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REFINING OF SUGAR

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This invention relates to sugar refining and is particularly concerned with a novel method for separating impurities from sucrose solutions.

Although the invention is applicable to any one of a number of the customary refining stages, the invention is of especial advantage when applied to certain particular solutions, for instance the so-called affination syrup from which it has heretofore been very difficult to recover sugar.

The principal object of the invention is the provision of a method, involving the use of a new and improved refining agent, by which the separation of impurities is greatly facilitated. The improvement in separation brought about by the invention is achieved by causing agglomeration of the impurities in the form of particles of increased size, as compared with prior processes, thereby greatly facilitating separation of the impurities by filtration or centrifuging.

Before describing the invention in greater detail, brief reference is here made to the principal steps customarily followed in the refining of sugar.

The initial steps of extracting the juices from the sugar cane are usually performed at the sugar plantation, the initial steps yielding sugar crystals still retaining substantial quantities of impurities including principally organic acids, sand, clay, gums, pectins, glucose, and glucose-lime compounds. Customarily the sugar is shipped in this form from the region of the plantation to a refinery, the shipment usually being made in bags, as a result of which fibers are introduced, in addition to the other impurities such as those already mentioned.

At the refinery the impure sugar crystals are customarily placed in a centrifuge with a small amount of water, so as to cause them to ride onto the surface of the centrifuge basket, and the crystals are at this time subjected to steam washing. This operation separates considerable quantities of the impurities which are carried away in the effluent from the centrifuge. This effluent, known as the affination syrup, has a pH value of from 6 to 6.8 and must be neutralized in order to prevent inversion. Lime slurry has customarily been employed for this purpose, the lime being added in an amount sufficient to raise the pH to 7 or somewhat above; and the syrup is then filtered. The filtered syrup is concentrated under vacuum

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and centrifuged to recover all possible sugar crystals therefrom, but even such centrifuging does not achieve economical recovery, the final effluent still containing anywhere up to about 65% of sugar. This final effluent (affination syrup) contains the various impurities in greatly concentrated form, as a result of which it has been very difficult heretofore to recover the sugar therefrom.

The steam washed and centrifuged crystals are further treated by adding water and lime slurry and heat is applied so as to effect reaction between the lime and impurities, such as remaining organic acids. Finally, the syrup is filtered to separate the impurities and the lime, and is passed through an adsorbent bed, suitably containing bone char.

As above indicated, the invention is applicable to various refining stages, and is particularly concerned with any of those stages in which lime is employed as a refining or neutralizing agent. An especially advantageous application of the method of the invention, however, is the treatment of the affination syrup, the invention making possible easy filtration for recovery of the sugar from the affination syrup, notwithstanding the high concentration of impurities therein.

According to the invention, a refining agent is used, comprising particles of lime mechanically or physically attached to particles of an inert material such as various clays, for instance bentonite and kaolin. In the preferred practice of the invention, the improved refining agent is prepared by fusing the lime and clay together in finely powdered and intimately admixed state. For this purpose the mixed lime and clay powders are heated to a temperature between about 2400° F. and 2900° F., for instance at about 2600° F. It is of importance in this heating to control the temperature so as to avoid formation of a vitreous or glass-like mass. The temperature and time of heating is desirably such as to yield a somewhat porous and frangible clinker. The clinkers so formed are then pulverized advantageously to a fineness such that 85 to 90% of the particles will pass through a 200 mesh sieve.

The effect of the fusion at high temperature is to convert the lime or calcium carbonate to calcium oxide, and, upon pulverizing the clinkers, the resultant powder comprises many particles

which are made up of both calcium oxide and clay which are physically or mechanically attached or bonded to each other.

Instead of employing clays such as bentonite or kaolin and mixing them with lime, certain of the clays found in low grade marl may be employed. This material contains not only an inert clay but also lime and the two constituents may therefore be derived from a single source.

It is of importance in preparing the improved refining agent of this invention to select ingredients as free as possible from magnesium carbonate.

