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PROCESS FOR DEFECATION OF SUGAR SOLUTIONS

Earl Jack Swarthout, Edwardsburg, Mich., assignor to Miles Laboratories, Inc., Elkhart, Ind., a corporation of Indiana

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This invention relates to an improved process for the defecation of sugar solutions. More particularly, it relates to the use of iron or copper salts to remove undesirable impurities from sugar solutions prior to subsequent crystallization.

Crystalline sugar, such as crystalline dextrose, is generally obtained by well known crystallization techniques from sugar-containing solutions. Certain well known purification procedures are also employed to remove impurities from the sugar solutions prior to subsequent crystallization. Color impurities are removed by color refining agents, such as natural bone char, synthetic bone char, activated vegetable carbons and ion exchange resins. Ash impurities are removed by ion exchange resins. It is also well known that certain other undesirable impurities which interfere with subsequent crystallization are also present in sugar solutions. These latter impurities, which are believed to be proteins, mucoproteins and high molecular weight polysaccharides, produce undesirable color in the crystalline sugar product, causing the sugar to darken upon standing. They also inhibit initiation of crystallization, prevent desirable crystal size from being attained, and prevent efficient separation of sugar crystals from the liquor after crystallization. Many techniques for removing these contaminating protein and polysaccharide impurities have been suggested and tried in the prior art. Such prior art techniques have included precipitation of these impurities by adding lime to a pH of about 6.8–7.2 and flocculation of these impurites by adding a mixture of phosphoric acid and calcium hydroxide to a pH of about 6.9–7.1. These prior techniques have not been completely satisfactory and have actually created some disadvantages. For example, it is frequently desirable to heat the defecated sugar solution elevated temperatures above about 140° F. (60° C.) prior to filtering the defecated material. At a pH of about 7 or above, hot sugar solutions will degrade somewhat causing an increase in undesirable color and the formation of levulinic acid, both of which are detrimental to the economy of subsequent recovery-purification steps.

It is an object of the present invention to provide a process for removing substantially all undesirable coloring and high molecular weight polymeric impurities from sugar solutions.

It is another object of the present invention to provide a process for defecation of sugar solutions which does not tend to cause sugar degradation at elevated temperatures.

In accordance with the present invention, an improved process for defecation of sugar solutions is provided which comprises adding to a sugar solution at least one water-soluble compound selected from the class consisting of iron salts and copper salts, mixing said sugar solution and water-soluble compound until a floc is formed with the undesirable impurities and then separating the floc and the so-purified sugar solution. Preferably the water-soluble compound is added in an amount containing from about 50 to about 1000 parts by weight iron, copper or mixtures thereof per million parts by weight of the sugar solution. Such compounds when added in the amounts indicated produce a pH of about 2.5–5 in the sugar solutions so that the solutions thus treated do not tend to degrade at elevated temperatures. Most of the iron and copper salts are removed during the formation and separation of the floc. The remainder of the iron and copper salts are removed during subsequent ion exchange treatment which is generally employed in sugar crystallization processes for ash removal. The resulting crystallized sugar is thus safe for human consumption.

The process conditions for carrying out this invention are not narrowly critical. As the temperature increases, the flocculation time tends to decrease. If the temperature is about 25° C., for example, the flocculation will require about 8 hours. If the temperature is about 100° C., the flocculation time is about 30 seconds. After flocculation is complete, the sugar solution is preferably cooled to room temperature prior to separation of the floc or precipitate from the remainder of the sugar solution.

Separation of the floc can take place in various ways. Filtration is a preferred technique, but decantation of supernatant liquid from floc which has settled through normal gravity or by centrifugation can also be employed.

Atmospheric pressure is conveniently and preferably employed in this process of forming the floc, but pressures above and below atmospheric can be employed if desired.

The water-soluble salts useful in the present invention are exemplified by ferric chloride, ferric lactate, ferric nitrate, ferric citrate, ferric bromide, ferric formate and the like; ferrous acetate, ferrous bromide, ferrous chloride, ferrous iodide, ferrous nitrate, ferrous thiocyanate, ferrous thiosulfate and the like; cupric nitrate, cupric salicylate, cupric sulfate and the like; and cuprous bromide and the like. The ferric and cupric salts are preferred since it is believed that the metals must be in these valence states to form the floc with the impurities. When ferrous and cuprous salts are used, they should desirably be oxidized to the ferric and cupric states. This is conveniently accomplished through the well known self-initiating spontaneous oxidation that will occur during the heating of the sugar solution and water-soluble salt additive to form the floc. The expression "water-soluble salts" refers to salts which are soluble enough in water to provide sufficient metal ions to form the floc with the impurities.

This invention is further described in the following examples:

Example 1

Crude slab sugar was dissolved in water to form a 25 weight percent sugar solution having a pH of approximately 4.6. The solution which was brown and very cloudy was then treated with supric sulfate to give a pale green color (about 305 parts by weight copper per million parts by weight sugar solution were added). The resulting solution was heated to boiling and a heavy brownish precipitate formed. After cooling to room temperature and removing the precipitate by filtration, a yellow sparkling clear liquid remained. It is quite apparent that substantial purification had taken place. Subsequent ion-exchange treatment to remove ash impurities and crystallization of the above defecated sugar solution resulted in sugar crystals of improved properties, such as improved color and crystalline character.

Example 2

Crude slab sugar was dissolved in distilled water giving a dark brown cloudy solution containing 30 percent by weight glucose. Ferric citrate was then added in an amount 408 parts by weight iron per million parts by weight sugar solution. The resulting mixture was heated to boiling, held at this temperature for 30 seconds, then cooled to room temperature. A heavy precipitate formed which settled rapidly. After removal of the precipitate by filtration, a sparkling clear solution remained from which sugar of improved properties can be obtained.

While the above examples were limited to use of spe-

cific iron and copper salts with solutions of crude slab sugar, it should be understood that other water-soluble iron or copper salts can be employed either alone or in admixture, and other sugar solutions, such as acid-converted starch, enzyme-converted starch and the like can be employed in this invention.

In summary, the present invention relates to an improved defecation process for removing undesirable impurities from sugar solutions. This improved process involves treating the sugar solution with a water-soluble salt of iron, copper or mixtures thereof to form a floc or precipitate with the impurities and then separating the so-purified sugar solution from the floc.

What is claimed is:

1. A process for defecation of sugar solutions containing undesirable impurities which consists essentially of adding ferric citrate to a sugar solution, said ferric citrate being added in an amount containing from about 50 to about 1000 parts by weight iron per million parts by weight sugar solution, mixing said sugar solution and said ferric citrate at a pH of from about 2.5 to about 5 until a floc is formed with the undesirable impurities, and then separating the floc and the so-purified sugar solution.

2. A process for defecation of sugar solutions containing undesirable impurities which consists essentially of adding cupric sulfate to a sugar solution, said cupric sulfate being added in an amount containing about 50 to about 1000 parts by weight copper per million parts by weight sugar solution, mixing said sugar solution and said cupric sulfate at a pH of from about 2.5 to about 5 until a floc is formed with the undesirable impurities and then separating the floc and the so-purified sugar solution.

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MORRIS O. WOLK, *Primary Examiner.*

M. E. ROGERS, *Assistant Examiner.*