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EXTRACTION OF SUGAR FROM SUGAR-CONTAINING LIQUIDS

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8 Claims. (Cl. 127—47)

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This invention relates to a process for the extraction of sugar from molasses and other sugar containing liquids.

It is known, for this purpose, to treat the molasses with barium hydroxide under heat, generally between 85° and 100° C., in order to precipitate barium saccharate, which is decomposed by carbon dioxide into barium carbonate and sugar solution. The methods hitherto proposed, however, have had little success in practice because the barium saccharate is incompletely separated out, and above all in a form in which the precipitate can only be separated from the residual solution with great difficulty.

The principal object of the present invention is to provide a process for recovering sugar from molasses or similar sugar containing liquids while using barium hydroxide as precipitating agent which process enables to extract the sugar present in such solutions in a higher percentage than heretofore. Another object of the invention is to provide a method by which the sugar present in molasses or other sugar containing liquids is precipitated in the form of a crystalline mass of barium saccharate, which can be readily filtered off from the waste liquor.

Other objects of the invention will appear as the description proceeds.

The process according to the invention consists substantially in adding barium hydroxide to the sugar containing liquids from which the sugar is to be extracted, while adjusting the concentration of barium ions so as to lie above the product of solubility of mono-barium saccharate, initiating the crystallization of mono-barium saccharate by grafting with crystals of mono-barium saccharate and maintaining the supersaturation of the solution with barium ions by the addition of barium hydroxide during the crystallization according to the quantity of barium ions consumed in the formation of mono-barium saccharate.

The grafting with crystals of mono-barium saccharate, which is of essential importance to the process according to the invention, is preferably effected with crystals of mono-barium saccharate which has been separately produced from pure sugar and pure barium hydroxide.

The process according to the invention may, for example, be carried out in the following manner:

Hot barium hydroxide solution is added to the slightly dilute molasses with simultaneous cooling. The temperature of the mixture should advantageously be below 50° C. and is preferably

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so controlled that it is at about 20° C. The quantity of barium hydroxide solution added is such, having regard to the sugar content of the molasses and the concentration of the barium hydroxide solution, that the concentration of barium ions at the working temperature lies above the product of solubility of the mono-barium saccharate, that is to say, a super-saturated solution in respect of barium ions is obtained. The super-saturated molasses are then grafted with crystals of mono-barium saccharate, which are preferably cultivated from pure sugar and barium hydroxide. The quantity of grafting material is such that sufficient crystal surface is available for the crystallization set up and that no uncontrolled formation of new crystals occurs under the conditions of crystallization produced, but that the crystals merely grow. When the crystallization has started, barium hydroxide solution is added, according to the quantity of barium ions disappearing owing to the formation of mono-barium saccharate, in such a quantity in each case that there is always a slight supersaturation with barium ions, so that a controlled progress of the crystallization is ensured. In order to check and control this process, the pH-value of the solution, which must lie in the alkaline zone, is constantly observed during the crystallization. The addition of barium hydroxide therefore takes place in such a manner that the pH-value is kept substantially constant until near the end of the crystallization process. The end of the crystallization is marked by a sudden increase in the pH-value owing to the cessation of the formation of saccharate. At this moment, the addition of barium hydroxide is discontinued. The precipitate of mono-barium saccharate formed is filtered off from the residual solution, for example by the aid of filter presses, and is subsequently washed with a dilute barium hydroxide solution, for example with a cold-saturated barium hydroxide solution or a hot 2-3% barium hydroxide solution. The washing liquid is taken up separately and can be used to dilute the molasses in the following operation.

The extraction of the sugar may be carried to any desired degree by suitable selection of the final concentration of barium ions. Up to 97.2%, referred to the sugar present, can rationally be extracted at the maximum. However, it is preferable to operate in such a manner that raffinose present, which has a tendency to precipitate at the same time at high barium ion concentrations, remains in solution. If in some particular case

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the waste liquor still contains sugar, this sugar can be precipitated as bi-barium saccharate by adding $\text{Ba}(\text{OH})_2$ and heating to temperatures above 60°C ., this bi-barium saccharate being filtered off and employed in the next operation.

The mono-barium saccharate yielded as a filter cake is decomposed into sugar solution and barium carbonate after being worked up into a paste with pure water at boiling temperature with CO_2 or waste gases containing CO_2 . The slight residue of barium ions is precipitated by the addition of dilute sulphuric acid. In order also to remove the last traces of barium hydroxide which cannot be precipitated with sulphuric acid owing to the danger of exceeding the neutral point, $(\text{NH}_4)_2\text{SO}_4$ is added until the barium reaction ceases. The pure sugar solution yielded in the decomposition may if necessary be treated with active charcoal, filtered off and evaporated. The barium carbonate formed in the carbonation may also be worked up again into barium hydroxide by known methods, for example by decomposing it with hydrochloric acid with the aid of anti-foaming agents and employing the carbon dioxide formed for the carbonation. The barium chloride yielded can be converted into $\text{Ba}(\text{OH})_2$ by electrolyses and returned into the process.

