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EXTRACTION OF NITROGEN COMPOUNDS FROM BEET COSSETTES TO RAW JUICE IN MODIFICATION CONDITIONS OF FEED WATER COMPOSITION

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Key words: beet sugar technology, modification of extractant, nitrogen compounds in juice

During beet cossettes extraction besides sucrose many other compounds, for instance nitrogen compounds, pass to the juice, too. Modified water extractants were used containing aluminium sulfate, calcium chloride and calcium hydroxide. The least amount of total nitrogen passed to the juice using calcium chloride.

During the process of beet cossettes extraction, besides sucrose, non-sugars pass to raw juice, too. Among other things there are nitrogen compounds. They are essential components of raw juice and particularly "harmful nitrogen" are of an important consideration in the technological process. They take active part in many reactions which are disadvantageous from the point of view of sugar technology.

In the process of extraction the application methods of an addition of various chemical substances [3, 7, 19, 24, 26, 27] with the purpose to improve the extraction process [1, 2, 6, 12, 14, 17, 18, 21, 25] are known. It is possible to improve simultaneously conditions of the pulp pressing [3-5, 8, 15, 28], owing to this, fuel consumption decreases during the drying of pulp [3].

Chemical substances applied in the extraction process usually have acidic character, e.g. sulfuric acid, gaseous sulfur dioxide, aluminium sulfate [12, 14, 15, 21, 25, 26, 28]. They also often contain calcium ions, e.g. calcium chloride, calcium sulfate, calcium bisulfite or calcium hydroxide [2, 7, 8, 13, 19, 24]. It has been known the effect of these substances on pectins. Crosslinking and stiffening of pectins follows under the effect of calcium, aluminium or hydrogen ions, which improves the pressing of pulp. It has been known also, that the least amount of pectins decomposes in acidic environment (pH = 4.5) [3].

Application of chemical substances in the extraction process was the subject of investigations in many countries, not only on a laboratory scale but also on a technical scale [1-3, 5-10, 13, 15, 16, 19, 21-23, 26].

The aim of our research was investigation of passing of the nitrogen compounds from beet cossettes to raw juice in the model extraction process, in modification conditions of feed water composition.

EXPERIMENTAL PART

In the process of extraction, carried out on the laboratory scale, selected model solutions were used, containing aluminium sulfate or calcium chloride. It has been proceeded also the beet cossettes extraction, previously sprinkled with calcium hydroxide.

The process of cossettes extraction was carried out in the apparatus set presented in Fig. 1. The apparatus consisted of extractor equipped with heating jacket through which flowed water of 80°C, peristaltic pump, causing juice to circulate and also thermostat, where was placed a container of the circulating juice.

