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A LABORATORY APPARATUS FOR VACUUM EXTRACTION

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A vacuum extraction apparatus for laboratory extraction of 1 kg of beet cossettes has been designed, built and tested. The extractor allows for comparisons between the quality of raw juice obtained under correct conditions of extraction with that obtained from the same cossettes under practical conditions. The apparatus can be used for determining the technological value of beets not only in a sugar factory but also in sugar beet seed breeding stations.

One of the essential processes in the sugar industry is extracting sucrose from sugar beet cossettes. Many apparatus have been introduced since battery diffusion has been replaced by continuous extraction. These devices, in spite of many advantages, bring inferior results in terms of juice quality [1-4]. It should be mentioned that the technological value of beet and quality of cossettes are of importance for the quality of raw juice. A suitable laboratory extraction unit [5-8] is necessary to make a decision whether inferior juice quality is caused by an unsuitable extraction process or poor quality of beet. A comparison of raw juice quality with the quality of industrial raw juice leads to the question whether the extraction process carried out in a sugar factory suits the quality of beet, and which parameters of the extraction process should be changed to achieve raw juice of best quality.

Having this in mind, a laboratory apparatus for vacuum extraction of cossettes has been designed, built and tested.

APPARATUS AND METHODS

The details of the apparatus are presented in Fig. 1. The apparatus consists of four parts: *1* — container for cossettes, *2* — extracting vessel heated by steam jacket, *3* — vacuum juice receiver, *4* — vacuum water spiral cooler.

The cossettes are placed in a vertical cylindrical container 1 with a sieve bottom. The container is placed inside extracting vessel 2. Condensate flows in at the top part of the vessel and raw juice flows down at the bottom through an overflow pipe 5; sensors of a continuous conductivity meter 7 [9] are placed in the pipe 6.

Raw juice flows gravitationally to a container where it is heated under reduced pressure to the boiling point. The heating element 9 is

