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(54) Process for the recovery of disaccharides from disaccharides containing tuberous plants by means of an extraction with unwarmed water.

(57) Process for the recovery of disaccharides from disaccharides containing tuberous plants by mechanically reducing the tuberous plants to a mush, extracting the reduced material with water of less than 60°C, subjecting the extract first to an ultrafiltration and then to a demineralisation and finally concentrating the obtained solution. In the process also mangels having a sugar content of 6–12% by weight can be used as starting material.

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Process for the recovery of disaccharides from disaccharides containing tuberous plants by means of an extraction with unwarmed water.

The invention relates to a process for the recovery of disaccharides from disaccharides containing tuberous plants according to which a solution of disaccharides in water is first subjected to ultrafiltration and subsequently to deionization.

Such a process according to which diffusion juice obtained by extracting cut sugar beets with warm (35-80°C) water is subjected to ultrafiltration by continuously filtrating it through a cellophane tube surrounded by other unassailable tissue tubes within a steel tube in a turbulent way is known from "Warenkennis en Technologie" Vol. 4, 81-87 (1957). According to said method all colloids are kept within the tube without clogging the membrane and finally pressed out of the tube. The sugar solution containing all the salts passes the membrane sterile and is removed through a small opening in a lower part of the steel tube.

The so-obtained sterile thin juice can be completely freed of all salts by means of cation- and anion exchangers so that a solution is obtained which completely corresponds with the refined juice. By working in this way a sugar is recovered which does not contain molasses as a by-product which generally contains at least 1/6 of the sugar from the beets and is inferior in comparison with sugar.

The colloidal products which have been removed separately contain proteins, glutamic acid, threonine, proline and phenyl alanine which are more valuable than molasses.

As far as known this known process is not applied on technical scale.

However, because the sugar industry which recovers sugar from sugar beets is constantly confrontated with increasing costs for energy and wages the following facets of the known process are subjected to an examination for promoting a more paying exploitation viz.

a) is it possible to find a solution for the limited keeping qualities of sugar beets namely two - three months because after a longer storage time the sugar content decreases.

b) is it possible to carry out the extraction of the sugar from the tuberous plants, especially the sugar beet in a more economic way.

c) is it possible to separate the extract and the pulp more efficiently.

d) is it possible to remove the contaminations from the sugar solution in such a way that purification by crystallizing the sugar from sugar solutions is not necessary anymore.

ad.a.

many years ago proposals have been made to improve the keeping qualities of sugar beets for instance by blowing cold air therethrough and/or drying the cut beets and processing them during a period of 8-9 months. However, none of these proposals have given a solution which is acceptable in technical and commercial view.

ad.b.

it is established practice to cut the washed beets into small strips (cossettes) and to extract these strips with warm water. This stage of the process is critical for the sugar loss balance. It is common practice to carry out the extraction with warm water according to the counter current principle. The sugar present in dissolved form in the juice of the cells of the sugar beets can only leave the cells when the plasma with its semi-permeable membrane is denaturated or when the cell is opened mechanically. When the sugar beets are pulverized in some way not only the sugar present in the cells is dissolved during the extraction with warm water from the destroyed cells but also all kinds of undesired substances present in the cell are dissolved. Examples thereof are proteins, salts, gums and polymers. This presents at the further processing so many more problems than when the cossettes are subjected to an extraction that nowadays still only beet cuttings are used for the extraction. During the cutting of the beets only a number of cells are destroyed but the major part remains undamaged. For extracting the sugar from the undamaged cells the cells should be denaturated and opened at elevated temperature. This change in structure is also indicated as plasmolysis. Dependent on the used temperature several phenomena appear. At 60°C the protoplasma dies after contraction whereas the denaturation at suddenly quick heating at 70°C takes place so fast that the proteins for the main part remain in the original condition and are fixated as it were.

At the extraction first juice is washed out of the damaged cells. Thereby also many colloids dissolve. Because hydrate water is liberated during the denaturation of the beet proteins which drives out juice also a disengagement of juice from the interior of the cuttings takes place.

The major part of the substances especially sugar present in dissolved form in the cuttings diffuses from the coherent system of strongly branched channels which is available for the removal of the substances after the effected denaturation in the cell tissue of the beets. For that reason this way of extracting is indicated as "diffusion process". In the denaturated beet tissue a liquid exchange takes place according to which through the small pores osmotic water enters and through the great pores a sugar solution comes out by means of hydrostatic pressure. Thereby the small ions and molecules of other compounds than sugar move more quickly into the juice than the larger ions and molecules. The behaviour of the cations is also determined by the size of the anions. In respect to the electron neutrality of the anions these may only diffuse in such a way as the diffusion potential is higher than the electric potential effected by the separation of the charges. Slower diffunding larger anions retard for that reason for instance the quick transport per se of the ions of alkaline metals.