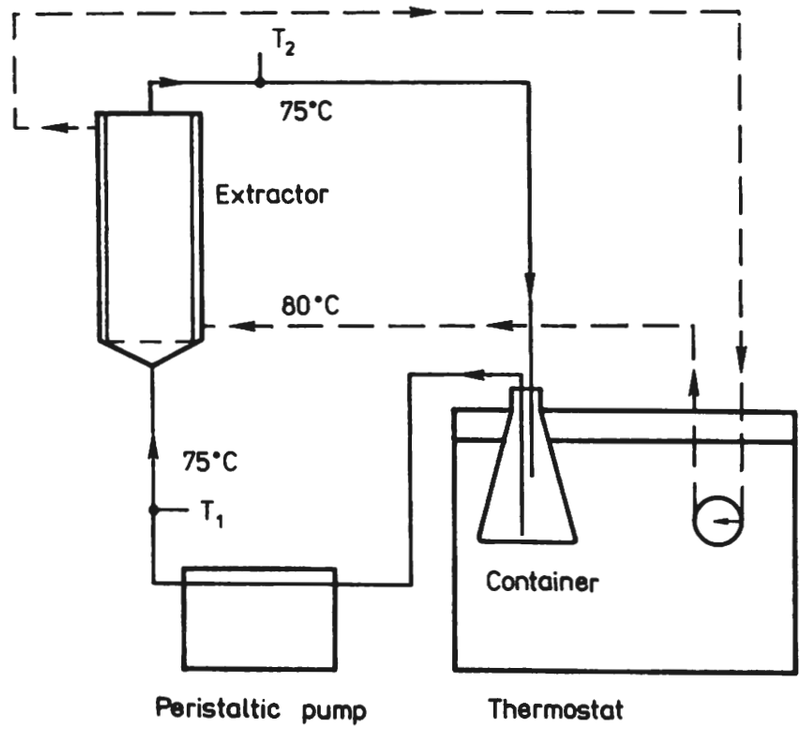

Fig. 1. Apparatus for extraction

650 g of beet cossettes and 720 g of extracting solution, previously heated to a temperature of 80°C were introduced into the extractor. At first, for 8 minutes, the extraction proceeded without forcing of juice circulation. Than, for 22 minutes, the extraction was carried out with juice circulation. The process was interrupted and the first batch of juice quickly was received from extractor, circulation conduits and juice container (so called raw juice I). The second stage of extraction proceeded by means of successive batch 1000 g of *a model* solution, at 80°C. Just as in the first stage, the extraction without forcing of juice circultion lasted 8 minutes and with juice circulation — 22 minutes. After finishing the second stage of extraction, so called raw juice II was received. The temperature of flowed out

Table 1. α-aminoacid nitrogen content and total nitrogen content in beet cossettes, in raw juice and in pulp in various conditions

Beet cossettes*)			Extractant composition			α-aminoacid nitrogen, mg/100 g of product			Total nitrogen content, mg/100 g of product		
Pol	α-N	total N		g of cation per 1 kg of beet cossettes	pH	juice I	juice II	pulp	juice I	juice II	pulp
%	mg/100 g										
16.2	42.8	142	tap water	0	7.5	17.0	8.0	3.8	56.1	23.8	67.6
			$Al_2(SO_4)_3$	0.021	6.5	21.4	7.3	3.7	51.4	20.5	47.9
				0.043	6.0	32.0	10.7	9.5	51.4	20.2	57.5
				0.064	5.5	27.4	10.3	6.4	50.0	18.5	65.4
				0.086	5.0	15.3	7.7	6.5	45.2	18.6	73.3
16.1	36.9	140	distilled water	0	5.5	15.0	6.7	8.2	53.6	21.0	26.5
			$CaCl_2$	0.140	5.5	13.6	7.2	8.8	46.9	20.0	71.1
				0.284	5.5	14.2	6.6	11.8	45.7	18.7	75.3
				0.880	5.5	14.5	6.4	16.5	41.8	15.7	82.4
15.1	56.2	184	distilled water	0	5.5	29.4	9.4	6.5	80.3	23.2	57.4
			$Ca(OH)_2$	0.357	5.5	30.7	13.1	12.4	88.9	37.5	55.5
				0.536	5.5	23.7	12.5	15.3	87.9	39.0	64.3
				0.714	5.5	24.1	13.2	10.0	87.8	45.3	44.6
				1.428	5.5	23.8	12.3	13.2	81.6	39.9	42.5

*) average values from 8-10 experiments

Table 2. Extraction grade of sucrose and nitrogen compounds from beet cossettes to raw juice*) in various conditions of extraction process (average results)

Extractant composition			Extraction grade α-N, %			α-N content, % pulp	Extraction grade total N, %			Total N content, % pulp	Extraction grade of sucrose, %			Sucrose content, % pulp
	g of cation per 1 kg of beet cossettes	pH	juice I	juice II	juice I+II		juice I	juice II	juice I+II		juice I	juice II	juice I+II	
tap water	0	7.5	55	35	90	10	42	23	65	35	54	30	84	16
$Al_2(SO_4)_3$	0.021	6.5	60	31	91	9	43	24	67	33	54	29	83	17
	0.043	6.0	57	31	88	12	40	23	63	37	53	31	84	16
	0.064	5.5	58	31	89	11	40	21	61	39	54	29	83	17
	0.086	5.0	51	34	85	15	36	21	57	43	53	30	83	17
Distilled water	0	5.5	48	31	79	21	49	28	77	23	55	30	85	15
$CaCl_2$	0.140	5.5	46	30	76	24	36	21	57	43	54	30	84	16
	0.284	5.5	41	28	69	31	34	21	55	45	54	29	83	17
	0.880	5.5	38	25	63	37	32	18	50	50	54	30	84	16
Distilled water	0	5.5	59	27	86	14	51	21	72	28	54	29	83	17
$Ca(OH)_2$	0.357	5.5	58	32	90	10	52	26	78	22	53	30	83	17
	0.536	5.5	48	31	79	21	50	19	69	31	54	30	84	16
	0.714	5.5	53	29	82	18	53	31	84	16	52	30	82	18
	1.428	5.5	49	31	80	20	49	30	79	21	52	31	83	17