The excess barium hydroxide can also be precipitated by CO_2 from the waste liquor from which the sugar has been extracted, whereafter the waste liquor, now free from barium, can be worked up into chemicals after evaporation.

I claim:

1. A process for the extraction of sugar from sugar containing liquids comprising the steps of adding barium-hydroxide to the sugar containing liquid while adjusting the concentration of barium ions so as to lie slightly above the product of solubility of mono-barium saccharate, grafting the said liquid supersaturated in respect of barium ions with crystals of mono-barium saccharate, adding barium hydroxide during the crystallization according to the quantity of barium ions consumed by the formation of mono-barium saccharate to maintain the supersaturation of the liquid with barium ions during the crystallization, said crystallization being effected at a temperature between 50°C . and 20°C ., separating the crystalline barium saccharate precipitated from the waste liquor, washing and carbonating it.

2. A process for the recovery of sugar from molasses comprising cultivating crystals of mono-barium saccharate from pure solutions of barium hydroxide and of sugar, grafting molasses supersaturated with respect to barium ions with said crystals, maintaining the concentration of barium ions in said solution until no further formation of mono-barium saccharate occurs, maintaining the temperature of the said grafted solution between 50°C . and 20°C ., separating the precipitated mono-barium saccharate and carbonating it.

3. A process for the extraction of sugar from a sugar containing liquid comprising adding barium-hydroxide to said liquid while continuously adjusting the concentration of barium ions to a point slightly above the product of solubility of mono-barium saccharate, grafting the said liquid supersaturated with barium ions with crystals of mono-barium saccharate to control crystallization of precipitated mono-barium saccharate, continuing the said adjustment of barium ions by the addition of barium-hydroxide

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during the said crystallization, said crystallization being effected at a temperature below 50°C ., separating the precipitated crystalline barium saccharate, washing and carbonating it.

4. In a process for the extraction of sugar from sugar containing liquids adding barium-hydroxide to the sugar containing liquid while adjusting the concentration of barium ions so as to lie slightly above the product of solubility of mono-barium saccharate, grafting the said liquid supersaturated in respect of barium ions with crystals of mono-barium saccharate, adding barium hydroxide during the crystallization according to the quantity of barium ions consumed by the formation of mono-barium saccharate to maintain the supersaturation of the liquid with barium ions during the crystallization, said crystallization of mono-barium saccharate being effected between 50°C . and 20°C ., separating the crystalline barium saccharate precipitated from the waste liquor, washing and carbonating it.

5. A process for the extraction of sugar from a sugar containing liquid comprising adding barium hydroxide to said liquid in amount necessary to adjust the concentration of barium ions to a point slightly above the product of solubility of mono-barium saccharate, determining the pH value of said liquid, grafting said liquid with crystals of mono-barium saccharate to control crystallization of precipitated mono-barium saccharate, adding barium hydroxide during said crystallization to maintain said determined pH value, discontinuing the addition of barium hydroxide upon sudden substantial increase in said pH value effecting said crystallization between 50°C . and 20°C ., separating precipitated crystalline mono-barium saccharate, washing and carbonating it.

6. A process for the extraction of sugar from a sugar containing liquid comprising adding barium hydroxide to said liquid in amount necessary to adjust the concentration of barium ions to a point slightly above the product of solubility of mono-barium saccharate, determining the pH value of said liquid, grafting said liquid with crystals of mono-barium saccharate to control crystallization of precipitated mono-barium saccharate, said crystallization being effected at a temperature below 50°C ., adding barium hydroxide during said crystallization to maintain said determined pH value, discontinuing the addition of barium hydroxide upon sudden substantial increase in said pH value, separating precipitated crystalline mono-barium saccharate, washing and carbonating it.

7. A process for the recovery of sugar from molasses comprising cultivating crystals of barium mono-saccharate from pure solutions of barium hydroxide and of sugar, grafting molasses supersaturated in respect of barium ions with said crystals maintaining the concentration of barium ions in said solution until no further formation of barium saccharate occurs, said crystallization of mono-barium saccharate being effected between 50°C . and 20°C ., separating the precipitated barium saccharate and carbonating it.

8. A process for the extraction of sugar from a molasses solution comprising the addition of barium hydroxide to said solution in amount necessary to maintain the barium ion concentration above the product of solubility of mono-barium saccharate, grafting said solution with crystals of mono-barium saccharate formed from pure solution of barium hydroxide and sugar to control crystallization of precipitated mono-barium saccharate, maintaining said barium ion

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concentration by further addition of barium hydroxide until no further precipitation occurs, effecting said crystallization between 50° C. and 20° C., separating said precipitated mono-barium saccharate and carbonating it.

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