With respect to the plasmolyse it is desired to heat the cuttings as fast as possible at 75-80°C. Thereby it is prevented that micro-organismn cause sugar loss by conversion of the present sugar.

At those high temperatures the cellulose of the cell structure remains almost unchanged, but the pectins will swell and are even destroyed in the long run. When the pectin content of the juice, the extract, increases the processing of the juice becomes more difficult. Besides that because of the pectin conversion the high temperatures produce simultaneously a change of the structure of the cuttings. The squeezability of the cutting then becomes worser and the capillary system in the cell tissue of the cuttings is closed by the swelling process. The fine channels become smaller and more or less closed so that the extraction of the sugar is slowed down and greater diffusion losses are obtained.

Therefore the extraction is complicated and not optimal.

ad.c.

for separating the extract and the pulp as good as possible the pulp is squeezed at the end of the process but in respect to the under b) discussed conversions in the beet tissue which inhibit the extraction of the sugar still present therein no optimal separation could be achieved till now.

ad.d.

the water used for the extraction should be as pure as possible. In particular the water should not contain soluble substances which affect the purification of the juice. Especially water containing alkaline metal salts is disadvantageous because such salts inhibit the crystallization of large amounts of sugar and increase thereby the not contemplated molasses yield.

For obtaining an optimal crystallization of the sugar during the further processing the crude juice is heated at 85°C and mixed with an excess of slaked lime. The present free acids form therewith insoluble salts which with many other contaminations for instance proteins flocculate. A part of the lime forms calcium monosaccharate and another part dissolves. The slimy precipitate cannot be filtrated. For that reason carbon dioxide gas and steam are introduced so that the saccharate is decomposed and forms with the dissolved lime insoluble calcium carbonate which can be filtrated.

After twice saturating and filtrating a thin juice is obtained which contains about 15 % by weight of sugar. This juice is concentrated under vacuum in a multi-stage process until a saturated solution is obtained which crystallizes out to the so-called "masse cuite" containing 86 % of sugar. For obtaining a product with a sugar content of almost 100 percent the "masse cuite" should be separated from the mother lye by centrifugation after which the described operation is repeated several times with the mother lye until molasses as undesired by-product remains.

It was found that all above mentioned disadvantages respectively problems can be avoided respectively solved by mechanically reducing the disaccharide containing tuberous plants to a mush, extracting the reduced material with water of less than 60°C, subjecting it first to an ultrafiltration and then to a demineralisation and concentrating the obtained solution.

By reducing the tuberous plants mechanically to a mush the sugar present therein does not need to be extracted by diffusion as necessary with cuttings but sugar present in dissolved form in the cells of the tuberous plants can be rinsed out by simply washing with water of less than 60°C of the cells crushed by the mechanical treatment.

Because water of less than 60°C is used less non-sugar components are extracted than at the under b) described extraction with warm water. For that reason a number of the above discussed purification operations can be omitted.

The mechanical production of a mush may for instance be carried out by grating with a in the potato industry usual grating apparatus, a centrifugal pump with open blower or an oscillating mill, however, preferably with the mentioned grating apparatus.

After the reduction the beet juice and beet pulp are separated. This can be carried out more efficiently than with the known process because during the reduction of the tuberous plants almost all cells are opened and no denaturation of non-opened cells at elevated temperature has taken place. Suitable separation methods are:

a. use of a centri-sieve with turning basket, in which the cell juice can be washed out;
b. use of a vacuum band filter;
c. use of a scroll containing solid Bowl decanter.

Subsequently the obtained solution is subjected to an ultra-filtration whereby all colloidal and undissolved components remain in the retentate. The permeate contains more sodium- and potassium salts than thin juice which is obtained according to the classic method because a considerable part of the potassium- and sodium salts which end up in the molasses according to the classic process now comes in the permeate according to the process according to the invention. For removing these salts the permeate should be led over an ion exchanger.

Preferably the reduced mass is first separated for the most part from the liquid present therein and then subjected to an extraction with water of at most 10°C.

The contact time at the extraction is at least ½ hour but such contact time is seldom necessary because in fact the extraction is momentary at the first contact with water.

Further it is very important that the amount of water used for the extraction is chosen in such a way that the obtained sugar solution has a sugar content of 12-14 % by weight.

A further important advantage of the present process is that the ultrafiltrated ion-exchanged and concentrated permeate is so pure that for obtaining of almost pure beet sugar the crystallization does not need to be carried out in many stages and finally molasses is obtained as by-product. It is self-evident that the concentrated solution can be concentrated and/or cooled to saturation so that the sugar present therein crystallizes out but this operation is not necessary for the purification. Delivery of pure concentrated sugar syrup is also important for economic reasons because there are many technical uses for sugar which start with a concentrated solution.

