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DECREASE OF SUGAR LOSSES AS A RESULT OF THIN JUICE DEIONIZATION IN AN ALKALINE MEDIUM

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Key words: sugar manufacture, deionization of thin juice.

An alkaline method of deionizing thin juice by the use of cation and anion exchange resins in ammonium and carbonate forms respectively, was developed. No increase of reducing sugars was found. No wastewater was formed. This method allows for a decrease of sugar losses in molasses and an increase of sugar yield from beets by about 1%.

During the last hundred years the people working in the sugar industry have been trying to find the causes of sugar losses in molasses. Normally in the technological process about 10-12% of sugar from beets passes into molasses. Studies have enabled, for example, to establish the amount of sugar retained by particular cations of alkali metals, alkaline earth metals and α -amino acids [1-5]. Therefore on the basis of cell juice composition it is possible to determine, to some extent, the technological value of sugar beets.

About forty years ago the attempts of introducing a new tool, i.e., ion exchangers, did not lead to satisfactory results. It seemed that it would be easy to remove non-sugars from juice and subsequently to decrease the amount of sugar remaining in molasses. There was an opinion that it would be possible to increase the sugar yield by 7-8% on beets [6]. Unfortunately deionization of juices in hydrogen and hydroxyl demineralization runs met with serious difficulties because of the susceptibility of sucrose of hydrolysis what caused the need of cooling the juice below 14°C [7, 8]. Ion exchangers, and particularly anion exchangers were poisoned quickly. Another difficulty was the formation of highly loaded wastewater

during the regeneration of ion exchangers. Therefore other methods of deionization of sugar juice were developed (8-11).

Since many years at the Technical University of Łódź has been studying the problem of application of ion exchangers in the sugar industry (12-17). As a basic principle of deionization of juices it was assumed that the process should be carried out in an alkaline medium to prevent sucrose hydrolysis and that the by-products ought to be utilised with no wastewater formed.

EXPERIMENTAL

Basing on our previous research further tests were conducted on a laboratory and a pilot scale. On these grounds the following procedure was adopted.

For deionization of thin juice a strongly acidic polystyrene cation exchanger and a strongly basic polystyrene anion exchanger were applied. To avoid the hydrolysis of sucrose the deionization of sugar juices was carried out using resins in an ammonium and a carbonate form. Therefore it was necessary to pretreat the juice to remove the excess of calcium salts and colouring matter, otherwise the calcium ions would be adsorbed by the cation exchanger, and colouring matter would irreversibly poison the anion exchanger [18].

A glass apparatus were used in tests on a laboratory scale, and steel apparatus in tests on a pilot scale. Thin juice was taken from a sugar factory after the main carbonation and filtration processes.

At the time of the test, sugar content in sugar beet slices was 17.4% and known and unknown sugar losses were about 1.18%. The thick juice purity was 93.05. Purity of molasses slightly exceeded 60, the content of sugar in molasses was 1.82% on beets.

1. DELIMING OF THIN JUICE

Lime milk was added (0.1% on beets) to the clear juice, after main carbonation and filtration, and the juice was finally carbonated. The suspension was introduced into a reactor and at the same time ammonium carbonate was added (Fig. 1). A considerable excess of ammonium ions, in relation to the content of calcium ions in the purified juice, was added [19, 20]. After 20 minutes the juice was filtered to separate the precipitated calcium carbonate.

