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DIMINUTION OF SUGAR LOSSES IN MOLASSES AND THE INCREASE OF CRYSTALLIZATION RATE OWING TO THE DECOLORIZATION OF THIN JUICE

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Key words: sugar technology, crystallization rate, sugar losses.

The measured values of crystallization rate of sucrose from various factory syrups were compared and on these grounds the coefficients of slowing down crystallization rate were calculated depending on contents of non-sugars and colouring matter in the crystallized syrup.

Basing on the obtained mathematical equations and the chemical analysis data it is possible to calculate the crystallization rate of sucrose from the factory liquor.

Losses of sugar in molasses under normal conditions of processing sugar beets amount to 10-12% in terms of sugar introduced with the beets. Many studies were carried to examine the effect of different agents on the amount of sugar remaining in molasses [1-5]. As it is known the removal of colouring matter from juices reduces the amount of molasses [6] and at the same time accelerates the crystallization of sugar [7].

Publications on the influence of the conditions of crystallization of sucrose on growth of crystals usually present results of measurements of the growth rate of single sucrose crystals or at least of their small quantity [8-15]. The majority of tests were carried out on model solutions. The range of temperatures at which the crystallization rate of sucrose was measured was usually small [12, 14, 15], higher temperature was applied only in some tests [11, 16-19].

The research carried out at Łódź Technical University in the years 1966-1976 comprised measurements of the sucrose crystallization rate in a wide of temperatures in solutions of various purity. The influence of non-sugars and colouring on the crystallization rate of sucrose in conditions similar liquor circulation was taken into account [22, 23].

Basing on the results obtained it was found that coefficient should be introduced to the formula defining the crystallization rate in pure solu-

tions. This coefficient slows down the sucrose crystallization rate due to amount of non-sugars contained in technical solutions.

$$\frac{dS}{dt} = A \frac{c - c_0}{\frac{1}{K_R} + \frac{\delta}{K_D}} \eta \qquad (1)$$

where S — mass of sucrose crystals,
t — time of crystallization,
A — summary surface of all crystals,
c — concentration of the supersaturated solution,
c_0 — concentration of the saturated solution,
$1/K_R$ — resistance of building in the molecules of sucrose into the crystallographic lattice,
δ/K_D — resistance of the transport of the hydrated sucrose molecules through a δ thickness layer,
η — coefficient defining the slowing down sucrose crystallization rate in impure solutions.

In pure solution the coefficient $\eta = 1$; in impure solutions $\eta < 1$. Basing on experiments the influence of non-sugars on the crystallization rate η_1 and that of colouring matter and other surface- active compounds η_2 separately ($\eta = \eta_1 \cdot \eta_2$) [20] were assessed separately.

EXPERIMENTAL

The purpose of this paper was to point to the advantages that can be achieved by a sugar factory in which decolorization of thin juice takes place.

DECOLORIZING OF THIN JUICE AND PREPARING SOLUTIONS FOR CRYSTALLIZATION RATE MEASUREMENT

Thin juice, thick juice and mother liquors taken from a sugar factory were used for the tests. The mother liquors were taken during sugar crystallization of 1st, 2nd and 3rd massecuite direct from vacuum pans.

The thin juice was decolorized by the multicolumn method on a pilot scale by means of granular activated carbon[6]. Table 1 lists the results of thin juice analysis and of thin juice caused the removal of 0.7 g of non--sugars in terms of 100 g dry substance in the juice. The granular activated carbon detained not only colouring matter and other surface-active compounds but also some amount of lime salts. It should be added that during the decolorization of the juice and the electrothermal regeneration of granular activated carbon no wastewater was formed.

The decolorized juice was concentrated to 65% of dry substance and then sugar crystallization was carried out in three boilings on a pilot scale.