*) The extraction grade of sucrose and nitrogen compounds (also content in pulp) was calculated in relation to their content in cossettes, taken as 100%

juice amounted to 74-76°C. Research methodology was worked out on the grounds of preliminary experiments.

In beet cossettes and in pulp, α-aminoacid nitrogen was determined by Kubadinow's and Wieninger's method in the digest [11], while the total nitrogen by the mineralization method of Kjeldahl [20].

Investigations were carried out from October to January. Sugar beets were delivered from sugar factory in October and stored in cold store at + 4°C. In the last period of investigations the quality of sugar beets significantly deteriorated (Tab. 1). Beet cossettes polarization decreased from 16.2% to 15.1%. α-amino-acid nitrogen content increased 0.25 g to 0.37 g per 100 g S (from 43 mg to 56 mg per 100 g of cossettes), while the total nitrogen content increased from 0.88 g to 1.22 g per 100 g S (from 142 mg to 184 mg per 100 g of cossettes).

Choice of appropriate chemical substances doses applied in the extraction process was fixed with the help of literature data [2, 7-9, 13, 23, 24] and preliminary investigations.

In the case of experiments with aluminium sulfate $Al_2(SO_4)_3$, to the reference extraction and to making of model solution, tap water was used [14]. In the other experiments, distilled water was used. In the case of experiments with calcium hydroxide, the beet cossettes was sprinkled with suspension of calcium hydroxide and remained in a covered vessel in the period of 10 minutes, at 20°C. Than, two-stage extraction was carried out by means of distilled water. All the experiments were repeated twice or three times.

Basing on analytical determinations (Tab. 1) and mass balance, the amount of nitrogen compounds passing to raw juice and remaining in pulp was calculated. Then, the extraction grade of nitrogen compounds to raw juice in relation to the amount of nitrogen compounds in cossettes, assumed as 100% (Tab. 2), was calculated.

DISCUSSION OF RESULTS

In the first series of the experiments, the model solutions of aluminium sulfate of concentration from 0.021 g Al to 0.086 g Al per 1 kg of cossettes, were used. pH-value of feed water appropriately varied from 6.5 to 5.0 pH. The extraction grade of α-aminoacid nitrogen and total nitrogen content from cossettes to juice I, juice II and the percentage remainder of nitrogen in pulp are shown in Fig. 2 and 3 (Tab. 2). As one know, the extraction grade of α-aminoacid nitrogen and total nitrogen content in successive experiments nearly did not depend on the quantity of aluminium ions dose and was similar to results obtained in the reference extraction with tap water (Tab. 2). The extraction grade of α-aminoacid nitrogen from beet cossettes to raw juice (I + II) amounted from 85% to 91%, just as in the reference extraction with tap water, where it amounted to 90% (Tab. 2). The extraction grade of total nitrogen amounted from 57% to 67% and in the