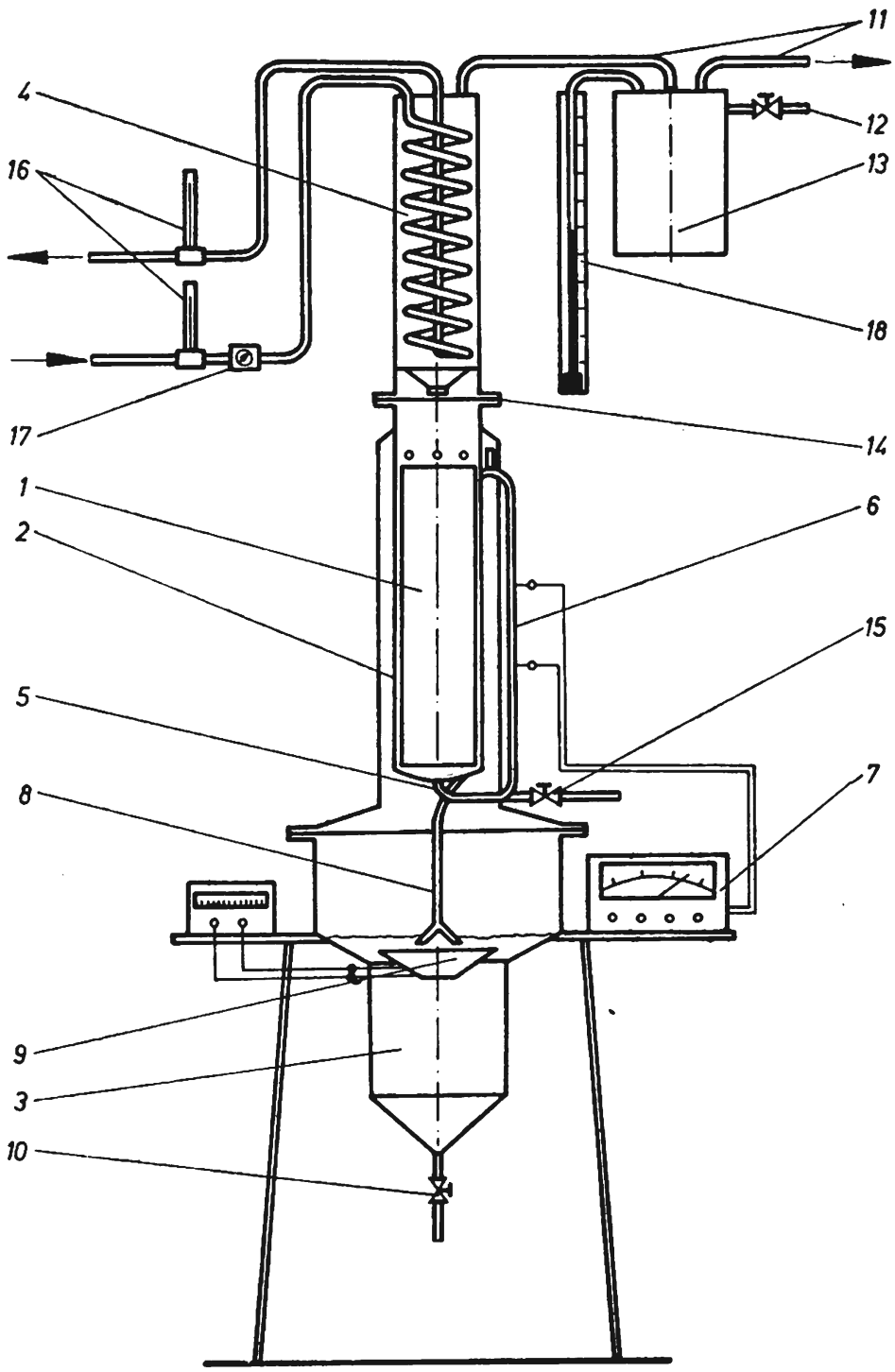

Fig. 1. Laboratory apparatus for vacuum extraction; 1 — container for cossettes, 2 — extracting vessel heated with steam jacket, 3 — vacuum juice receiver, 4 — vacuum spiral cooler, condenser of vapours, 5 — outflow of juice with overflow pipe, 6 — inductometer for measurement of juice conductivity, 7 — conductivity meter, 8 — juice outflow from the inductometer, 9 — heating element, 10 — raw juice valve, 11 — vacuum pipe, 12 — air valve, 13 — vacuum vessel, 14 — vacuum cooler flange, 15 — pulp water valve, 16 — cooling water thermometers, T_1 and T_2, 17 — water-meter, 18 — vacuum-gauge, mm Hg

placed inside the receiver, 20 mm below juice level. Juice vapours flow through the heating jacket of the extracting vessel and condense in the water spiral cooler. The condensate flows down directly to the cossettes container. The amount of vapours and condensate being in circulation is adjusted so that the sucrose extraction process is completed in 75 to 80 minutes. In the receiver 3 there is 110% to 120% juice on cossettes. A continuous conductivity measurement of flowing out raw juice allows for an indirect measurement of the degree of cossettes exhaustion.

The laboratory vacuum extractor has been designed for extracting sucrose from 1 kg of cossettes. In the juice receiver 3 there is about 1200 cm^3 of juice. The extractor works under reduced pressure which can be adjusted in the range from 35.0 kPa to 50.0 kPa. Owing to this the process can be carried out at a constant temperature within the limits 70°C to 80°C. The dependence of boiling temperature of water on pressure is presented in Table 1.

Table 1. Boiling temperature of water depending on pressure

Pressure kPa	Vacuum mm Hg	Boiling temperature °C
35.0	497.2	72.0
40.0	459.5	75.1
45.0	422.0	78.0
50.0	384.6	80.6

EXTRACTION OF BEET COSSETTES

For the experiment several kilograms of cossettes are taken and carefully mixed. From a batch of about 300 g cossettes a brei is prepared, and the sugar content is determined by polarimetric analysis. The remainder of the brei about 200 g, is centrifuged in a dry basket centrifuge and the juice obtained is filtered through a thin cotton-wool layer to trap any remaining brei. Then the juice is deaerated. The dry substance content of the juice is then determined refractometrically and the sucrose content by polarimetric analysis.