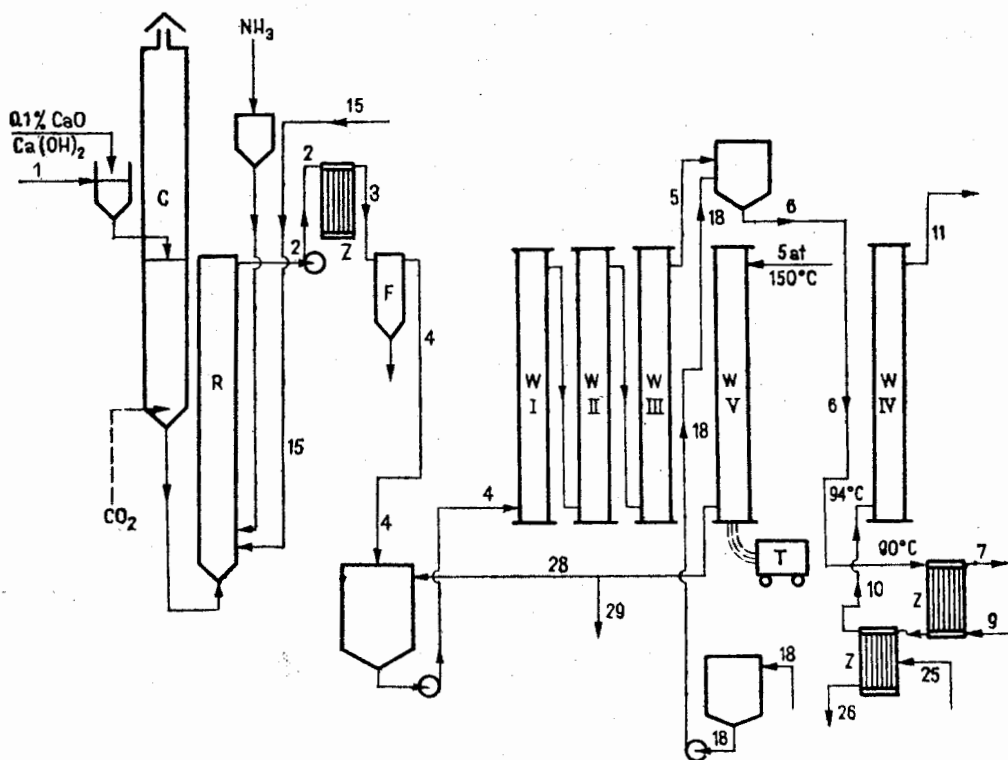


Fig. 1. Delimiting and decolorization of thin juice; C — carbonator, R — reactor, Z — heat exchanger, F — filter, W — column with granular activated carbon, W I, W II, W III — set of columns for decolorizing of delimited thin juice, W IV — column with fresh regenerated carbon for decolorizing of deionized thin juice, W V — activated carbon in column — electrothermal regeneration, T — AC-transformer, 1 — juice to delimiting, 2 — suspension with precipitated CaCO_3 , 3 — heated suspension, 4 — clear delimited juice, 5 — juice after decolorizing, 6 — juice to deionization, 7 — cooled juice, 9, 10 — deionized juice, 11 — redecolorized juice, 15 — ammonium carbonate, 18 — juice remaining from the ion exchange unit — recycled to decolorized thin juice, 28 — juice remaining from the activated carbon unit — recycled to delimited thin juice, 29 — exhausted steam after electrothermal regeneration of activated carbon.

2. DECOLORIZATION OF THIN JUICE

The hot delimited juice was decolorized by means of granular activated carbon by the multicolumn method [21]. The delimited juice, at a temperature nearing the boiling point, was introduced in series into I, II, and III column (Fig. 1). The juice was introduced into the bottom of the column and flowed up, according to the principle of natural current. This prevents the aeration of the bed of activated carbon. The exhausted bed of activated carbon was sweetened-off with downflow condensate. The above procedure makes it possible to remove preliminarily about 80% of colour-

ring matter and surface-active substances from the thin juice with the use of 1% granular activated carbon in relation to the dry substance in the juice.

The decolorization process takes place in a series of three columns. At the same time the regeneration of activated carbon takes place in column V which was exhausted in the previous cycle of decolorization. After removing the residue of juice from the column by means of hot condensate, the regeneration of activated carbon takes place at a 520°C and lasts from 3 to 5 hours [22]. The overheated steam flows through the column and removes the gasified compounds which were previously adsorbed by activated carbon. It was found out that the granular activated carbon can be regenerated 20 times and used again for decolorization of thin juice. In this way the consumption of fresh activated carbon can amount to 0.05%-0.10% only in relation to the dry substance of the decolorized juice.

Column IV, containing activated carbon directly after its regeneration, is used in the first sequence for the removal of the residue of colouring matter from the deionized juice. At the same time the broken beads of ion exchangers, which could be carried away by the deionized juice, are arrested. Owing to this the deionized juice and then white sugar are protected against trace impurities of ion exchangers. In the further stage column IV is used for the decolorization of thin juice as the final column before deionization.

Because of the series system of connection of the decolorizing columns delimed juice is next introduced into column II and subsequently flows through columns III and IV. In this procedure every 6 or 8 hours the whole system is changed over by one column according to the rule of countercurrent.