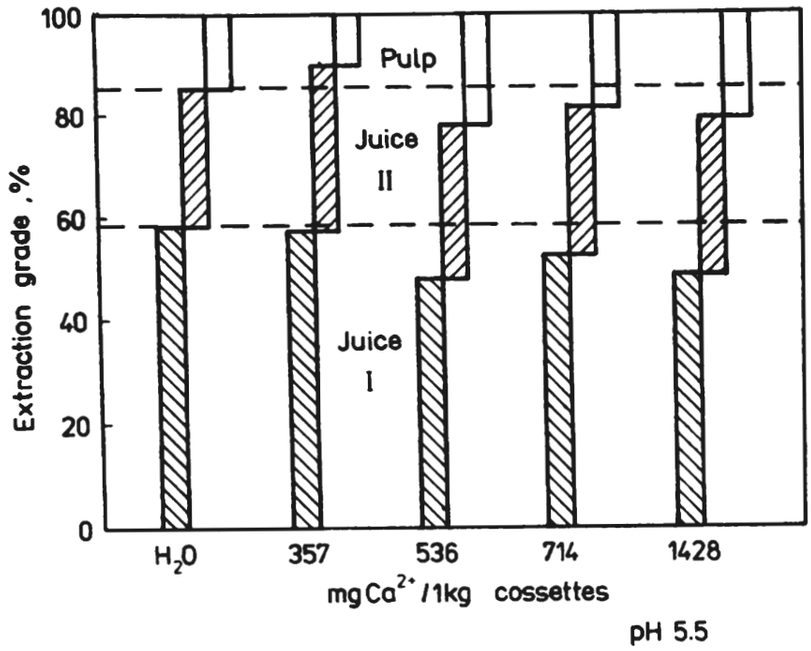

Fig. 2. Extraction grade of α-aminoacid compounds from beet cossettes to raw juice and their residue in pulp. Extractant — tap water with $Al_2(SO_4)_3$

reference extraction amounted to 65%. The extraction grade of sucrose in these experiments amounted from 83% to 84%.

In the second series of the experiments, distilled water was modified by means of calcium chloride added in the amount from 0.140 g Ca to 0.880 g Ca per 1 kg of cossettes (Tab. 2, Fig. 4, 5). The lowest extraction grade of α-aminoacid nitrogen from cossettes to raw juice (63%) was obtained in the case of the highest dose of $CaCl_2$, amounting 0.88 g Ca per 1 kg of cossettes. In the reference extraction with

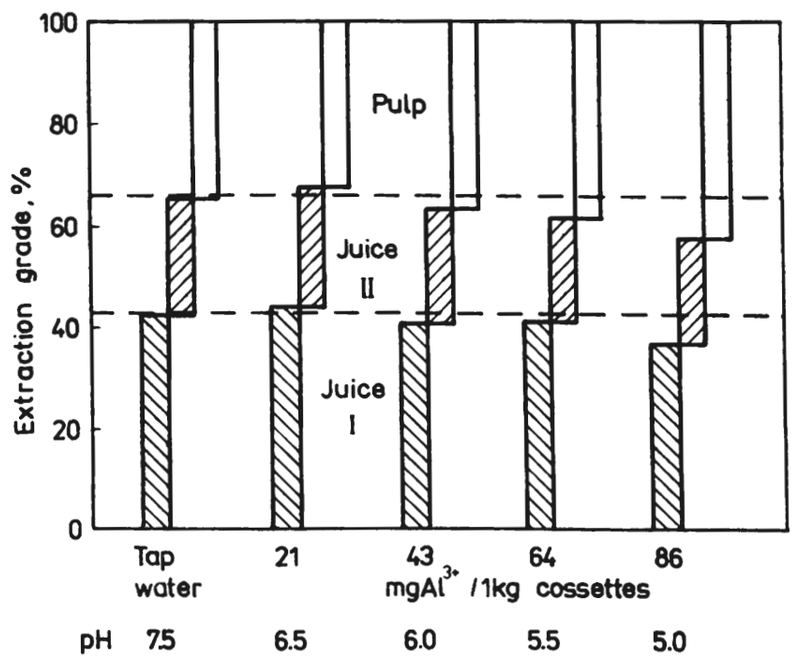

Fig. 3. Extraction grade of total nitrogen from beet cossettes to raw juice and its residue in pulp. Extractant — tap water with $Al_2(SO_4)_3$