Table 1. Factory thin juice and decolorized thin juice analysis

		Factory thin juice	Decolorized thin juice	Reduction of non-sugars and colouring substances
pH	—	8.8	8.6	—
Dry substance refr.	%	14.40	14.25	—
Sucrose	°S	13.45	13.40	—
Purity	—	93.4	94.1	0.7
Non-sugars	—	6.6	5.9	0.7
Specific absorbancy				%
at 420 nm	—	1.110	0.215	80.6
500 nm	—	0.319	0.063	80.3
560 nm	—	0.163	0.30	81.6
650 nm	—	0.058	0.012	99.3
740 nm	—	0.015	0.003	80.0
Lime salts	mg CaO/100 g DS	46	34	—
Reducing sugars	g/100 g DS	0.062	0.079	—

Table 2. Analysis of liquors prepared for crystallization

Liquors	Purity	Non-sugars %	pH	Specific absorbancy, E			650 nm	740 nm
				420 nm	500 nm	560 nm		
Refined sugar	99.98	0.02	7.3	0.032	0.019	0.012	0.006	0.003
	Factory liquors							
Thick juice 65 %	93.4	6.6	8.3	2.280	0.776	0.354	0.140	0.049
Mother liquor of 1st massecuite	89.8	10.2	8.3	6.310	2.285	1.190	0.428	0.175
Mother liquor of 2nd massecuite	82.2	17.8	8.3	14.145	5.658	3.010	1.251	0.490
Mother liquor of 3rd massecuite	70.9	29.1	8.3	28.012	11.082	5.910	2.462	1.016
	Liquors from the decolorized thin juice							
Thick juice 65 %	94.1	5.9	8.3	0.441	0.146	0.065	0.024	0.008
Mother liquor of 1st massecuite	90.9	9.1	8.3	1.221	0.431	0.218	0.073	0.028
Mother liquor of 2nd massecuite	84.1	15.9	8.3	3.278	1.115	0.552	0.221	0.080
Mother liquor of 3rd massecuite	74.0	26.0	8.3	6.250	2.175	1.080	0.436	0.163

The mother liquors were taken during sugar crystallization of 1st, 2nd and 3rd massecuite. In this way mother liquors of low colour were obtained. The crystallized white sugar was of lower colour than the one obtained from technical syrup.

The following solutions were prepared for basic research: A. syrup of refined sugar. B. Normal factory solutions — thick juice 65% dry substance and the mother liquors of 1st, 2nd and 3rd massecuite. C. Liquors from decolorized thin juice: thick juice 65% dry substance and mother liquors of 1st, 2nd and 3rd massecuite. Table 2 presents the results of analysis of all the syrups prepared.

DETERMINING OF THE CRYSTALLIZATION RATE OF SUCROSE FROM SOLUTIONS

The Dewar vessel (Fig. 1) was applied for the measurement of the crystallization rate; the vessel was rotated at the speed of 6 rpm in an appropriate thermostat [20, 21]. Inside the Dewar vessel, filled with the syrup to be crystallized, there were six wire cages, each containing 4 g of refined sugar crystals of possibly similar granulation. The linear velocity of movement of the crystals in relation to mother liquor was 113 cm·$\cdot$min^{-1}.

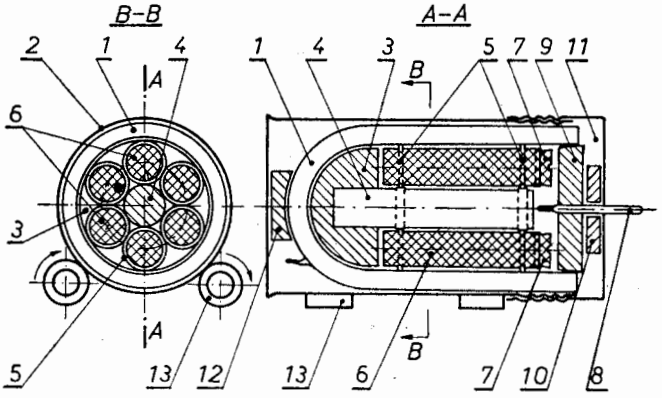

Fig. 1. Crystallizer; 1 — Dewar vessel, 2 — housing, 3 — rubber packing, 4 — steel bolt, 5 — cage holder, 6 — wire cages, 7, 9 — rubber stoppers, 8 — thermometer, 10 — rubber compression ring, 11 — lid, 12 — rubber cushion, 13 — drive rolls.

Each cage comprised an average of about 800 crystals. The average mass of one sucrose crystal, therefore, was s = 5 mg. The surface of one crystal was calculated from the equation $a = 4.12\sqrt[3]{s^2}$, where 4.12 is the coefficient of proportionality determined by Kucharenko for big crystals. The mean surface of one crystal was a = 0.1205 cm^2, the total surface of all the crystals, therefore, in one cage was

$$A = 0.00964 \text{ m}^2 \quad (800 \cdot 0.1205 = 96.4 \text{cm}^2).$$

The crystallization process was carried out at 70°C and supersaturation 1.10. After 30 minutes of crystallizing the cages with crystals were taken out of the Dewar vessel and were centrifuged at 3000 rpm during 5 minutes, then they were rinsed in ethanol, dried at room temperature and weighed.

