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(54) Titre : METHODE DE RAFFINAGE DU SUCRE BRUT, EN PARTICULIER DU SUCRE BRUT PRODUIT PAR
L'INDUSTRIE DE LA CANNE A SUCRE

(54) Title: PROCESS FOR REFINING A RAW SUGAR, PARTICULARLY RAW SUGAR FROM THE SUGAR CANE
INDUSTRY

(57) **Abrégé/Abstract:**

A process for refining a raw sugar, particularly raw sugar from the sugar cane sugar industry, characterized in that it comprises the steps of : (a) remelting of the raw sugar for obtaining a raw sugar syrup, (b) carbonatation or phosphatation of said raw sugar syrup, and (c) tangential microfiltration and/or tangential ultrafiltration of the raw sugar syrup, which has been subjected to carbonatation or phosphatation. The process is completed by the steps of : (d) decolorization of the sugar syrup resulting from step (c), and (e) crystallization and/or demineralization of the sugar syrup resulting from step (d).



ABSTRACT OF THE DISCLOSURE

A process for refining a raw sugar, particularly raw sugar from the sugar cane sugar industry, characterized in that it comprises the steps of :

(a) remelting of the raw sugar for obtaining a raw sugar syrup,

(b) carbonatation or phosphatation of said raw sugar syrup, and

(c) tangential microfiltration and/or tangential ultrafiltration of the raw sugar syrup, which has been subjected to carbonatation or phosphatation.

The process is completed by the steps of :

(d) decolorization of the sugar syrup resulting from step (c), and

(e) crystallization and/or demineralization of the sugar syrup resulting from step (d).

production of low-grade products (raw sugar and colored molasses) at the end of said run-off purification.

5 The invention seeks to remedy the disadvantages of the processes known previously, and it therefore proposes a process for refining a raw sugar, characterized in that it comprises the steps of :

 (a) remelting of the raw sugar for obtaining
10 a raw sugar syrup,

 (b) carbonatation or phosphatation of said raw sugar syrup, and

 (c) tangential microfiltration and/or tangential ultrafiltration of the raw sugar syrup, which has been
15 subjected to carbonatation or phosphatation.

The process of the present invention is intended particularly to raw sugar from the sugar cane sugar industry.

As will be observed, this process is free of any
20 tedious and costly preliminary step of raw sugar refining; this was made possible quite unexpectedly, according to the invention, by the use of the well known tangential microfiltration and/or tangential ultrafiltration technique, which is a simple, flexible,
25 high-efficiency, quick and well controlled technique having a much lower operating cost than the above-mentioned refining operation. In other words, the present invention makes it possible to dispense with a tedious and costly operation by using a simple operation, which
30 is little time and energy consuming.

It should be noted that the process according to the invention may further comprise a flocculation step making use of a flocculating agent, which step may be performed simultaneously with step (b) or before or after
35 said step (b), the use of such a flocculation step permitting a very appreciable reduction in the amounts of reagents (calcium oxide or lime milk, carbon dioxide and phosphoric acid) to be used in the carbonatation or

2a

phosphatation step.

According to a preferred embodiment of the
5 invention, the process comprises a flocculation step and
a phosphatation step, in which case the calcium oxide (or
lime milk), phosphoric acid and flocculating agent are
used in the phosphatation and flocculation steps in an
amount of 200 to 900 ppm (expressed in CaO), 200-900 ppm
10 (expressed in pure H₃PO₄) and 200-900 ppm (expressed in
active product) with respect to the dry matter of the raw
sugar syrup, respectively.

It will be noted that the flocculating agent used
in the flocculation step may particularly be formed by a

cationic surfactant, especially a quaternary ammonium compound of tallow fatty acids, for example dioctadecyldimethylammonium chloride such as NORAMIUM® M2SH marketed by the French company CECA. It may also be derivatives from deacetylated chitosan poly-N-acetylglucosamine derived from chitin, such as PROFLOC® SD 340 from the Norwegian company PROTAN BIOPOLYMER.

It will be further noted that the steps of carbonatation or phosphatation, flocculation, and tangential microfiltration and/or tangential ultrafiltration will be advantageously implemented at a temperature of the order of 70 to 95°C.

Finally, it should be specified that the refining process according to the invention will be completed by a step (d) of decolorization of the sugar syrup resulting from the microfiltration and/or ultrafiltration, and by a step (e) of crystallization and/or demineralization of the sugar syrup resulting from the decolorization step for thus obtaining white crystal sugar in the case of crystallization and liquid sugar (syrup) in the case of demineralization.