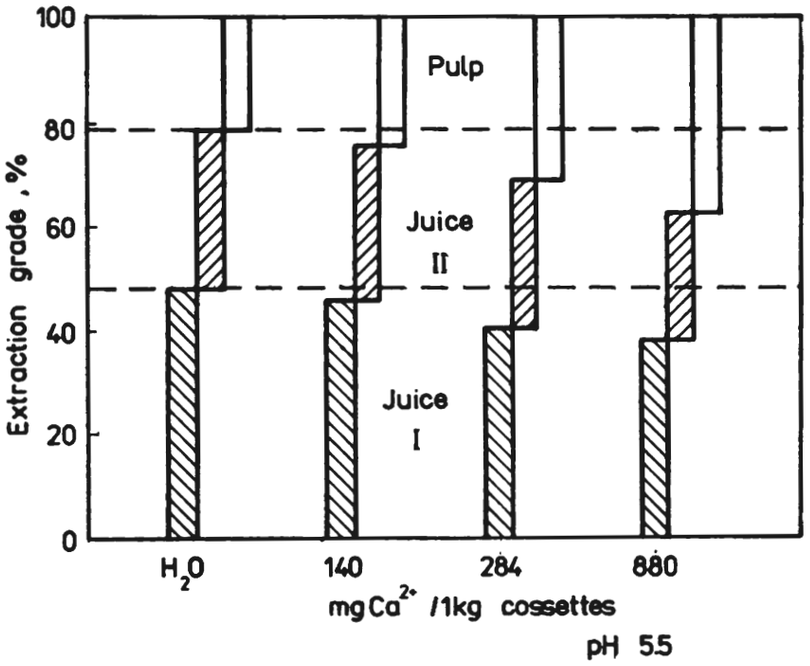

Fig. 4. Extraction grade of α-aminoacid compounds from beet cossettes to raw juice and their residue in pulp. Extractant — distilled water with $CaCl_2$

distilled water, the extraction grade of α-aminoacid nitrogen amounted to 79%. Also, the extraction grade of the total nitrogen content was the lowest (50%) in these conditions. In reference extraction, the extraction grade of the total nitrogen content amounted to 77%; in pulp remained at that time 23% of nitrogen, that is about two times less than as a result of the extraction with an addition of $CaCl_2$ (0.88 g Ca per 1 kg of cossettes). The extraction grade of sucrose was similar to the results of the first series of experiments and amounted from 83% to 84% (extraction with distilled water — 85%).

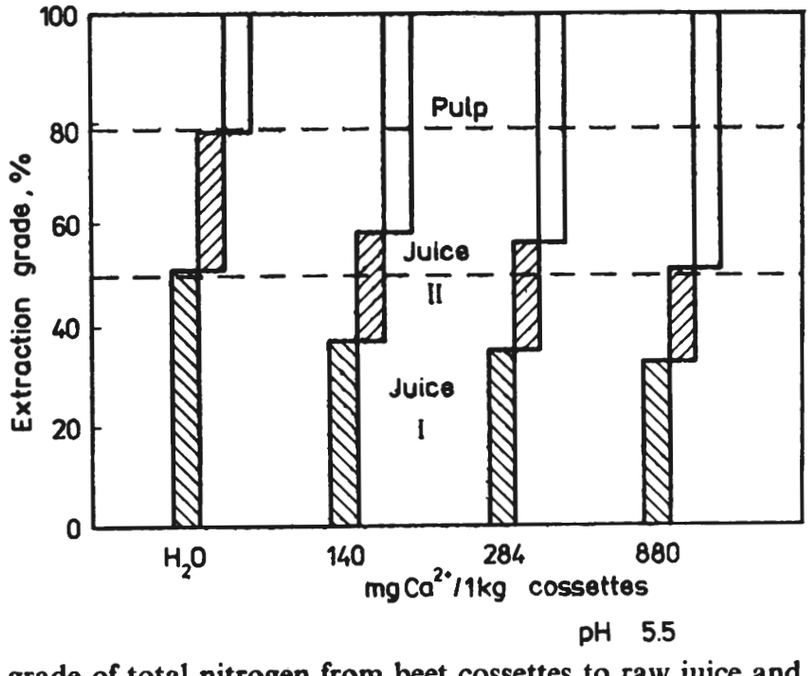

Fig. 5. Extraction grade of total nitrogen from beet cossettes to raw juice and its residue in pulp. Extractant — distilled water with $CaCl_2$

In the third series of the experiments, for the sprinkling of cossettes before extraction, calcium hydroxide solution containing from 0.357 g to 1.428 g CaO per 1 kg of cossettes, was used (Tab. 2., Fig. 6, 7). The pass grade of α-aminoacid