On the basis of the growth of the crystals mass and taking into account the total surface of the crystals, the number of which did not change, the crystallization rate was calculated as the increment of mass dS a given surface A, in time dt [20].

$$dS/Adt[mg \cdot m^{-2} \cdot min^{-1}]$$

Table 3 presents the crystallization rate of sucrose from technical syrups as determined by measurements. Comparing the crystallization rate of sucrose in pure solution and in other solutions the value of the slowing down crystallization rate coefficient η was found.

Fig. 2 presents the determined values of coefficient η depending on the purity of technical syrups from which the sucrose was crystallized. Points

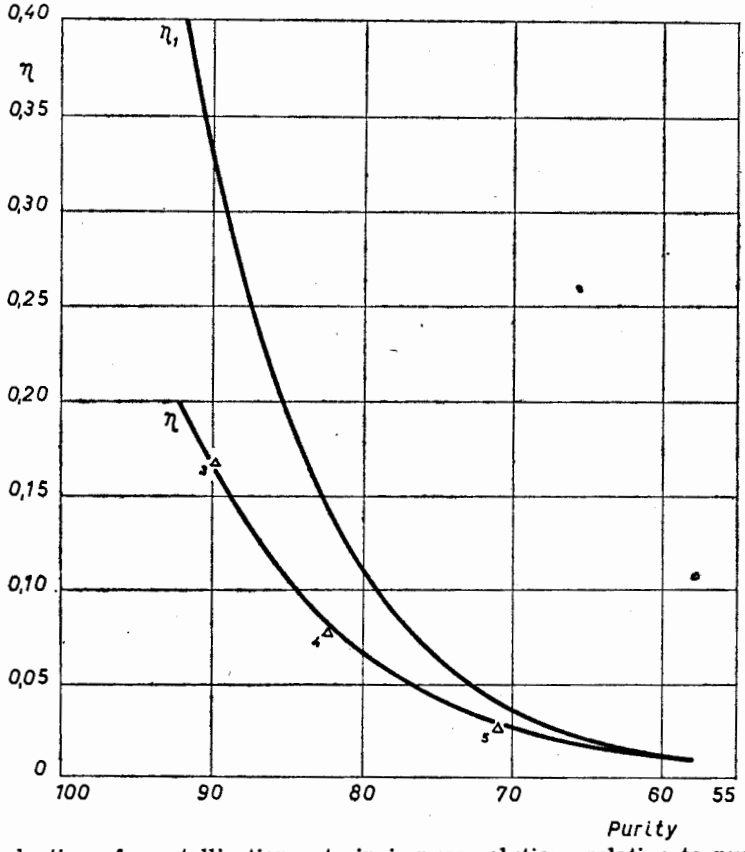

Fig. 2. Reduction of crystallization rate in impure solutions relative to pure sucrose solutions as a function of purity ($\eta = \eta_1 \cdot \eta_2$). η_1 — coefficient of slowing down sucrose crystallization rate in impure solutions

Table 3. The measured crystallization rate of sucrose dS/Adt from pure solution at 7.3 pH and from factory liquors at 8.3 pH

Liquors	Purity	Non-sugars	Specific absorbancy at 560 nm	Crystallization rate of sucrose $mg \cdot m^{-2-} \cdot min^{-1}$	Coefficient of slowing down sucrose crystallization rate η
Refined sugar	99.98	0.02	0.012	7080	1.000
Mother liquor of 1st massecuite	89.8	10.2	1.190	1203	0.1700
Mother liquor of 2nd massecuite	82.2	17.8	3.010	561	0.0793
Mother liquor of 3rd massecuite	70.9	29.1	5.910	206	0.0295

3, 4 and 5 were marked on the basis of results of measurements compiled in Table 3. The curve η_1 represents the influence of non-sugars on slowing down the crystallization rate in the fully decolorized solutions [20]. Both curves intersect at a solution purity below 60. As it is known, in such conditions the crystallization cannot take place because of a substantial increase of the solubility of sucrose in the presence of a large amount of non-sugars.