Another advantage is that according to the present process no sugar-foreign substances should be added to effect specific reactions which substances should be removed again after the end of the reaction. The present process is namely carried out without the addition of any reaction component.

It has appeared that besides to the abovementioned advantages the present process has another important advantage, namely that so-called mangels instead of sugar beets can be used as starting material.

Since many decennia beet sugar is recovered from sugar beets with a sugar content of 16-19 % by weigth by extracting the sugar beets with water in the above described way, followed by roughly removing the pulp, treating the obtained product with lime and carbon dioxide for precipitating the pectins and albumins followed by removing the precipitate concentrating the obtained juice (thin juice) by evaporation, crystallizing sugar from the saturated solution and separating the crystallized sugar.

Up to now it was for economic and process-technological reasons not considered possible to use in the recovery of sugar mangels with a sugar content of only 6-12 % by weight as starting material. It was namely expected that at the processing of mangels much more pulp would be obtained than at the processing of sugar beets.

The roots of mangels contain in respect to the roots of sugar beets very much sodium, much potassium, little magnesium and little calcium. Therefore the ratio K + Na to Mg + Ca is much greater than for sugar beets. Detailed data thereabout are mentioned in "De onderscheiding van suikerbietenrassen op grond van minerale samenstelling van loof en wortel" by P.J.H. van Ginneken, in "Mededelingen van het Instituut voor Suikerbietenteelt", Bergen op Zoom, Holland, September 1940, and part IX - 1939 of the same institute.

As already mentioned in the above under "ad d", the presence of many sodium- and potassium ions in the sugar containing extract in the processes carried out up to now is a very detrimental aspect for the economy of the process.

Moreover high potassium contents promote the production of much molasses.

However, the advantages of the use of mangels would be:

1) instead of payment for the supplied beets on sugar content which is necessary and usual in the case of sugar beets the price of the supplied beets in the case of mangels as starting material might be determined by means of the weight. The yield of mangels per acre surface unit is namely much greater than the yield of sugar beets per acre surface unit so that variations in sugar content become less important;

2) by using mangels as starting material for the recovery of beet sugar the process in the sugar factory might better be stream-lined because the keeping qualities of mangels is at least 7 months whereas sugar beets only can be stored for 2 to 3 months. The "campagne" would be extended therethrough from instead of about 3 months to about 7 to 10 months.

In this respect it is important to give some illustration with respect to the differences between sugar beets and mangels.

Both species of beets originate from the beet Beta-vulgaris. From this original beet several species have been developed by mutations in the cause of decennia whereby the properties are directed to the final use. Thereby two aspects are more or less contrary namely the yield per surface unit and the sugar content.

Mangels or mangolds give the highest yield of beets per surface unit and also the highest yield of sugar per surface unit but the sugar content per beet is low so that much pulp will be obtained as

by-product. Mostly they are ripe very late so they have much foliage at the harvest time.

These beet species utilizes the water supply of the soil much better than other beet species. For that reason they are better resistent against long periods of dryness. With the same amount of water respectively nutritious substances they produce more dry material than sugar beets. However, for the recovery of sugar according to the known process they are not suitable because the amount of pulp which is obtained per weight unit of beets is considerably larger than when sugar beets are used as starting material and moreover the sugar content per weight unit of beets is much lower.

Sugar beets posess a high sugar content but give less beets per surface unit than mangels. The amount of foliage thereof is at the harvest time much less than that of the original Beta-vulgaris and the mangels because they are ripe early. For the recovery of sugar they are more suitable than mangels because per weight unit of beets less pulp and more sugar can be recovered.

EXAMPLE I

Washed mangels were reduced with a grater as used in the potato starch industry. The so obtained grating or mush was washed out with water of 18°C in a continuous multi-stage wash operation according to the counter current principle and then the pulp and the liquid were separated. This operation was carried out with a scroll containing solid-bowl decanter. Three of such decanters were used in series whereby from the last (3rd) decanter the desugarized and also up to 35 % by weight of dry material centrifugated pulp was removed. This last decanter was fed with an already partly desugarized pulp from the second stage decanter which was mixed with pure water of 10°C. The amount of water was minimal because the operation was carried out according to the counter current principle. That quantity can be determined by calculation with a tolerance of a certain sugar loss in the pulp when it is known that this pulp from the third stage is easily centrifugated to 35 % by weight of dry material.

The same operation was repeated three times. From the first stage decanter a cell juice was obtained which was used as feed for the ultrafiltration.