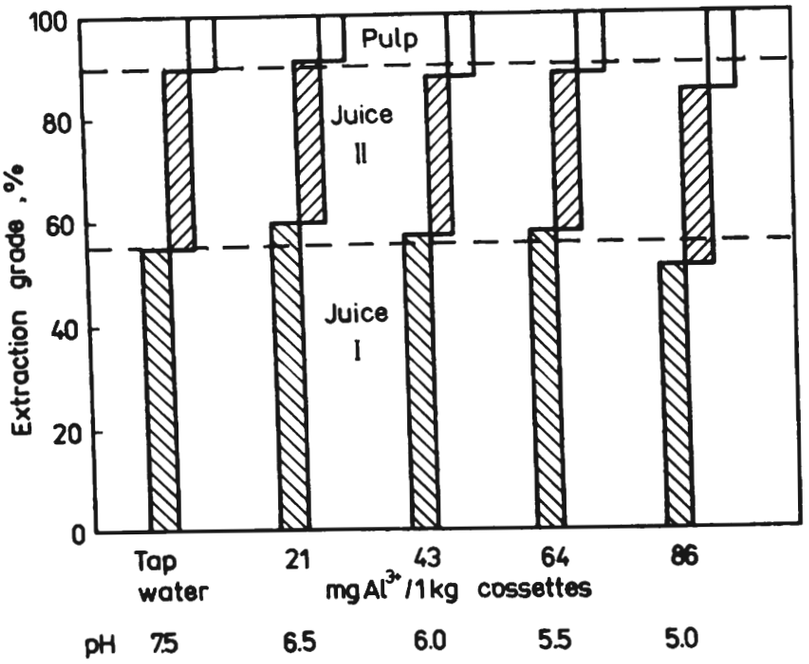

Fig. 6. Extraction grade of α-aminoacid compounds from beet cossettes to raw juice and their residue in pulp. Cossettes sprinkled with $Ca(OH)_2$, extractant — distilled water

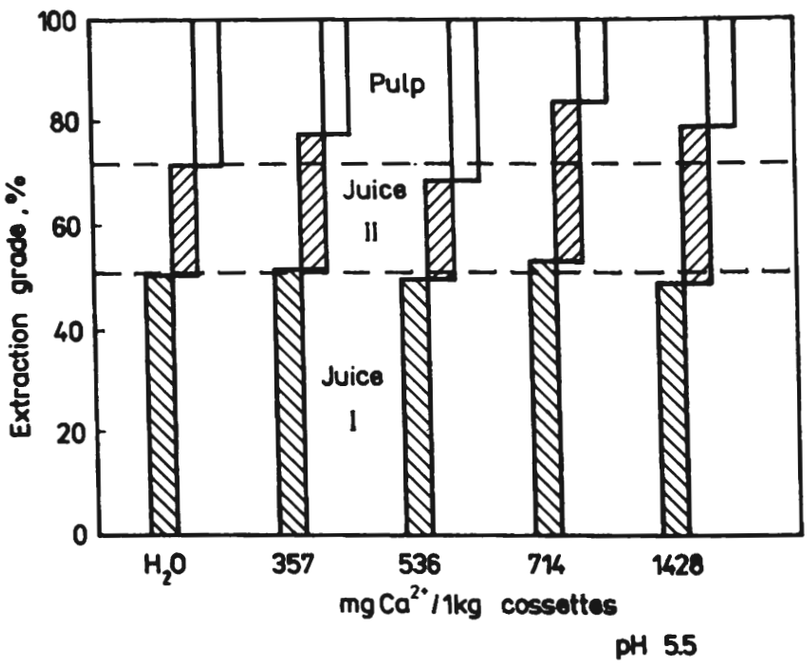

Fig. 7. Extraction grade of total nitrogen from beet cossettes to raw juice and its residue in pulp. Cossettes sprinkled with $Ca(OH)_2$, extractant — distilled water

nitrogen from beet cossettes to the juice (I+II) comparison to the extraction grade of α-aminoacid nitrogen in control experiments with distilled water (79%-90% and 86%). In the same experiments the extraction grade of the total nitrogen content was also similar to the results of the extraction with distilled water (69-84% and 72%). Observed in successive experiments changes of the

extraction grade of nitrogen compounds could be caused by the fact, that cossettes surface was not precisely covered by suspension of calcium hydroxide, in consideration of relatively little volume of used suspension of calcium hydroxide [10]. The extraction grade of sucrose was similar to the results of previous series of the experiments and amounted from 82% to 84% (extraction with distilled water — 83%).