Other aspects and advantages of the present invention will become apparent from the following description of a preferred embodiment with reference to the accompanying drawing, the single figure of which is a schematic representation of an installation for implementing the inventive process.

In this embodiment, the raw sugar to be refined is a raw sugar from a sugar cane sugar factory, said raw sugar having a dry matter (essentially saccharose) content of the order of 97 to 99% by weight.

After possible screening of the raw sugar to be refined, the latter is remelted, i.e. dissolved in an aqueous medium, such as a sugar aqueous solution or preferably water. The aqueous medium is at a sufficient temperature for the remelting operation to take place at a temperature of the order of 50 to 90°C, preferably of the order of 80°C.

Remelting results in the production of a raw sugar syrup, the amount of the aqueous medium used being preferably selected so that said syrup has a dry matter content of the order of 40 to 70% by weight. This remelting step is carried out in a tank 1 provided at the top thereof with a raw sugar supply 2 and a hot water supply 3. For obtaining an homogeneous syrup, said tank is further provided with agitator means 4.

The raw sugar syrup resulting from the above remelting step and which usually has a coloration of 2,500-4,500 ICUMSA units (international units) and a turbidity of 3,000-4,000 ICUMSA units is then subjected to a phosphatation step in conjunction with a flocculation step. To this effect, the syrup is extracted from the base of tank 1 through a conduit 5 connected to the intake of a circulating pump 6 having its delivery continued by a conduit 7, which opens to the upper part of a phosphatation/flocculation tank 8. If necessary, the syrup is reheated, for example through indirect heat exchange in a heat exchanger 9 arranged in the path of the syrup between tank 1 and tank 8. Said heat exchanger is selected to raise the temperature of the syrup to a sufficient value so that the temperature prevailing within tank 8 is of the order of 70°C.

A flocculating agent is then introduced into tank 8. More specifically, said flocculating agent is fed to the upper part of tank 8 from a flocculating agent tank 10, which is formed in its base with an extraction conduit 11 connected to the intake of a circulating pump 12 having its delivery connected to a conduit 13 leading to tank 8. Tank 10 may be provided with heating means (not shown), such as an inner jacket where a hot fluid, for example hot water or steam, is circulating, said heating means allowing, in case when the flocculating agent is solid or pasty at ambient temperature, to turn it into the liquid form for pumping by pump 12. Thus, for example, if said flocculating agent is formed by NORAMIUM® M2SH having a melting point of the order of 60°C, the heating means are arranged to raise the temperature of

the flocculating agent to a value of the order of 60-65°C. Besides, the amount of flocculating agent used is of the order of 200 to 900 ppm of active product with respect to the dry matter of the raw sugar syrup; for example, in the case of NORAMIUM® M2SH, said amount will preferably be of the order of 500 ppm of active product with respect to the dry matter of the syrup.

Subsequently, lime milk (at a concentration of 200g/l expressed in CaO) in an amount of the order of 300 ppm expressed in CaO with respect to the dry matter of the syrup is introduced into tank 8, followed by phosphoric acid (for example, a 90% solution) until neutralization, that is approximately 250 ppm expressed in pure phosphoric acid and with respect to the dry matter of the syrup. The introduction of the lime milk and phosphoric acid into tank 8 is achieved in the same manner as for the flocculating agent, i.e. from a lime milk tank 14 and phosphoric acid tank 15, respectively, through extraction conduits 16, 17, circulating pumps 18, 19 and conduits 20, 21 connected to said pumps and leading to tank 8.

It should be added that tank 8 is provided with agitator means for performing a vigorous stirring of its contents. It should also be specified that the flocculating agent may, as an alternative, be introduced into tank 8 simultaneously with or after the lime milk and phosphoric acid, although the operating mode described above is more preferred. Under the effect of the flocculating agent, a part of the coloring agents and solid matters in suspension precipitates. A major part of the remaining coloring agents and solid matters in suspension is eliminated by the phosphatation step, as the calcium phosphate formed by the reaction between the lime milk and phosphoric acid precipitates with an occlusion of said coloring agents and matters in suspension.

After a retention time of 10 to 30 min, preferably 15 min, in tank 8, the syrup, which has been subjected to the flocculation/phosphatation is drawn off from said tank 8 by

a pump 22 delivering into a tangential microfiltration or tangential ultrafiltration unit 23. If necessary, the thus drawn off syrup may be reheated so that the step in said unit 23 takes place at about 80°C. The membrane used in unit 23 may have a cut-off value of 10 nm to 1 μm (preferably of the order of 0,1 μm), the syrup flowing at a tangential velocity of the order of 1 to 8 m/s (preferably 4 to 6 m/s) and the permeate flow rate being of the order of 20 to 80 liters/hour.m² of membrane (preferably 35-50 liters/hour.m² of membrane). For the membrane, use may be made of membranes made of organic polymers or ceramic materials. Good results have been obtained with a membrane of zirconium oxide on a ceramic support, produced by the French company TECH-SEP.