The agent may conveniently be made up by employing powdered limestone and clay. The proportion of limestone to clay may be varied according to the particular circumstances in which the refining agent is to be used. For most purposes an admixture containing from about 250 parts to about 350 parts of limestone for each 100 parts of clay. The figures just given represent parts of the ingredients by weight when dry. After the fusion at high temperature there remains in the material a quantity of calcium oxide equal to about 56% of the weight of limestone originally used. Stating the preferred range in percentage figures, it may be said that in the fused agent the calcium oxide should comprise from about 58% to about 66% of the weight of the agent.

In adding the refining agent to any sucrose solution to be purified, it is advantageous to initially determine the acidity of the particular solution being treated and then add a quantity of the agent sufficient to bring into the solution enough lime to insure complete neutralization of all acids present. Advantageously the quantity of lime introduced in this way should be sufficient to yield a solution having a pH value of at least 7 and preferably between about 7.3 and 7.6. This is of importance in providing against inversion of the sucrose to glucose during heating of the solution.

At any of the stages of refining where the improved refining agent of this invention is employed, the sucrose solution, with the agent dispersed therein is heated, preferably in the range extending from about 175° F. to 205° F., most advantageously in the neighborhood of 190° F. The effect of the heating is to cause various of the impurities, for instance organic acids and any glucose present, to react with the lime, and because of the initial mechanical attachment of the lime to the particles of inert clay, the reaction products so formed also adhere to the clay particles. This apparently results in the building up of larger agglomerates than occurs in prior practice where lime or other refining agents are used alone. In consequence the impurities are much more readily removed by filtration or centrifuging.

Because of the foregoing improvement, it is even possible to treat the affination syrup with the improved refining agent of this invention and to thereby recover a high percentage of the sucrose present in the affination syrup. Heretofore, attempts have been made to recover the sugar from the affination syrup, but all efforts along this line have led to the employment of more or less complicated technique, requiring substantial equipment, all of which are not needed when employing the refining agent of this invention. Moreover, processes heretofore applied to recover sugar from syrup containing impurities in concentrated form, frequently required the employment of temperatures so high as to result in at least some substantial inversion of sucrose to glucose.

Still another advantage is secured by the invention in refining steps where phosphoric acid has been used heretofore. In certain processes, phosphoric acid is added at the time of adding lime, but when lime is used alone, very fine particles of tri-calcium phosphate are formed and these are very difficult to filter out or separate by centrifuging. Removal of such particles of tri-calcium phosphate was therefore effected in other ways, for instance by subjecting the syrup to the action of steam jets in trays of large surface area, over which scrapers were operated to collect and remove the impurities from the surface of the solution.

Use of the refining agent of this invention in instances where phosphoric acid is employed, is highly effective since the reaction products formed by reaction of the phosphoric acid and the lime also adhere to the inert clay particles and are readily removable therewith, as by filtration or centrifuging.

It may be noted that the improved refining action secured by employment of the refining agent of the present invention is also secured in instances where other materials and refining agents are concurrently present. For instance, wherever very fine or colloidal particles, for example, colloidal particles of bentonite are present, the refining agent of the present invention tends to cause the building up of larger agglomerates to which the colloidal particles apparently attach, thereby facilitating separation. In fact, in some cases particularly effective separation and refining takes place as a result of the concurrent use of the improved refining agent of this invention and a final pulverized or colloidal inert clay such as bentonite.

Examples

For purposes of illustration, reference is made to the following examples, all of which were performed on an aqueous solution (50% concentration) of the raw, impure sugar crystals of the type customarily shipped from the plantation to the refinery. The pH value of the syrup prepared for treating was 6.2. In each example, a Buckner vacuum filter was employed, with the same filter paper, i. e., Reve Angel No. 202, 18.5 cm. In all of the examples the solution was heated to 85° C. before filtration.

