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MOLASSES PURIFICATION

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ABSTRACT OF THE DISCLOSURE

Liquid sugar is obtained from cane molasses by subjecting the molasses to two defeco-saturation treatments at different pH before they are passed into an ion exchanger.

This invention relates to the production of an only slightly colored liquid sugar of high purity from cane molasses by means of ion exchange resins.

So far, an economic commercial preparation of sugar from cane molasses has not been possible. The saccharate process cannot be applied to the processing of cane molasses because of their invert sugar content. Solvent processes can only precipitate the saccharose but not the invert sugar and have the drawback of high solvent cost.

Methods of removing the non-sugars by means of ion exchange resins have, due to the high proportion of the non-sugar substances in the molasses, the disadvantage of requiring large amounts of ion exchange material and regenerating agents. In addition, in the deionization of cane molasses, a sufficient decoloration of the deionized juice is difficult because the coloring agents which are believed to be formed by the reaction of the invert sugar with nitrogen compounds are bound only incompletely by the exchange resins. The aftertreatment with specific decoloration exchangers (e.g. strongly basic exchangers in Cl form) do not help much either. Such exchange resins have the additional drawback that their decoloring capacity decreases very soon because of the irreversible charge with high molecular substances.

Also the fats and waxes which are contained especially in cane molasses, interfere with the efficiency of the exchange resins. Therefore, ion exchangers have not been used in commerce for the purification of cane molasses.

It is also known that molasses contain a number of various coloring agents the constitution of which is not completely known. Part of such coloring agents is retained by ion exchangers; however, certain coloring agents which are found especially in cane molasses, are not bound by ion exchangers and impart an intense yellow color to the juices from which the salts have been removed by ion exchangers.

It is a principal object of the invention to provide a method which avoids the recited drawbacks and produces in an economic manner only slightly colored liquid sugar of high purity by refining cane molasses by means of ion exchangers.

Other objects and advantages will become apparent from a consideration of the specification and claims.

According to the invention, said objects are attained by applying to the cane molasses, before they are subjected to an ion exchanger purification, the following pretreatment: In a first step, the molasses is diluted to about 35 Bx. and the mud substances contained therein are removed, preferably by separation; in a second step, a defeco-saturation is carried out at a temperature up to 45° C., preferably below 40° C., with lime and carbon dioxide at a pH of 10–12, preferably about 10.5, with an amount of lime corresponding to 30 to 60 percent of CaO, calculated on the non-sugars present; the formed mud is filtered off, and finally the filtrate is brought, by a second defeco-saturation with lime and carbon dioxide, to a pH of 8–8.5, whereby 3 to 6, preferably 5% of lime, calculated on the non-sugars of the original molasses, are used and filtered again.

The invention is based on the discovery that it is possible to remove, prior to the treatment with ion exchange resins, a considerable amount of the non-sugars and the major part of coloring substances not removed by the ion exchangers, when the juice is subjected to a specific prepurification procedure.

In the following, the individual prepurification steps will be described in more detail.

The first step, the removal of the mud substances contained in the molasses by dilution of the molasses to about 35 Bx. and subsequent separation, filtration, or decantation can be carried out in apparatus known for this purpose, preferably in separators, and with advantage at room temperature.

In the subsequent defeco-saturation at a pH of 10–12, a difficulty has been due to the poor filterability of the mud. I have found that said poor filterability is caused by very fine mud substances contained in the molasses and I have discovered that the filterability of the defeco-saturation mud is very much improved when the fine mud substances contained in the molasses are removed in said first purification step.

The second step, i.e. the defeco- saturation with lime and carbon dioxide at a pH of 10–12, preferably 10.5, is known in the purification of sugar juices. However, in the instant case, there is a solution which, in contrast to juices normally subjected to defeco-saturation, has a content of 10 to 15 percent of non-sugars at about 35 Bx. Dependent on the amount of non-sugars and on the degree of coloring of the molasses, the removal of the coloring ingredients not absorbed by the ion exchangers requires lime in an amount of 30 to 60 percent of the non-sugars contained in the molasses, in other words, calculated on the solution, my purification requires about 10 times the amount employed in the conventional raw juice purification.

During the defeco-saturation and the subsequent filtration of the mud in the usual manner by means of rotary filters, filter presses, and the like, a temperature adjustment to a bout 45° C., preferably below 40° C., has the result that a decomposition of the invert sugar in the alkaline range, in which new coloring substances and water soluble calcium salts are formed, is substantially avoided.

The filtration of the mud prior to the subsequent treatment is necessary because otherwise the coloring substances absorbed by the mud dissolve again when the juice is saturated down to a pH of 8–8.5.