An interesting material presents the comparison of nitrogen compounds extraction and simultaneous sucrose extraction (Tab. 3). In the first series of the experiments, the quality of sugar beets was similar. The cossettes extraction by means of tap water of pH = 7.5 caused, that α-aminoacid nitrogen content had been more by a 30% in the II batch of juice (0.281 g/100 g S) and the total nitrogen content more by a 14% on sugar (0.821 g/100 g S), than in the I batch of juice. The cossettes extraction by distilled water of pH = 5.5 gave approximate results. In the second batch of juice α-aminoacid nitrogen content was more by a 21% (0.229 g/100 g S) and the total nitrogen content more by a 5% (0.710 g/100 g S). Examining the average results of successive cossettes extraction by means of tap water modified by aluminium sulfate it has been found out, that the same amount of nitrogen compounds pass to juice I and to juice II, too. It was similarly in the case of extractant modified by calcium chloride — the total nitrogen content was the same in both juices. Whereas, the amount of α-aminoacid nitrogen, on sugar, was more by a 19% (0.218 g/100 g S) in juice II than in juice I.

The extraction of nitrogen compounds from cossettes obtained from beets of deteriorated technological value (III series of the experiments) proceeded completely differently. It has been stated that α-aminoacid nitrogen content was more by a 50% and the total nitrogen content more by a 40% in cossettes, on sugar; reducing substances content was more by a 70%. During the cossettes extraction by means of distilled water most of nitrogen compounds went to the juice I. It has been found out, that in the juice II α-aminoacid nitrogen content was less by a 11% (0.348 g/100 g S) and the total nitrogen content less by a 20% (0.857 g/100 g S). Examining the average results of successive extractions of cossettes sprinkled with calcium hydroxide of various concentration, it has been found out the inverse situation. In the juice II α-aminoacid nitrogen was more by a 33% and the total nitrogen content more by a 24%, on sugar, than in the juice I.

SUMMATION

Summing up the results of our investigation one can state that the quantity of the dose applied chemicals (aluminium sulfate, calcium chloride, calcium hydroxide) did not have the considarable effect on the extraction grade of nitrogen compounds from cossettes to raw juice. The extraction grade of sucrose amounted hardly about 85%, which was connected with experiments methodology proceeded in laboratory conditions.

Table 3. Nitrogen compounds content in beet cossettes and in raw juice on 100 g of sugar

Beet cossettes					Extractant composition			α-aminoacid nitrogen, g/100 g S		Total nitrogen content, g/100 g S	
Pol	reducing compounds*)		α-N	total N		g of cation per 1 kg of beet cossettes	pH	juice I	juice II	juice I	juice II
%	%	g/100 g S	g/100 g S								
16.2	0.066	0.409	0.264	0.875	tap water	0	7.5	0.217	0.281	0.718	0.821
					$Al_2(SO_4)_3$	0.021	6.5	0.281	0.256	0.700	0.720
						0.043	6.0	0.361	0.369	0.685	0.697
						0.064	5.5	0.376	0.353	0.675	0.636
						0.086	5.0	0.198	0.241	0.587	0.582
16.1	0.081	0.520	0.229	0.869	distilled water	0	5.5	0.189	0.229	0.674	0.710
					$CaCl_2$	0.140	5.5	0.176	0.210	0.609	0.587
						0.284	5.5	0.181	0.231	0.585	0.656
						0.880	5.5	0.191	0.214	0.554	0.519
15.1	0.123	0.826	0.372	1.218	distilled water	0	5.5	0.393	0.348	1.080	0.857
					$Ca(OH)_2$	0.357	5.5	0.396	0.458	1.146	1.315
						0.536	5.5	0.333	0.469	1.238	1.472
						0.714	5.5	0.352	0.454	1.281	1.565
						1.428	5.5	0.345	0.513	1.183	1.664

*) reducing compounds and α-aminoacid nitrogen determined in water extract (the digest) from beet brei

The biggest differences were stated during the extraction with an addition of calcium chloride. The extraction grade of α-aminoacid nitrogen from cossettes to raw juice amounted from 63% to 76%, in pulp remained from 37% to 24%. Whereas, the extraction grade of total nitrogen content amounted from 50% to 57%. Less nitrogen compounds underwent from cossettes to raw juice, owing to this more ones remained in pulp. In the case of the extraction with distilled water, the extraction grade of α-aminoacid nitrogen amounted to 79% and of the total nitrogen — 77%. In the case of dose of $CaCl_2$ amounting to 0.88 g Ca per 1 kg of cossettes, in pulp remained about two times more nitrogen compounds than in pulp from the reference extraction, i.e. with distilled water.