Fig. 3 represents the dependence of coefficient η on the syrup purity in the semi-log graph. In consequence the points 3, 4 and 5, representing the coefficient of slowing down the crystallization rate of sucrose from technical syrups, are situated along the straight line. The larger the amount of colouring matter and surface-active compounds in a given technical syrup the bigger the distance of the lower straight line η from that of η_1 at the same purity of the solution. It should be added that depending on

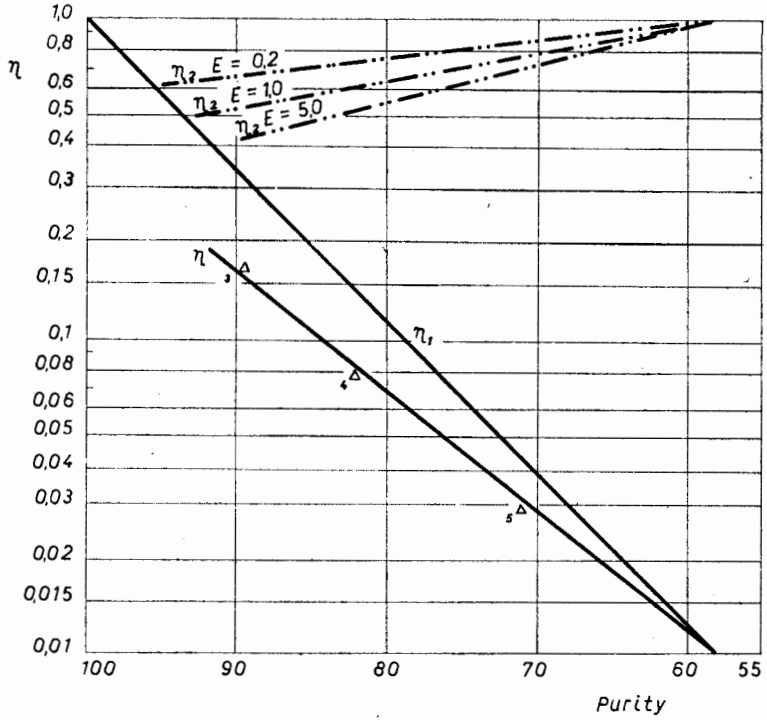

Fig. 3. Reduction of crystallization rate in impure solutions relative to pure sucrose solutions as a function of non-sugars quantity and specific absorbancy

the composition of non-sugars in sugar syrup the interections of the straight line η with that of η_1 may occur in a point corresponding to a purity of syrup from 56 to 60 and coefficient $\eta = 0.01$. That means that in

syrups of such a low purity crystallization rate of sucrose is a 100 times smaller than in pure syrups.

During crystallization from pure syrup the coefficient of slowing down crystallization rate equals $\eta = 1$, but in the case of impure syrups $\eta < 1$.

$$\eta = \eta_1 \cdot \eta_2 \tag{2}$$

The coefficient η_1, as mentioned before, represents the slowing down influence of non-sugars on the crystallization rate and the coefficient η_2 the slowing down influence of colouring matter as well as of other surface-active compounds.

Let us assume that technical solutions have been completely decolorized. In that case it is the content of non-sugars in the solution that is of determining importance to the slowing down of the crystallization rate. The value of the coefficient η is then equal to the value of η_1 ($\eta = \eta_1$), since the value of coefficient η_2 is equal to one ($\eta_2 = 1$).

In Fig. 3 values of the coefficient η_1 determine the straight line described by the equation [20, 21].

$$\lg \eta_1 = -0.04762 \cdot NS \tag{3}$$

where NS — content of non-sugars in dry substance.

Table 4 shows the crystallization rate of sucrose determined experimentally for technical syrups and for syrups obtained after decolorizing of thin juice (column 1). By comparing the crystallization rate of sucrose in a pure solution and in an impure one the value of the slowing down crystallization rate — coefficient η was determined (column 2).

Dependence of the coefficient η_2 on the content of colouring matter and other surface-active compounds is described by the equation

$$\Delta \lg \eta_2 = -0.187 \frac{42 - NS}{42} \Delta \lg E_{560} \tag{4}$$

where NS — content of non-sugars in dry substance,

E_{560} — specific absorbancy of the solution, $\lambda = 560$ nm.

