – colorimeter КФК-3-01 (ЗОМЗ, Russia), method for determining the activity of hydrogen ions – Laboratory ionomer И-160М (АНТЕХ, Belarus), ash content – conductivity apparatus OK 102/1 (Radelkis, Hungary), the weighing of analysis samples was carried out using an analytical balance AS 220.R2 (RADWAG, Poland).

RESULTS AND DISCUSSION

In order to evaluate the initial qualitative characteristics of various crystallizing agents, they were analyzed dispersedly and it was found that the size of the crystals affects the rate of formation and uniformity of sugar crystals (Schlumbach et al., 2018; Srisanga et al., 2015; Zinab et al., 2015). Results obtained are displayed in Figure 2.

An analysis of the composition particles of powdered sugar when dispersed (Fig. 2A) showed that the average particle size was 16.2 μm, whilst the distribution was uneven with a peak mode of 32.1 μm. Furthermore, particles with a size of less than 1.0 microns and a maximum size of 53.2 microns were found in the powdered sugar. From an analysis of the particle



Median D	20,187	Mean V	16,172	Absorbance	1,339
Modal D	32,051	Std Dev	0,425		

Median D	6,369	Mean V	4,744	Absorbance	1,104
Modal D	10,057	Std Dev	0,491		

Median D	10,315	Mean V	10,214	Absorbance	0,000
Modal D	10,057	Std Dev	0,219		

Fig. 2. Differential particle size distribution of crystallizing agent: A – icing sugar, B – a standard factory suspension, C – the crystal former "Esther K 01"

distribution of a standard factory suspension (Fig. 2B), we can say that dividing is bimodal with modes of 0.7 μm and 10.1 μm, that the average particle size is 4.7 μm, and that the distribution is uneven. Particles with the smallest size of 0.3 microns and a maximum size of 29.8 microns were also found. The dispersed particle distribution of the "Ester K 01" crystal former (Fig. 2C) showed that the average particle size was 10.2 microns. The distribution was uniform with a peak mode of 10.1 μm. Particles with the smallest diameter of 1.5 μm in an amount of 0.144% by volume and a largest of 39.8 μm in amount of 0.19% by volume were present in the crystallizing agent.

The results of the massecuite boiling durations using different crystal formers are provided in Table 2.

An analysis of the data provided shows that the longest duration of massecuite boiling occurred with the icing sugar sample. The shortest duration occurred with a standard factory suspension. The duration of boiling massecuite on the crystal former "Esther K 01" was slightly higher than on the factory suspension, yet quite close to it.

Table 2. Duration of massecuite boiling

Crystallizing agent	Duration of massecuite boiling min
Icing sugar	90–95
Factory suspension	70–75
"Ester K 01" crystal former	75–80

To assess the effectiveness of various crystal formers on the quality of intermediates the following parameters were analyzed: massecuite, intercrystal solution, and sugar. The results obtained are presented in Table 3.

As can be seen in the table above, the highest sucrose content was observed in the sugar sample obtained using the "Ester K 01" crystal former. In this case, it also had the lowest ash content in that sample. Its color is at the same level as a sugar sample obtained with a standard factory suspension, but lower by 11 units in comparison with powdered sugar. Morphological research of sugar crystals in massecuite obtained on various crystal-forming agents, as well as their sizes, are shown on Figure 3.

In the first five minutes after the addition of the crystal former (Fig. 3), we observe the formation of uniform sugar crystals with the "Ester K 01" crystal former, the size of which is mainly 0.11–0.15 mm. On factory slurry and icing sugar, sugar crystals are uneven in size, some individual crystals reach 0.3 mm in the presence of the bulk of the crystals (which ranged from 0.07–0.17 mm). Ten minutes after adding up the crystals (Fig. 3), one can observe almost the same size distribution of sugar crystals obtained on standard factory paste and the "Ester K 01" crystal former

Table 3. Complete analysis of intermediates when massecuite is boiled up on different crystal-forming agents

Intermediate	Crystal-forming agents	pH_{20}	Dry matter %	Sucrose content %	Purity %	Color, ICUMSA units	Ash content %
Massecuite	icing sugar	6.1	89.6	81.4	90.85	806	0.155
	factory suspension	6.1	91.2	82.25	90.19	828	0.155
	"Ester K 01"	6.1	91.6	82.15	89.68	818	0.156
Massecuite syrup	icing sugar	6.0	58.0	52.4	90.34	801	0.086
	factory suspension	6.0	58.2	51.85	89.09	822	0.090
	"Ester K 01"	6.1	58.6	53.2	90.78	808	0.092
Sugar obtained	icing sugar	–	–	97.8	–	204	0.069
	factory suspension	–	–	97.75	–	192	0.065
	"Ester K 01"	–	–	98.15	–	193	0.064