Before the experiment is started the extracting vessel is connected to the juice receiver. 1000 g of cossettes are introduced into the container with the sieve bottom. Then 1000 g of boiling distilled water is poured into the extracting vessel, and the container with the cossettes is introduced. After complete immersion of the cossettes in water the surface is covered with a sieve disc and another portion of 1200 g of boiling distilled water is added. The juice from the extracting vessel flows down to the juice receiver. The vapour condenser is immediately connected to the extracting vessel, a flange 14 is tightened and cold water inflow is

connected to the coils; the water flows through a water-meter at a rate of about 2 dm^3/min. At the same time the vacuum valve is open to keep the pressure in the apparatus at about 45.0 kPa (about 420 mm Hg), so that the extraction process is carried out at a temperature of about 78°C.

After the pressure has been adjusted the heating of the juice receiver is connected. Based on the measurement of temperature difference between the feed water and the water outflowing from the condenser, and taking into account the flow rate of the water through the condenser, it is possible to calculate the flow rate of the condensate introduced to the cossettes. The temperature of water flowing out from the condenser should not exceed 30°C. The pressure in the apparatus should not vary, and to achieve this an automatically adjusted pressure valve is applied.

The extraction process is carried out until the conductivity of the outflowing juice is reduced to 0.4 mS/cm. When the experiment is finished the heating of the apparatus is disconnected. When the temperature of outflowing water from the condenser is equal to that of inlet water, the input of water is stopped, and air is introduced into the apparatus by means of valve 12. The raw juice, after cooling, is emptied by valve 10 and pulp water by valve 15 to an amount of about 1000 cm^3. After the condenser has been disconnected the vessel containing exhausted cossettes is taken out and the products are analysed.

DISCUSSION OF RESULTS

Table 2 gives some typical results of such an extraction experiment. The process was carried out at a temperature of about 78°C (422 mm Hg). At the beginning the conductivity of raw juice was above 5 mS/cm, after 30 minutes it was 3 mS/cm, and after 75 minutes it was below 0.4 mS/cm, which indicates that the extraction process was carried out in a correct manner and was completed.

Table 3 presents the results of analysis of cossettes and the end products of the extraction. The raw juice is of a much higher purity than the cell juice. From 1000 g of the cossettes 169 g sucrose in the form of raw juice was obtained. About 2 g of sucrose remained in the pulp, and 1 g of sucrose in pulp water, which in total makes 0.3% sucrose. There were practically no unknown sugar losses.

Table 4 presents the results of analysis of the extraction process under practical conditions at a sugar factory. To make a better comparison of the laboratory results with practical ones these results have been recalculated on the basis of 1000 g cossettes. The draft at the sugar factory was 120% on beets. Purity of the raw juice was 89.0 i.e. not much higher than that of beet juice. 0.4% sucrose remained in the exhausted cos-

Table 2. Extraction of cossettes in laboratory vacuum extractor
Introduced: cossettes 1000 g, distilled water 2200 g

Hour	Pressure	Vacuum	Conductivity 75°C	Temperature °C			Water flow
	kPa	mm Hg	mS/cm	T_1	T_2	ΔT	dm^3/5 min
10^{00}	45.0	422	0.0	9.0	9.0	0	10
10^{05}	44.7	424	5.02	9.0	19.0	10.0	12
10^{10}	45.0	422	5.49	9.0	20.0	11.0	13
10^{15}	45.0	422	5.30	9.0	20.5	11.5	13
10^{20}	45.3	420	4.65	9.0	21.0	12.0	13
10^{25}	45.0	422	3.80	9.0	21.0	12.0	13
10^{30}	45.0	422	2.92	9.0	20.5	11.5	13
10^{35}	45.0	422	2.32	9.0	20.5	11.5	13
10^{40}	45.0	422	1.82	9.0	21.0	12.0	12
10^{45}	44.6	425	1.46	9.0	20.5	11.5	13
10^{50}	44.5	418	1.18	9.0	20.5	11.5	13
10^{55}	45.0	422	0.98	9.0	20.5	11.5	13
11^{00}	45.0	422	0.82	9.0	21.0	12.0	12
11^{05}	45.0	422	0.69	9.0	21.0	12.0	12
11^{10}	45.0	422	0.57	9.0	20.5	11.5	13
11^{15}	45.0	422	0.46	9.0	20.5	11.5	13
11^{20}	100.0	0	0.39	9.0	12.0	3.0	0