Said just mentioned down saturation to a pH of 8–8.5, subsequently to the mud filtration, is carried out in form of a defeco-saturation with lime and carbon dioxide, whereby the temperature referred to above of below 45° C., preferably below 40° C. is maintained and the amount of lime used is adjusted to about 3–6, preferably 5 percent of the non-sugars of the molasses.

The following filtration of the calcium carbonate mud is carried out again in the conventional manner over rotary filters or the like whereby the sweet filtrate from the filter to about 10 Bx. is combined with the filtrate proper.

The subsequent treatment of the juice consists in a treatment with cation-anion exchangers, as described for instance, in the journal "Zucker," vol. 15 (1962), pp. 340–345.

Then, the deionized juice is concentrated to about 75 Bx., e.g., by means of flash boilers. The sugar containing

waste waters of the muds below about 10 Bx. and those of the ion exchange plant, as far as not incorporated in the purified juice, are used to dilute the molasses.

If the defeco-saturation of the pH of 10–12 is carried out with sulfur dioxide instead with carbon dioxide, the adsorption of coloring substances and non-sugar components is considerably lower and the mud, due to its extremely fine structure, is very difficult to filter, even though the diluted molasses may have been previously separated.

A single defeco-saturation at a pH below 10 does not retain sufficiently those coloring substances which are difficulty removed in the ion exchangers.

In the method of the invention, the degree of the non-sugar removal and juice decoloration can be widely varied during the pre-purification by the amount of the lime added.

Even from very poor and strongly aged molasses, liquid sugar of a purity up to 99% total sugar/TS and a color of 3–5° St. are obtained. Such products, known on the market as "Golden syrup," are used in the food and beverage industry.

The particular advantages of the process of the invention over the known methods are as follows:

Relief of the ion exchange plant required for complete deionization by the pre-purification steps where up to 50 percent of the non-sugars and the major part of those coloring substances are removed which are not retained by the ion exchangers.

Considerable reduction of the investment cost for an ion exchange plant which, without the prepurification plant of the invention, would require double size.

Considerable reduction of the operative cost because the regenerating agents for the ion exchange plant cost four to five times as much as the corresponding amounts of lime and carbon dioxide.

The solutions fed into the ion exchangers are completely free of colloids, fats, and waxes, thereby preventing capacity losses of the ion exchange plant by irreversible loads.

Possibility of economical utilization of the sugar contained in cane molasses.

The following example is given for the sake of illustration and without intention of limiting the invention to the specific details disclosed.

EXAMPLE

Strongly aged cane molasses are diluted with water to 35° Bx., whereby the solution contains 47.4% of non-sugars/TS and has a color of 3.830° St.

The solution is separated in a mud separator at 20° C. After removal of the mud, the solution has still 44.4% non-sugars/TS and a color of 3.710° St. Therefore, the removed amount of non-sugar is 12 percent.

The solution is defeco-saturated with 50% CaO, calculated on the non-sugars of the starting molasses at a pH of 10.7 and a temperature of 25–32° C., and with CO_2 and the mud is filtered off.

The filtrate contains 35% of non-sugars/TS and has a color of 320° St. The removed amount of non-sugars corresponds to 40 percent of the original content.

The filtrate is defeco-saturated with 3% of CaO, calculated on non-sugars of the starting molasses, and CO_2; thereby, the pH value gradually drops to 8.5. The filtrate of the mud contains 32.5 percent of non-sugars/TS and has a color of 330° St.

47 percent of the original non-sugars and 91 percent of the color have been removed.

The thus obtained juice is desalted in cation-anion exchangers, concentrated in vacuo at 70 to 80° C. to 75° Bx. and produces a liquid sugar of 98.9 percent total sugar purity and a color of 7.4° St.

We claim:

1. A process of preparing liquid sugar of high purity and low color from cane molasses comprising diluting cane molasses to 35° Brix, removing mud substances, then subjecting the diluted molasses to a defeco-saturation at a temperature not higher than 45° C. with lime and carbon dioxide at a pH of 10 to 12, the amount of lime being 30 to 60 percent calculated on the non-sugars of the molasses, filtering off the mud formed in the defeco-saturation, subjecting the filtrate again to a defeco-saturation with lime and carbon dioxide at a pH of about 8 to 8.5, the amount of lime being about 3 to 6 percent, calculated on the weight of the non-sugars of the original molasses, filtering, and passing the filtered juice into an anion-cation exchanger.

References Cited

UNITED STATES PATENTS

1,727 738	9/1929	Van Nes	127—52 X
2,075,127	3/1937	Mead	127—48
2,093,759	9/1937	Hartmann	127—48 X
2,697,049	12/1954	Brieghel-Muller	127—52 X
2,774,693	12/1956	Brieghel-Muller	127—50
2,868,677	1/1959	Kopke	127—46
2,916,404	12/1959	Battistoni	127—48 X
3,257,236	6/1966	Schwer	127—55 X

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