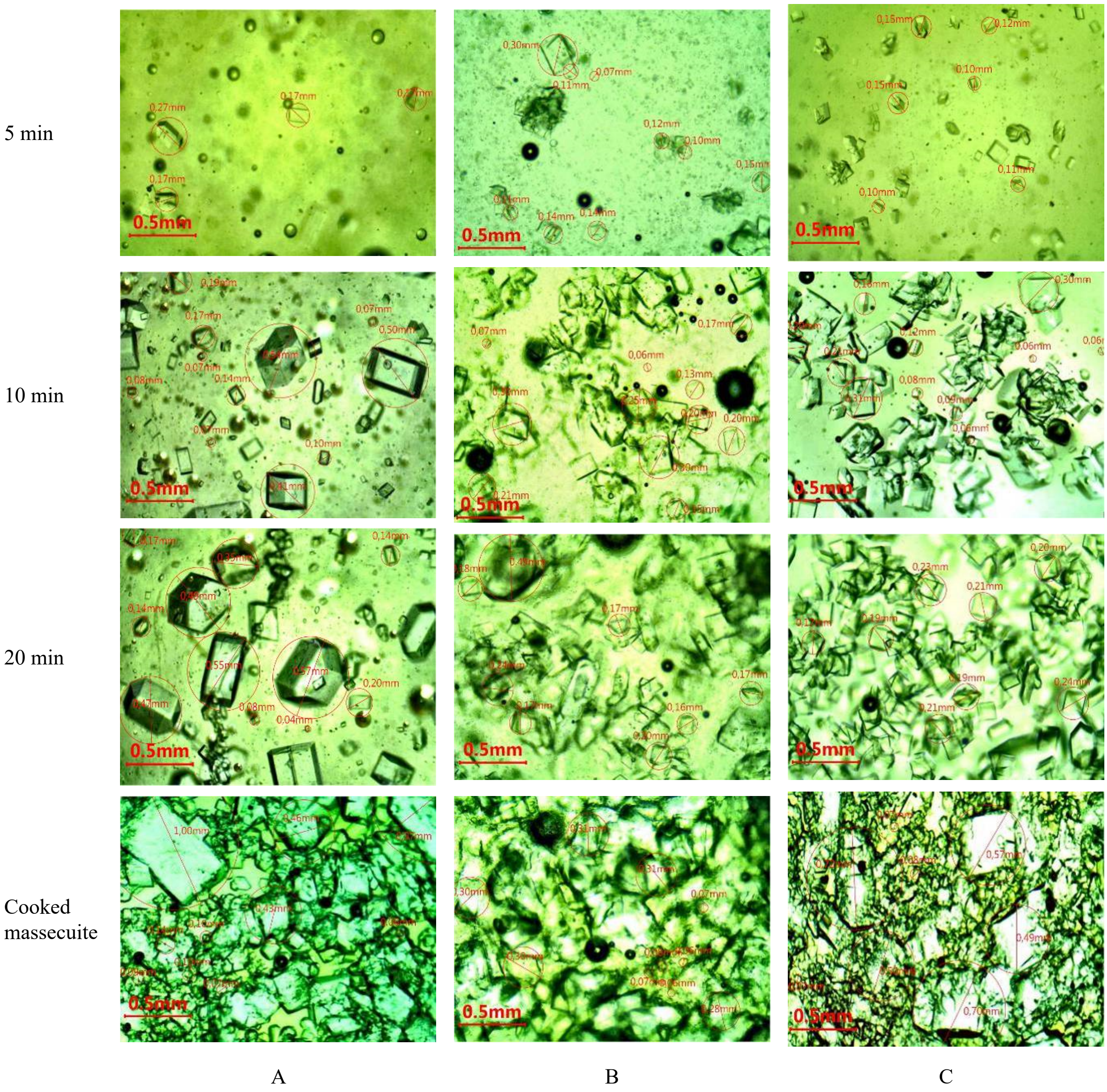


Fig. 3. Microphotographs of sugar crystals in the massecuite after 5, 10, and 20 minutes with the establishment of crystal-forming agents and microphotograph of cooked massecuite (crystals in a sector of 0.25×0.18 cm in size with a 4-fold increase): A – icing sugar, B – a standard factory suspension, C – the crystal former "Ester K 01"

at the maximum size values of 0.3 mm. On powdered sugar, we see crystal formations with a maximum size of 0.41–0.5 mm together with crystals with sizes of 0.07–0.2 mm.