Table 1 presents, as an example, an analysis of delimed and decolorized juice. It shows that the purity of juice has been increased and the colour decreased. The activated carbon has retained the other surface-

Table 1. Delimed thin juice and decolorized thin juice analysis

		Factory thin juice	Delimed thin juice	Decolorized thin juice
Purity	—	93.05	93.2	93.8
pH	—	8.9	8.9	8.7
Reducing sugars	g/100 g DS	0.049	0.049	0.054
Lime salts	mg CaO/100 g DS	143	28	24
Effect of deliming	%	—	80.4	83.2
Specific absorbancy $\lambda = 560$ nm	—	0,121	0.130	0.026
Effect of decolorization	%	—	—	80

active compounds as well. The delimed and decolorized juice does not cause poisoning of ionic groups of ion exchangers. Owing to this the same resin and particularly the anion exchangers can live up to three times longer.

3. DEIONIZATION OF THIN JUICE

The system for the deionization of thin juice consists of four pairs of columns filled up with cation and anion exchange resins (Fig. 2).

The juice, after being decolorized, is cooled down in a heat exchanger to 60°C and immediately introduced into the bottom of the first column with the cation exchanger and flows up. Then it passes through the column with the anion exchanger in the same direction. The partly deionized juice flows through the second pair of freshly regenerated ion exchange columns. The deionization effect is more than 90% and the deionization process lasts for a relatively short time. The juice flows through the cation exchange column in about 10 minutes and in 15 minutes through the anion exchange column. The exchange capacity of the anion exchange column was by 50% higher than that of the cation exchange column.

As the resins for deionization have the ammonium and carbonate forms, the juice is always in an alkaline medium and there is no danger for the sucrose to be hydrolyzed. The deionized juice is heated up in a heat exchanger to 90°C and introduced into the column with freshly regenerated activated carbon. The decolorized and the deionized thin juice contains a considerable amount of ammonium and carbonate ions.

As mentioned before deionization takes place in series in two pairs of columns with the cation and the anion exchangers. At the same time the two other pairs of columns with the ion exchangers are sweetened-off with condensate in counter-current to the direction of juice. Then the anion exchanger columns are regenerated in series with 2N (10%) ammonium carbonate solution, again in counter-current to the direction of juice. The use of the natural current principle allows for a considerable reduction of juice dilution as well as dilution of regenerating solutions. The regenerating effluent comprises non-sugars removed from the juice and a certain surplus of ammonium carbonate.

4. THICKENING OF REGENERATING EFFLUENT

After regeneration of ion exchangers the solution of ionic non-sugars flows out from the bottom of the cation exchanger column. The solution is directed to a small three-effect evaporator in which it is thickened to above 50% of dry substance. The whole amount of ammonium carbonate is distilled from the non-sugar solution which after condensation is retur-