From equations (3) and (4) and basing on the results of an analysis of solutions as compiled in Table 2 the values of coefficients η_1 and η_2 were calculated followed by a product of these values, that is coefficient η'. The calculated values are presented in the three final columns of Table 4.

The differences between the value of coefficient η, determined experimentally on the basis of measurements and the value of coefficient η' calculated from the equation based on the results of the solution analysis are smaller than 5%, they average 2% to 3%. The small value differences of coefficient η determined in two different ways may be caused by some

Table 4. Sucrose crystallization from factory liquors and liquors of lower colour

Liquors	Measured crystallization rate dS/Adt		Coefficients calculated from equations (2), (3) and (4)		
	Coefficient				
	$mg \cdot m^{-2} \cdot min^{-1}$	η	η_1	η_2	η'
	1	2	3	4	5
Refined sugar	7080	1.000	1.000	1.000	1.000
	Factory liquors				
Mother liquor of 1st massecuite	1203	0.1700	0.3268	0.5218	0.1705
Mother liquor of 2nd massecuite	561	0.0793	0.1420	0.5514	0.0783
Mother liquor of 3rd massecuite	206	0.0295	0.0411	0.7001	0.0288
	Liquors from the decolorized thin juice				
Mother liquor of 1st massecuite	1670	0.2360	0.3680	0.6540	0.2400
Mother liquor of 2nd massecuite	770	0.1088	0.1750	0.6410	0.1122
Mother liquor of 3rd massecuite	282	0.0399	0.0575	0.7250	0.0417

Table 5. Coefficient of slowing down sucrose crystallization rate as a function of solution purity and specific absorbancy ($\eta = \eta_1 \cdot \eta_2$)

Purity	Non-sugars %	Coefficient η_1	Coefficient of slowing down sucrose crystallization rate η						
			Specific absorbancy, λ = 560 nm						
			0.040	0.100	0.200	0.400	1.000	2.000	5.000
98	2	0.80308	0.6481	0.5505	0.4866	0.4301	0.3653	0.3229	0.2743
96	4	0.64494	0.5253	0.4505	0.4007	0.3563	0.3052	0.2714	0.2324
94	6	0.51794	0.4270	0.3687	0.3299	0.2953	0.2549	0.2281	0.1970
92	8	0.41595	0.3467	0.3018	0.2717	0.2447	0.2129	0.1917	0.1669
90	10	0.33404	0.2814	0.2470	0.2237	0.2027	0.1779	0.1612	0.1414
88	12	0.26826	0.2284	0.2021	0.1842	0.1679	0.1486	0.1356	0.1199
86	14	0.21544	0.1854	0.1654	0.1517	0.1391	0.1241	0.1139	0.1016
84	16	0.17301	0.1505	0.1354	0.1249	0.1153	0.1037	0.0957	0.0861
82	18	0.13894	0.1222	0.1108	0.1029	0.0955	0.0866	0.0804	0.0729
80	20	0.11158	0.0992	0.0907	0.0847	0.0791	0.0724	0.0676	0.0618
78	22	0.08961	0.0805	0.0742	0.0697	0.0656	0.0604	0.0568	0.0524
76	24	0.07196	0.0653	0.0607	0.0574	0.0543	0.0505	0.0478	0.0444
74	26	0.05779	0.0530	0.0497	0.0473	0.0450	0.0422	0.0401	0.0376
72	28	0.04641	0.0431	0.0407	0.0399	0.0373	0.0352	0.0337	0.0319
70	30	0.03727	0.0350	0.0333	0.0321	0.0309	0.0294	0.0284	0.0270
68	32	0.02993	0.0284	0.0272	0.0264	0.0256	0.0246	0.0238	0.0229
66	34	0.02404	0.0230	0.0223	0.0218	0.0212	0.0205	0.0200	0.0194
64	36	0.01931	0.0187	0.0183	0.0179	0.0176	0.0172	0.0168	0.0164
62	38	0.01550	0.0152	0.0149	0.0147	0.0146	0.0143	0.0142	0.0139
60	40	0.01245	0.0123	0.0122	0.0121	0.0121	0.0120	0.0119	0.0118

Table 6. Comparison of crystallization rate of sucrose obtained from factory liquors and of liquors of lower colour (Table 4)