Twenty minutes after adding up the crystal-forming agents (Fig. 3), the bulk of uniform sugar crystals with a size of 0.17–0.25 mm were those that were obtained using the "Ester K 01" crystal-forming agent. Sugar crystals obtained on a standard factory suspension also had uniform sizes, although single crystals with sizes up to 0.5 mm were present in the solution. The use of powdered sugar led to the formation of different sized

Table 4. Granulometric composition of the obtained sugar samples crystals

Screen, diameter	The average size of crystal, d", mm	Sugar mass on screen, g		
		icing sugar	factory suspension	Ester K 01
2.50	2.70	0	0	0
1.25	1.35	1.86	1.03	3.27
1.00	1.10	13.87	8.51	13.57
0.80	0.90	32.75	18.99	29.28
0.50	0.65	22.93	39.80	48.04
0.20	0.35	21.89	31.67	5.84
Bottom	0.10	1.58	0.23	0.56
The average linear size MA, mm	–	0.7482	0.648	0.7896
Heterogeneity factor CV, %	–	36.18	38.63	27.62

crystals with a size range between 0.14 and 0.57 mm. An analysis of microphotographs of the finished massecuite (Fig. 3) showed that the most uniform crystals were formed using the "Ester K 01" crystal former and a standard factory suspension in comparison with the massecuite obtained with powdered sugar.

Thus, the morphological, dimensional, and quantitative parameters of sugar crystals obtained were analyzed at various stages, and its results allow us to conclude that the best quality crystals were obtained as a result of the crystal formation of "Ester K 01". In second place was a standard factory paste. Sugar crystals obtained using icing sugar were uneven.

To assess the quality of the crystals of the obtained sugar samples, an analysis of their granulometric composition was carried out. The results are displayed in Table 4.

From Table 4, we can see that the most equal crystals were obtained using "Ester K 01".

In order to verify the effectiveness of the "Ester K 01" crystal former, production tests were conducted at one of the sugar factories in the central region of Ukraine.

Comparative results of the granulometric composition of sugar obtained in laboratory conditions and production conditions using a crystal former "Ester K 01" are presented in Table 5.

From the analysis of the data presented in Table 5, it is clear that the usage of the crystal former in manufacturing conditions ensures the production of equal sugar crystals. The number of crystals with an average size of 0.65 mm is 49.16%, which is 1.12% higher in comparison to sugar obtained in laboratory conditions. The number of crystals with a fraction size of 0.10 mm is only 0.08%, which is 0.48% less compared to conditions in the laboratory.

The main aim of sugar syrup boiling using the crystallizing agent is to ensure the uniform composition of sugar crystal size. This is difficult to achieve in manufacturing conditions. By adding crystals with a size less than 10 mkm, they pass the critical growing stage with unpredictable results However, this can be resolved in a solution or can become a new center of crystallization.

In cases where crystals with a size greater than 10 mkm are used, the possibility of resolving is totally excluded since these types of crystals have quite a big surface. This is a reason for the intensification process of the crystals growing (Atsukawa et al., 2020).

At this stage, the appearance of new crystallization centers and clumping of crystals with an irregular shape can only be avoided by using the dedicated seed material and a precise dosage of syrup in the boiling process. As a result, the crystallizing agent

Table 5. Granulometric composition of the obtained sugar samples crystals both from manufactory and laboratory conditions using a crystal former "Ester K 01"

Aperture of screen, mm	The average particle size, d", mm	Sugar mass on screen, g	
		laboratory conditions	manufacturing
2.50	2.70	0	0
1.25	1.35	3.27	5.14
1.00	1.10	13.57	10.41
0.80	0.90	29.28	26.28
0.50	0.65	48.04	49.16
0.20	0.35	5.84	8.93
Bottom	0.10	0.56	0.08
The average linear size MA, mm	–	0.7896	0.7718
Heterogeneity factor CV, %	–	27.62	30.64

"Ester K 01" was found to be particularly suitable for the manufacturing process. The aggregate state of the suspension ensures its long-term storage without any quality changes.

This is achieved since the processes of recrystallization and conglomeration (the formation of drusen) are blocked.

CONCLUSIONS

From the results of this research, it can be concluded that better physicochemical and quality indicators of sugar result from using "Ester K 01" crystal former. In this case, the use is reduced for 15–20 minutes, while the volumetric amount of crystals in the massecuite increases and sugar with high quality equal crystals is obtained.

The crystal former has an equal size of sucrose crystals: the content of crystals of 10–12 microns in size is more than 70%. The consumption of the crystal former is 0.4–1.0 g/t massecuite and depends on the purity of the massecuite and the given size of crystals in the finished product. In addition, it is possible to produce an "Ester K 01" crystal former with a controlled microcrystal size for usage in various technological processes.

The introduction of modern crystal-forming suspensions makes it possible to intensify the centrifugation process and achieve a more uniform granulometric composition of sugar produced.

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