Example 1

The pH of the sugar solution was adjusted to 7.75 by adding the refining agent prepared according to this invention (by heating marl to 2600° F. and pulverizing), the agent containing 60% of calcium oxide and 40% of clay. Filtration time for this example was 45 seconds.

Example 2

To a 300 cc. batch of the sugar solution two drops of phosphoric acid were added and the pH was then adjusted to 7.6 by adding the refining agent of this invention, as in Example 1. Filtration time for this example was 58 seconds.

Filtration times in the foregoing examples compare with a time of 1 minute and 35 seconds when the same sugar solution was treated with the customary milk of lime or lime slurry heretofore employed.

Color readings were also taken on the filtered product of Examples 1 and 2 above, as well as on

the sample of the syrup which was treated with the customary milk of lime. These color readings were made on a photometer, the readings given in the following table representing the per cent of light transmission through a 20% sugar solution of each example.

Product tested:	Color reading
Product of Example 1	40.2
Product of Example 2	49.7
Same syrup treated with milk of lime	39.75
Refined white sugar	85

I claim:

1. A process for refining sucrose solutions containing, as impurities, organic acids, which process comprises dispersing in a sucrose solution a refining agent comprising finely divided particles of conjointly prefused lime and clay, the quantity of lime present being greater than that required to neutralize the organic acids present, heating the dispersion to a temperature between about 175° F. and 205° F., whereby to form reaction products by reaction between the lime and the organic acids without appreciable inversion of sucrose to glucose, and separating said reaction products with the particles of the refining agent.

2. A process according to claim 1 wherein the refining agent comprises from about 58% to about 66% of lime.

3. A process for refining sugar solutions comprising dispersing in a sugar solution a refining agent comprising finely divided particles of conjointly prefused lime and clay, heating the dispersion to form reaction products by reaction between the lime and impurities, and separating said reaction products with the particles of the refining agent.

4. A process for refining sugar solutions comprising concurrently treating a sugar solution with phosphoric acid and with a finely divided solid refining agent, said agent comprising finely divided particles of conjointly prefused lime and clay, heating the solution containing the phosphoric acid and the refining agent to form reaction products by reaction between the lime and impurities and by reaction between the lime and the phosphoric acid, and separating said reaction products with the particles of the refining agent.

5. A process for refining sugar solutions containing organic acids which process comprises dispersing in a sugar solution, a refining agent comprising finely divided particles of conjointly prefused lime and clay, the quantity of said agent added being sufficient to provide a quantity of lime present in the solution sufficient to insure a pH value between about 7.3 and about 7.6, heating the dispersion to a temperature in the neighborhood of 190° F., and separating reaction products formed by reaction between the lime and impurities in the solution, the separation of said reaction products being effected along with the particles of the refining agent.

6. A process for refining sugar solutions comprising dispersing in a sugar solution a refining agent comprising particles of lime and clay conjointly prefused at a temperature between 2400° F. and 2900° F. and thereafter ground to a fineness such that 85 to 90% will pass a 200 mesh sieve, heating the dispersion to form reaction products by reaction between the lime and impurities and separating said reaction products with the particles of the refining agent.

7. A process in accordance with claim 6 in which the amount of refining agent dispersed in the sugar solution is sufficient to insure a pH value between 7 and 7.6.

8. A process for refining sugar solutions comprising dispersing in a sugar solution a refining agent comprising finely divided particles of low grade lime-containing marl which has been fused at a temperature between 2400° F. and 2900° F. and thereafter ground to a fineness such that 85 to 90% will pass a 200 mesh sieve, heating the dispersion to form reaction products by reaction between the lime and impurities and separating said reaction products with the particles of the refining agent.

9. A process in accordance with claim 8 in which the fused and ground marl comprises from about 58% to about 66% of lime.

10. A process for refining sugar solutions comprising dispersing in a sugar solution a refining agent comprising finely divided particles of fused lime-containing marl, heating the dispersion to form reaction products by reaction between impurities and said agent and separating said reaction products with the particles of the refining agent.

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