The first stage was fed with mush from the grater which was mixed with diluted cell juice from the second stage.

At the ultrafiltration a permeate and a retentate were obtained.

The retentate contained proteins, pectins and gums which for instance were mixed with the obtained desugarized pulp and sold as cattle-fodder.

The permeate was further purified by leading it over ion exchangers and treating it with decolorizing absorbentia.

The so obtained very pure sugar syrup is directly fit for human consumption.

EXAMPLE II

The mush from the grating apparatus of example I was desugarized and dewatered by means of a multi-stage counter-current washing in centrifugal sieves.

Subsequently the desugarized pulp was further squeezed for adjusting the water content and therefore the sugar loss on a economic level.

The so obtained somewhat diluted cell juice was processed in the same way as described in example I.

EXAMPLE III

1000 kg of mangels were reduced to a mush with a grater as used in the potato starch industry. This mush was directly separated into 490 kg of pulp and 900 kg of cell juice in a centrifuge. Thereby a washing with 390 kg of water of 9°C was carried out to desugarize the pulp as much as possible. The somewhat diluted cell juice was subjected to a combination of ultrafiltration. Before the diafiltration 90 kg of water of 9°C were added to dilute the 89 kg of retentate of the first ultrafiltration and to desugarize it for the most part.

The final retentate contained only 1% by weight of sugar. The so purified permeate or filtrate with a mass of 900 kg contained 9.7 % by weight of sugar (polarimetric) and 13.4 (w/w) % of dry material.

The content of invert sugar was 5.4 (w/w) % on the basis of the dry material.

This purified juice was then demineralized by means of subsequently a cation exchange resin and an anion exchange resin. This purified juice was almost colourless and contained only 5 (w/w) % non-sugar substances on the basis of the dry material content.

This juice was concentrated in an evaporator to 71 (w/w) % dry material content. The white sugar obtained by crystallization was of excellent quality and was not second to other granulated sugar.

EXAMPLE IV

450 kg of mangels were reduced to pieces with a greatest size of 8 cm. by means of a cutting apparatus. These beforehand reduced pieces were supplied by means of a screw pump to a homogenizer for grinding the beets to a mush.

This mush was then treated in the same way as described in example III.

EXAMPLE V

600 kg of mush or grating (vide example III and V) was desugarized in a quick turning sieve. The desugaration was carried out in four stages whereby the pulp mass was counter-current to the juice.

The pulp in the last stage was desugarized with 235 kg water of 9°C. The centrifugate of this fourth stage was used to desugarize the centrifugated pulp from the second stage. This was done in the third stage. The centrifugate was led to the centrifugated pulp from the first stage and the pulp from the third stage was led to the fourth stage.

The mixture of centrifugates, coming from the third stage and pulp of the first stage were dewatered in the second stage.

The juice from the second stage was joined with undiluted cell juice from the first stage. In this way a somewhat diluted cell juice (520 kg) was obtained, which was led to the ultrafiltration for the first purification with respect to proteins, gums and pectins.

The further processing was carried out as described in example IV. Also in this case a granulated sugar with a good quality as in the preceding examples was obtained.

CLAIMS

1. A process for the recovery of disaccharides from disaccharides containing tuberous plants according to which a solution of disaccharides in water is subjected first to ultrafiltration and then to ion exchange, characterized by mechanically reducing the tuberous plants to a mush, extracting the reduced material with water of less than 60°C, subjecting the extract first to an ultrafiltration and then to a demineralisation and concentrating the obtained solution.

2. The process according to claim 1, characterized by first removing the liquid present in the grated tuberous plants for the main part and then subjecting it to an extraction with water of at most 10°C.

3. The process according to claim 1 or 2, characterized by carrying out the extraction with water of at most 10°C and during a contact time of at most 0.5 hour.

4. The process according to any of the claims 1-3, characterized, by carrying out the extraction with such an amount of water that the obtained sugar solution has a sugar content of 12-14 % by weight.

5. The process according to any of the claims 1-4, characterized, by using mangels with a sugar content of 6-12 % by weight as tuberous plants.

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European Patent Office

EUROPEAN SEARCH REPORT

0126512
Application number

EP 84 20 0723

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
A	NL-C- 79 743 (VAN OSS) * Claims 1,4,5; column 1, lines 10-15; column 2, line 44 - column 3, line 11 *	1	C 13 D 1/10
A	GB-A-1 361 674 (DANSKE SUKKERFABRIKKER) * Claim 1; example 14 *	1	
A	US-A-2 382 407 (ERICKSON)		

TECHNICAL FIELDS SEARCHED (Int. Cl. ³)

C 13 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	24-08-1984	VAN MOER A.M.J.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
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