The permeate from unit 23 (coloration of the order of 1,000-2,000 ICUMSA units and turbidity below 20 ICUMSA units) is conveyed through a conduit 24 to a storage tank 25, from which it is drawn off by a pump 26 to be fed to the head of a two-stage decolorization column 27, 28. Such columns are packed with a coloring agent absorbing material, such as animal black, active charcoal, or preferably with a decolorization resin; it may be a strong anionic resin in the chloride form (for example, resin IRA® 900 from Rohm and Haas). The decolorizing step in this column is preferably performed at 70-90°C, particulaly at 80°C. At the outlet of decolorizing stage 28, the decolorized syrup (coloration < 400 ICUMSA units) is fed into tank 29.

Said decolorized syrup may then be changed into either crystal sugar or demineralized sugar. In the former case, the sirup is fed through a pump 30 into an evaporator 31, such as a falling float evaporator, and the concentrated syrup is then fed by a pump 32 into a crystallisation unit 33 where it undergoes several successive crystallizations (three in the example illustrated in the figure) for delivering crystal sugar and a crystallization run-off. In the latter case, the decolorized syrup is fed, through a pump 34, to tank 29 in the demineralization unit 35, which may for example be formed by a column packed with a mixture of a cationic resin in the

H⁺ form and an anionic resin in the OH⁻ form. This resin mixed bed column may be replaced by two columns of which one is filled with a cationic resin in the H⁺ form and the other is filled with an anionic resin in the OH⁻ form. After the demineralization treatment, a syrup having a coloration < 20 ICUMSA units is obtained.

By way of example, it will be indicated that processing under the above-mentioned conditions of a remelt syrup having a dry matter content of 50%, a coloration of 3,800 ICUMSA units and a turbidity of 2,000 ICUMSA units, results in a syrup having a coloration of 1,500 ICUMSA units and a turbidity < 20 ICUMSA units at the outlet of the tangential microfiltration/tangential ultrafiltration unit, a syrup having a coloration below 400 ICUMSA units at the outlet of the decolorization unit and a crystal sugar or syrup having a coloration < 20 ICUMSA units at the outlet of the crystallization or demineralization unit. This shows that the process object of the present invention provides refining performance as high as that of the conventional refining technique, but with a highly simplified implementation.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A process for refining a raw sugar, characterized in that it comprises the steps of

(a) remelting of the raw sugar for obtaining a raw sugar syrup,

(b) carbonatation or phosphatation of said raw sugar syrup, and

(c) tangential microfiltration and/or tangential ultrafiltration of the raw sugar syrup, which has been subjected to carbonatation or phosphatation.

2. The process according to claim 1, characterized in that it further comprises a step of flocculation making use of a flocculating agent, said step being performed simultaneously with step (b) or before or after said step (b).

3. The process according to claim 2, characterized in that the flocculating agent is a quaternary ammonium compound of tallow fatty acids.

4. The process according to claim 2 characterized in that the flocculating agent is a deacetylated chitosan poly-N-acetylglucosamine derived from chitin.

5. The process according to claim 2, wherein step (b) is a phosphatation, characterized in that the calcium oxide or lime milk, phosphoric acid and

flocculating agent are used in the phosphatation and flocculation steps in an amount of 200 to 900 ppm (expressed in CaO), 200 to 900 ppm (expressed in pure H₃PO₄) and 200 to 900 ppm (expressed in active product) in
5 relation to the dry matter of the raw sugar syrup, respectively.

6. The process according to any one of claims 1 to 4, characterized in that the carbonation or
10 phosphatation and tangential microfiltration and/or tangential ultrafiltration steps are implemented at a temperature of 70 to 95°C.

7. The process according to any one of claims 2
15 to 5, characterized in that the flocculation step is implemented at a temperature of 70 to 95°C.

8. The process according to claim 5, characterized in that the phosphatation step is
20 implemented at a temperature of 70 to 95°C.

9. The process according to claim 1 or 2, characterized in that it further comprises the steps of
(d) decolorization of the sugar syrup
25 resulting from step (c), and
(e) crystallization and/or demineralization of the sugar syrup resulting from step (d).

10. The process according to any one of claims 1
30 to 9, wherein the raw sugar is from the cane sugar industry.