Liquors	Crystallization rate, $mg \times m^{-2} \times min^{-1}$		Increase of crystallization rate %
	Factory liquors	Liquors from the decolorized thin juice	
Mother liquor of 1st massecuite	1203	1670	39
Mother liquor of 2nd massecuite	561	770	37
Mother liquor of 3rd massecuite	206	282	35

Table 7. Comparison of sugar losses in molasses

		Standard juice purification	Juice purification and decolorization of thin juice
Sucrose in beet, average	°S	17.4	17.4
Thick juice purity	—	93.40	94.10
Molasses purity, approx.	—	60	60
Non-sugars in thick juice	g/100 g DS	6.60	5.90
Non-sugars in molasses	g/100 g DS of thick juice	6.60	5.90
Sucrose in molasses	ditto	9.90	8.85
White sugar	ditto	83.50	85.25
Sugar yield	% on beet	14.5	14.7
Known and unknown sugar losses	% on beet	1.18	1.18
Sugar losses in molasses	% on beet	1.72	1.52
Total sugar losses	% on beet	2.90	2.70
Diminution of sugar losses	% on beet	—	0.20

inaccuracy of preparing the solutions for crystallization of a definite concentration corresponding to supersaturation 1.10.

Basing on equation (4) straight lines were drawn in Fig. 3 illustrating values of the coefficient η_2 for solutions of a given colour. Specific absorbancy $E_{560} = 0.200$, $E_{560} = 1.000$, $E_{560} = 5.000$. As it can be seen the colour of the next syrup is five times higher than the previous one. It should be added that in technical solutions, for example the colour of the mother liquor of 3rd massecuite is also about five times higher than colour of the mother liquor of 1st massecuite. The changes of value of coefficient η_2 are represented depending on the purity of the syrup (Fig. 3).

Together with a decrease of the syrup purity the value of coefficient η_2 increases. In syrups of a purity close to 60 the coefficient $\eta_2 = 1.000$.

On the basis of the tests and theoretical considerations we can assume that equations (2), (3) and (4) make it possible to calculate, with a sufficient accuracy, the value of coefficient slowing down the crystallization rate. The only necessary thing is to determine the purity and colour of the syrup from which the sucrose crystallizes. The possibility of calculating the value of coefficient η based on an analysis of the syrup is of practical importance as it allows to define a priori the crystallization rate of sucrose from a given technical syrup in relation to the crystallization rate of sucrose from pure syrup. Table 5 shows values of coefficient η_1 and η as calculated from equations (2), (3) and (4) assuming a defined content of non-sugars in and colour of the syrup. It is possible, using this Table, to read off the value of coefficient slowing down crystallization rate η in a given technical syrup.

The performed measurements of crystallization rate of sucrose from technical syrups and from similar syrups prepared after decolorizing thin juice (Table 4) enables a numerical determination of the advantages gained due to decolorizing of thin juice. According to Table 6 the crystallization rate of sucrose from syrups of lower colour is by over 30% higher than the crystallization rate of sucrose from normal factory syrups. This shows that the boiling time of each massecuite can be cut down by about 30%, and therefore the flow capacity of all the vacuum pans increases. According to Łękawski [24] the decolorizing of thin juice permits a reduction in the number of the existing vaccum pans. This can be important in designing new sugar factories.

COMPARISON OF SUGAR LOSSES IN MOLASSES

At the time of the tests the sugar content of beets averaged 17.4°S, purity of thick juice 93.4, purity of molasses about 60, yield of sugar 14.5% on beets (Table 7).

Let us assume that the whole quantity of non-sugars contained in thick juice passes into the molasses. Therefore, in molasses of a purity of about 60, where there is 6.60 g of non-sugars in relation to a 100 g dry substance of thick juice, 9.90 g of sugar is retained in relation to a 100 g dry substance of thick juice.

In 100 g dry substance of thick juice there was 93.40 g of sugar: 9.90 g sugar passed into molasses and 83.50 g sugar was produced as white sugar.

Since the sugar content of beets was 17.4°S and the yield of sugar was 14.5%, total losses amounted to 2.9%; the known and unknown sugar losses were 1.18% and sugar remain in molasses was 1.72% (Table 7).