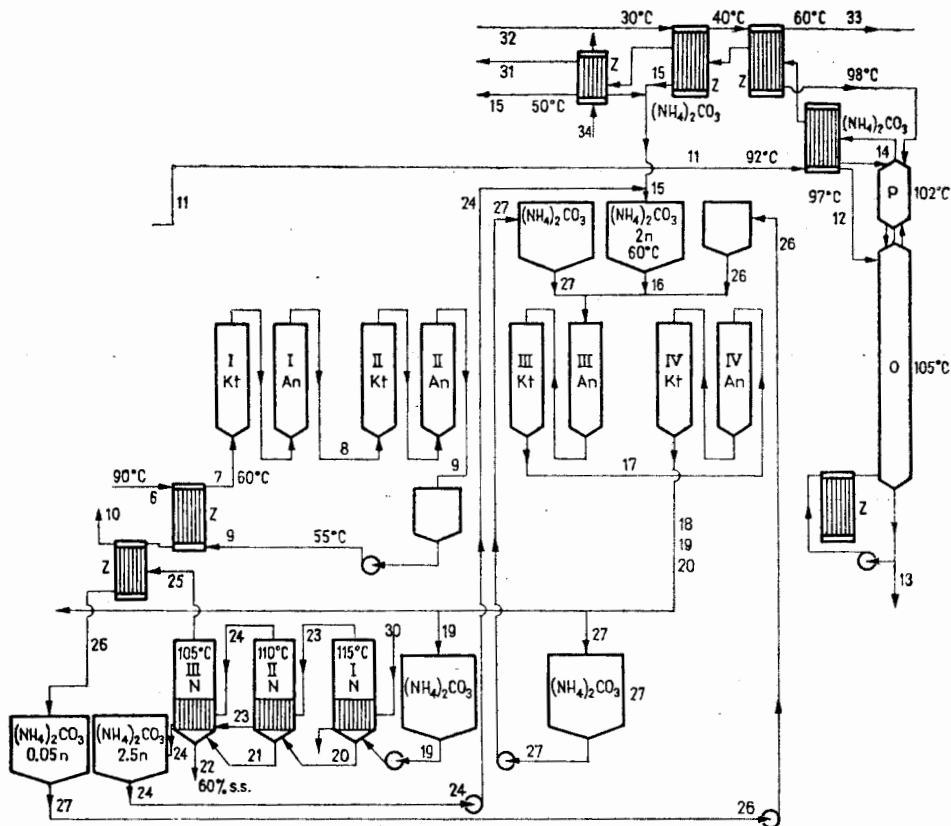


Fig. 2. Deionizing of thin juice, thickening of regenerating effluent with non-sugars and removal of ammonium carbonate from deionized thin juice; Z — heat exchanger, I — IV — set of columns for deionization, Kt — cation exchanger in ammonium form, An — anion exchanger in carbonate form, N I, N II, N III — three-effect evaporator, O — fluid bed distiller with packing, P — distiller 6 — juice to deionization, 7 — cooled juice, 8 — preliminary deionized juice, 9 — deionized juice, 10, 12 — heated juice, 11 — deionized juice for ammonium carbonate reclaiming, 13 — juice after ammonium carbonate removal, 14 — vapours with ammonium carbonate, 15 — 2N (10%) solution of ammonium carbonate for regeneration of the resins, 16 — ammonium carbonate for the final regeneration, 17 — partly used ammonium carbonate solution for the preliminary regeneration of the resins, 18 — juice remaining from the ion exchange unit — recycled to thin juice, 19 — regenerating effluent for non-sugars for thickening, 20 — partly thickened regenerating effluent, 21 — thickened regenerating effluent, 22 — thickened non-sugars directed to pulp, 23-26 — vapours condensate with ammonium carbonate, 27 — regenerating effluent with unused ammonium carbonate recycled to ammonium carbonate solution, 30 — vapours for heating of nonsugars solution evaporator, 31 — gases to vacuum pump, 32 — cold raw juice, 33 — heated raw juice, 34 — cold water

ned for regeneration of the ion exchanger bed in the following deionizing runs. Fig. 2 shows a diagram of the evaporator for thickening of the regenerating effluent and recovery of ammonium carbonate, The thickened ionic non-sugars separated from this juice can be used as an addition to beet pulp before being dried, this increases its fodder value. Owing to the use of ammonium carbonate in the closed cycle and the utilization of the separated non-sugars in the process of deionization of juices no wastewater is formed.

5. REMOVAL OF AMMONIUM CARBONATE FROM DEIONIZED THIN JUICE

As mentioned before, the decolorized and deionized juice contains a considerable amount of ammonium and carbonate ions in the place of the removed non-sugars of ionic character.

To separate ammonium carbonate a fluid bed distiller with packing was applied (Fig. 2). The vapours from the distiller are condensed in a raw juice heater. A concentrated solution of ammonium carbonate is obtained, which is returned either for the purpose of deliming thin juice or for the regeneration of ion exchangers.

Because of the small ammonium carbonate losses the required amount of ammonia water is added to the reactor in which the deliming of juice is carried out. To supplement the number of carbonate ions, a certain surplus of gas from the lime kiln is introduced during the final carbonation of juice.

After distillation of ammonium carbonate in a fluid bed distiller the hot juice has a purity of about 97. Table 2 shows an analysis of thin juice

Table 2. Analysis of factory thin juice, thin juice deionized by alkaline method and thickened deionized juice

		Factory thin juice	Delimed thin juice	Decolorized thin juice	Deionized thin juice	Thickened deionized juice
Dry substance, refr.	%	14.3	14.2	14.1	13.8	65.8
Purity	—	93.05	93.20	93.80	96.90	97.00
pH	—	8.8	8.6	8.6	8.6	8.3
Alkalinity	g CaO/100 cm ³	0.014	0.012	0.012	0.010	0.006
Reducing sugars	g/100 g DS	0.050	0.040	0.030	0.035	0.040
Lime salts	mg CaO/100 g DS	143	28	24	12	10
Sulphated ash, gravim.	g/100 g DS	2.36	2.37	2.36	0.10	0.10
Specific absorban- cy = 560 nm	—	0.163	0.164	0.030	0.003	0.006

purified by standard method and juice deionized in an alkaline medium. It should be added that the deionized juice is characterised by good thermostability.

In order to protect the evaporator against corrosion a small quantity of lime water is added to the deionized juice, so that the juice should contain about 12 mg CaO/100 g of dry substance. During the thickening process of the deionized juice no foaming is observed and the colour increase is very small.

The thick juice can be used for the production of refined sugar. It may also be applied directly as liquid sugar in different branches of the food industry, for example in the confectionery industry or for sweetening soft juices and alcoholic drinks.