Obtained: raw juice 1180 g
exhausted cossettes 920 g
pulp water 1030 g
3130 g
Introduced: cossettes + water 3200 g
Evaporated 70 g

Table 3. Analysis of cossettes and extraction products

	Mass	Dry subst. ref.	Purity	Sucrose		Non-sugar
	g	%		%	g	g
Cossettes	1000	—	—	17.2	172	—
Cell juice	925	21.0	88.6	18.6	172	22.2
Raw juice	1180	16.0	89.8	14.3	169	19.6
Pulp	920	—	—	0.2	1.8	2.0
Pulp water	1030	0.16	62	0.1	1.0	0.6

Unknown losses
$172 - (169 + 1.8 + 1.0) = 172 - 171.8 = 0.2$ G
$\frac{0.2}{1000} = 0.02\%$ on beet

settes. The balance shows that the unknown losses are 0.25% sucrose on beet. The cossettes remained in the extractor for about 2 hours.

It should be added that in the laboratory extractor we obtain more than 1 kg of raw juice. This is a sufficient quantity to perform different other analytical determinations when required.

Table 4. Analysis of extraction process in sugar factory

	Mass	Dry subst. ref.	Purity	Sucrose		Non-sugar
	g	%		%	g	g
Cossettes	1000	—	—	17.2	172	—
Cell juice	925	21.0	88.6	18.6	172	22.2
Raw juice	1200	15.5	89.0	13.8	165.5	20.4
Sucrose losses in pulp	1000	—	—	0.4	4.0	—

Unknown losses

$172 - (165.5 + 4.0) = 172 - 169.5 = 2.5$ g

$\frac{2.5}{1000} = 0.25\%$ on beet

Similar analytical results were obtained from many experiments when sound beet were processed, and suitable parameters of extraction process were kept. The unknown losses at the sugar factory were about 0.2% on beet.

The results of the experiments were quite different when the beet used were not completely ripe, or of reduced technical value due to freezing followed by thawing. The results were much better in the laboratory extractor than in the sugar factory. Depending on the state of beet, suitable extraction parameters were selected; extraction time was shortened or temperature reduced. Taking into account optimum extraction results under laboratory conditions, parameters of technological process were modified accordingly.

On the basis of the experiments performed the following conclusions may be drawn.

1. The laboratory apparatus for vacuum extraction can be successfully used for controlling the extraction process at a sugar factory. In sugar beet breeding stations it can be used to assess the technological value of individual varieties of beet.

2. It is possible to state, on the basis of laboratory extraction results, whether the quality of beet and cossettes permits production of raw juice of an acceptable purity, and it is also possible to define, in an approximation, the extraction output of sucrose from beet.

3. Extraction performed in a laboratory makes it possible to establish whether low purity of raw juice at a sugar factory is caused by poor

quality of cossettes, unsuitable parameters of the extraction process, or a too long retention of a part of the cossettes in the sugar extraction plant.

LITERATURE

1. Wrężel A.: Gaz. Cukrown., 1961, **63**, 268.
2. Schliephake D., Mechias B., Reinefeld E.: C.R. 15 Ass. Gen. CITS, Wien 1975, 31.
3. Cronewitz Th. et al.: Ind. Alim. Agr., 1978, **95**, 771.
4. van der Poel P. W. et al.: Z. Zuckerind., 1973, **98**, 71.
5. Zagrodzki S., Zagrodzki S. M. jr: Gaz. Cukrown., 1963, **71**, 9-14; Z. Zuckerind., 1964, **14**, 568.
6. Asselbergs C. J. et al.: Int. Sugar J., 1960, **62**, 95.
7. Zagrodzki S.: Zuckerind., 1979, **29**, 206.
8. Henry J., Vandewijer R., Pieck R.: Zesz. Probl. Post. Nauk Roln. 1966, **62 b**, 49.
9. Zagrodzki S., Zagrodzki S. M. jr: Gaz. Cukrown., 1970, **78**, 229; Zucker 1970, **23**, 504.