When calculating similar sugar losses in the case of decolorized juice it was found that sugar losses in molasses are 1.52% on beets. Assuming that the known and unknown sugar losses are the same, that is 1.18%

total losses amount to 2.70%. This means that the sugar yield from beets is 14.7% instead of 14.5%.

It was found on the basis of the tests made, that decolorizing of thin juice reduced sugar losses by 0.2% on beets.

CONCLUSIONS

Decolorizing of thin juice by means of granular activated carbon enables to obtain the following advantages.

1. Increase by 0.2% of the yield of sugar from beets.
2. Cutting down the crystallization time of sugar of all products by about 30%.
3. Reducing colour of the white sugar.
4. The process of decolorizing of juice and regenerating of activated carbon does not cause any pollution of environment.

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MOŻLIWOŚĆ ZMNIEJSZENIA STRAT CUKRU I PRZYSPIESZENIA KRYSTALIZACJI DZIĘKI ODBARWIANIU SOKU RZADKIEGO

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Streszczenie

Straty cukru w melasie, w normalnych warunkach przerobu buraków, wynoszą 10-12% licząc na masę cukru wprowadzonego wraz z burakami.

Badano szybkość krystalizacji sacharozy z roztworów cukrowniczych o różnym zabarwieniu. W tym celu przygotowano techniczne syropy międzykryształowe pobrane podczas krystalizacji cukru I, II i III rzutu oraz syropy międzykryształowe pobrane podczas krystalizacji cukru I, II i III rzutu z syropów o znacznie niższym zabarwieniu otrzymanych z odbarwionego soku rzadkiego (tab. 1 i 2). W specjalnie przygotowanej aparaturze (rys. 1) wykonano pomiary szybkości krystalizacji sacharozy z roztworów cukru rafinowanego, z technicznych syropów międzykryształowych oraz z syropów międzykryształowych o obniżonym zabarwieniu. Porównano zmierzone wartości liczbowe szybkości krystalizacji sacharozy i na tej podstawie wyliczono współczynnik hamowania szybkości krystalizacji η, zależny od zawartości niecukrów i zawartości ciał barwnych w syropie poddawanym krystalizacji (tabl. 3 i 4).

$$\eta = \eta_1 \cdot \eta_2$$

gdzie η — współczynnik określający hamowanie szybkości krystalizacji sacharozy w roztworach nieczystych,

η_1 — współczynnik hamowania szybkości krystalizacji zależny od zawartości niecukrów,

η_2 — współczynnik hamowania szybkości krystalizacji zależny od zawartości ciał barwnych i innych związków powierzchniowo czynnych.

Wyliczone zależności posłużyły do ułożenia równań

$$\lg \eta_1 = -0{,}04762\ Nc$$

$$\Delta \lg \eta_2 = -0{,}187 \frac{42 - Nc}{42} \Delta \lg E_{560}$$

gdzie Nc — zawartość niecukrów w roztworze

E_{560} — absorbancja właściwa roztworu, $\lambda = 560$ nm. Zmiany wartości liczbowej współczynników określających hamowanie szybkości krystalizacji sacharozy w zależności od ilości niecukrów i od zabarwienia roztworów przedstawiono na wykresie o siatce logarytmiczno-proporcjonalnej (rys. 3).

Wyliczono wartości liczbowe współczynnika η dla roztworów o czystości od 98 do 60 i o zabarwieniu odpowiadającemu absorbancji właściwej od 0,040 do 5,000 przy długości fali światła 560 nm (tab. 5).

Na podstawie wykonanych analiz porównano wielkość strat cukru w melasie w przypadku stosowania klasycznego sposobu oczyszczania soku oraz oczyszczania soku połączonego z odbarwianiem soku rzadkiego (tab. 7). Dzięki odbarwianiu soku rzadkiego stwierdzono następujące korzyści: a) znaczne zmniejszenie ilości niecukrów w odbarwionym soku, co wpływa na zmniejszenie ilości cukru pozostałego w melasie,

b) podwyższenie wydajności cukru białego oraz c) przyspieszenie krystalizacji poszczególnych cukrzyc o ok. 30%.

Na podstawie uzyskanych równań matematycznych oraz oznaczenia czystości i zabarwienia syropu, z którego krystalizuje się sacharozę można wyliczyć szybkość krystalizacji sacharozy z danego roztworu technicznego w stosunku do szybkości krystalizacji sacharozy z czystego roztworu.