6. ADVANTAGES RESULTING FROM THE DEIONIZATION OF THIN JUICE IN AN ALKALINE MEDIUM

The essential advantage resulting from the deionization of thin juice in an alkaline medium is a decrease of sugar losses in molasses (Table 3).

Table 3. Decrease of sugar losses in molasses owing to the deionization of thin juice

		Standard juice purification	Juice purification and deionization of thin juice	Difference
Sucrose in beet, average	°S	17.4	17.4	0.0
Thick juice purity	—	93.05	97.00	+3.95
Molasses purity	—	60.0	60.0	0.0
Non-sugars in thick juice	g/100 g DS	6.95	3.00	-3.95
Non-sugars in molasses	g/100 g DS	6.95	3.00	-3.95
	of thick juice			
Sucrose in molasses	ditto	10.43	4.50	-5.93
White sugar	ditto	82.52	92.50	+9.98
Sugar yield	% on beet	14.40	15.35	+0.95
Known and unknown sugar losses	% on beet	1.18	1.30	+0.12
Sugar losses in molasses	% on beet	1.82	0.75	-1.07
Total sugar losses	% on beet	3.00	2.05	-0.95

It was found out that when applying the standard juice purification method, the sugar content in molasses was 1.82% on beets. The deionization of juice allows to reduce the amount of molasses-sugar to 0.75% on beets. This means that sugar losses in molasses decrease by about 1% on beets. A certain increase of known and unknown sugar losses, for example from 1.18% to 1.30% on beets should be anticipated. The yield of sugar

will be then 15.35% instead of 14.4% on beets. If the thickened juice is used as liquid sugar the yield of sugar will be about 16%.

Compared with the standard juice purification method the increase of sugar yield, owing to the deionization of thin juice, will be 6.5% in relation to sugar. In case of producing liquid sugar the yield increase will be 11% in relation to sugar, as there will be no molasses. At the same time no crystallization of sugar, centrifuging and packing will be necessary.

It should be added that the presented process of deionizing juices and production of liquid sugar allows for a considerable heat economy and therefore a decrease of energy consumption. The process calls for the addition of a small amount of relatively cheap chemicals which remain in a closed circuit. The regeneration of granular activated carbon and the regeneration of ion exchangers do not cause environment pollution.

CONCLUSIONS

a. The resins for deionization are in the ammonium and carbonate forms and there is no danger for the sucrose to be hydrolyzed.

b. The delimed and decolorized thin juice does not cause poisoning of ionic groups of ion exchangers. Owing to this, the anion exchanger can live up to three times longer.

c. Owing to the use of ammonium carbonate in a closed cycle and the utilization of separated non-sugars — no wastewater is formed.

d. Thin juice deionization allows for a 1% increase of the sugar yield from beets. In case of producing liquid sugar the sugar yield may be increased by 1.5%.

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OBNIŻENIE STRAT CUKRU WSKUTEK ODSALANIA SOKU RZADKIEGO W ŚRODOWISKU ALKALICZNYM

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Streszczenie

Opracowano alkaliczną metodę odsalania soków w cyklach amonowym i węglanowym w celu zabezpieczenia sacharozy przed hydrolizą.

Wymieniacze jonowe są łatwo blokowane przez jony wapniowe oraz związki koloidowe i powierzchniowo czynne występujące w soku. Z tego względu oczyszczony sok rzadki wstępnie odwapniano metodą amoniakalną oraz odbarwiano kolumnowo stosując ziarnisty węgiel aktywny. Odbarwiony sok rzadki, po ochłodzeniu go do temp. 60°C, odsalano za pomocą dwóch par kolumn kationitowych i anionitowych. W wyniku odsalania następowało zwiększenie czystości soku rzadkiego z 93 do 97 i całkowite odbarwienie koku. Nie stwierdzono wzrostu zawartości inwertu. Po oddestylowaniu nadmiaru węglanu amonowego sok można zagęścić do 67% s.s. i stosować jako tzw. płynny cukier. Odzyskany węgiel amonowy zawraca się do regeneracji jonitów, a zagęszczony roztwór niecukrów zostaje skierowany do wysłodków paszowych. Dzięki temu w procesie odsalania nie tworzą się żadne ścieki. Metoda została wypróbowana w skali mikrotechnicznej.

Odsalanie soku rzadkiego w środowisku alkalicznym pozwala na zmniejszenie strat cukru w melasie a jednocześnie na zwiększenie o około 1% wydajności cukru z buraków.