Comparing the results of our experiments one can find that considerable more nitrogen compounds passed to raw juice during the extraction with an addition of aluminium sulfate or calcium hydroxide than using calcium chloride. The addition of aluminium sulfate caused that the extraction grade of α-amino-acid nitrogen had amounted from 85% to 91% and the extraction grade of the total nitrogen content from 57% to 67%. In the case of sprinkling the cossettes with calcium hydroxide the extraction grade of α-aminoacid nitrogen amounted from 79% to 90%, while the extraction grade of the total nitrogen content amounted from 69% to 84%.

On the grounds of the experiments carried out, it has been found out that the least amount of nitrogen compounds passed from the cossettes to raw juice using for the extraction the solution of $CaCl_2$. However, as one know, calcium chloride has melassigenic features. That is why, e.g. in England, it has been replaced by less melassigenic calcium sulfate [24]. In Austria a little amount of calcium hydroxide is added to supply a suitable amount of calcium ions in order to stiffen the pulp tissue, and to increase pH-value of the juice in the extractor only insignificantly.

CONCLUSIONS

1. During the beet cossettes extraction, in laboratory conditions, by means of model solutions of water with an addition of aluminium sulfate, calcium chloride or calcium hydroxide, only slight effect of the dose of separate chemical substances on the pass grade of nitrogen compounds to raw juice has been found out.

2. The extraction grade of sucrose amounted hardly about 84%, which was connected with the methodology of experiments proceeding in laboratory conditions.

3. The least amount of nitrogen compounds passed to raw juice when the extractant with calcium chloride was used: below 70% of α-aminoacid nitrogen and about 55% of the total nitrogen content. Unfortunately, calcium chloride has melassigenic features.

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EKSTRAKCJA ZWIĄZKÓW AZOTOWYCH Z KRAJANKI BURACZANEJ

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Streszczenie

W procesie ekstrakcji, oprócz sacharozy, do soku surowego przechodzą także inne substancje. Między innymi są to związki azotowe, które niekorzystnie wpływają na przebieg dalszych procesów jednostkowych. W celu polepszenia warunków ekstrakcji dodaje się różne substancje chemiczne, które jednocześnie mogą ułatwiać wyżymanie wysłodków, co pośrednio wpływa na oszczędność energii podczas suszenia wysłodków.

Celem pracy było zbadanie stopnia ekstrakcji związków azotowych z krajanki do soku surowego, w warunkach modelowych, z zastosowaniem wybranych substancji chemicznych.

Badania prowadzono w skali laboratoryjnej. Do ekstrakcji 1 kg krajanki stosowano roztwory modelowe zawierające różne ilości odczynników: siarczan glinowy 21 do 86 mg Al, chlorek wapniowy 140 do 880 mg Ca oraz wodorotlenek wapniowy 357 do 1428 mg Ca. W krajance buraczanej, a

następnie w soku surowym oraz w wysłodkach oznaczano azot α-aminokwasowy metodą Kubadinowa i Wieningera oraz ogólną zawartość azotu metodą mineralizacji Kjeldahla. Obliczono stopień ekstrakcji związków azotowych z krajanki do soku surowego i pozostałość tych związków w wysłodkach.

Stwierdzono, że najmniej związków azotowych (ogólna zawartość azotu) przechodziło do soku surowego w przypadku stosowania chlorku wapniowego — stopień ekstrakcji wynosił ok. 55%. W analogicznych doświadczeniach z dodatkiem siarczanu glinowego stopień ekstrakcji związków azotowych wynosił ponad 60% a w przypadku wodorotlenku wapniowego blisko 80%. Nie stwierdzono wpływu zmiany wielkości dawki użytej substancji chemicznej na stopień przechodzenia związków azotowych z krajanki do soku surowego.