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APARAT LABORATORYJNY DO EKSTRAKCJI PRÓŻNIOWEJ

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Streszczenie

Opracowano, zbudowano i zbadano ekstraktor próżniowy do laboratoryjnej ekstrakcji 1 kg krajanki buraczanej. Ekstraktor umożliwia porównanie jakości soku surowego otrzymanego w prawidłowych warunkach ekstrakcji z jakością soku surowego otrzymanego z takiej samej krajanki w ekstraktorze technicznym.

Ekstraktor (Fig. 1) składa się z pojemnika krajanki *1*, naczynia ekstrakcyjnego ogrzewanego płaszczem parowym *2*, próżniowego zbiornika soku *3* oraz chłodnicy próżniowej *4*. Krajankę buraczaną umieszcza się w pojemniku *1* w kształcie pionowego walca, zakończonego w dolnej części sitem. Pojemnik napełniony krajanką wstawia się do naczynia ekstrakcyjnego *2*, do którego uprzednio wlano 1000 g wrzącej wody destylowanej. Przed rozpoczęciem procesu ekstrakcji również w zbiorniku *3* znajduje się 1200 g wrzącej wody destylowanej.

Wewnątrz zbiornika *3* znajduje się element grzejny *9*, umieszczony 20 mm poniżej poziomu soku. Para wodna płynie przez płaszcz grzejny naczynia ekstrakcyjnego *2* i skrapla się w chłodnicy *4*. Temperaturę wody chłodzącej mierzą termometry *16* a natężenie przepływu wodomierz *17*. Skropliny z chłodnicy *4* spływają do pojemnika z krajanką *1*. Sok surowy spływa z dolnej części naczynia *2* przez rurę przelewową *5*, w której są umieszczone czujniki ciągłego induktometru *6*. Sok spływa grawitacyjnie *8* do zbiornika *3*, w którym pod zmniejszonym ciśnieniem zostaje ogrzany do temperatury wrzenia.

W zbiorniku soku *3* znajduje się 110%—120% soku w stosunku do masy krajanki. Ilość oparów i skroplin w obiegu jest tak dobrana, aby proces ekstrakcji cukru został zakończony w czasie 75 min do 80 min. Ekstraktor pracuje pod zmniejszonym ciśnieniem *11*, które mierzy się za pomocą manometru rtęciowego *18*. Ciśnienie reguluje się w zakresie od 35,0 kPa do 50,0 kPa, dzięki czemu proces ekstrakcji można prowadzić w stałej temperaturze, w granicach od 70°C do 80°C. Przebieg procesu ekstrakcji jest kontrolowany na podstawie ciągłego pomiaru 7 konduktywności soku surowego wypływającego z naczynia ekstrakcyjnego *2*. Podczas ekstrakcji konduktywność zmienia się od około 5 mS/cm do 0,4 mS/cm. Po zakończeniu procesu ekstrakcji wyrównuje się ciśnienie w aparacie (zawór *12*). Następnie spuszcza się sok surowy (zawór *10*) i wodę wysłodkową (zawór *15*). Wszystkie produkty waży się i analizuje w celu wykonania bilansu mas.

Aparat może służyć do określania wartości technologicznej buraków bezpośrednio w cukrowni a także w stacjach hodowli buraków cukrowych. Porównanie jakości soku surowego otrzymanego w laboratoryjnym ekstraktorze próżniowym z jakością fabrycznego soku surowego pozwala na dobranie optymalnych parametrów procesu ekstrakcji krajanki w warunkach technicznych.