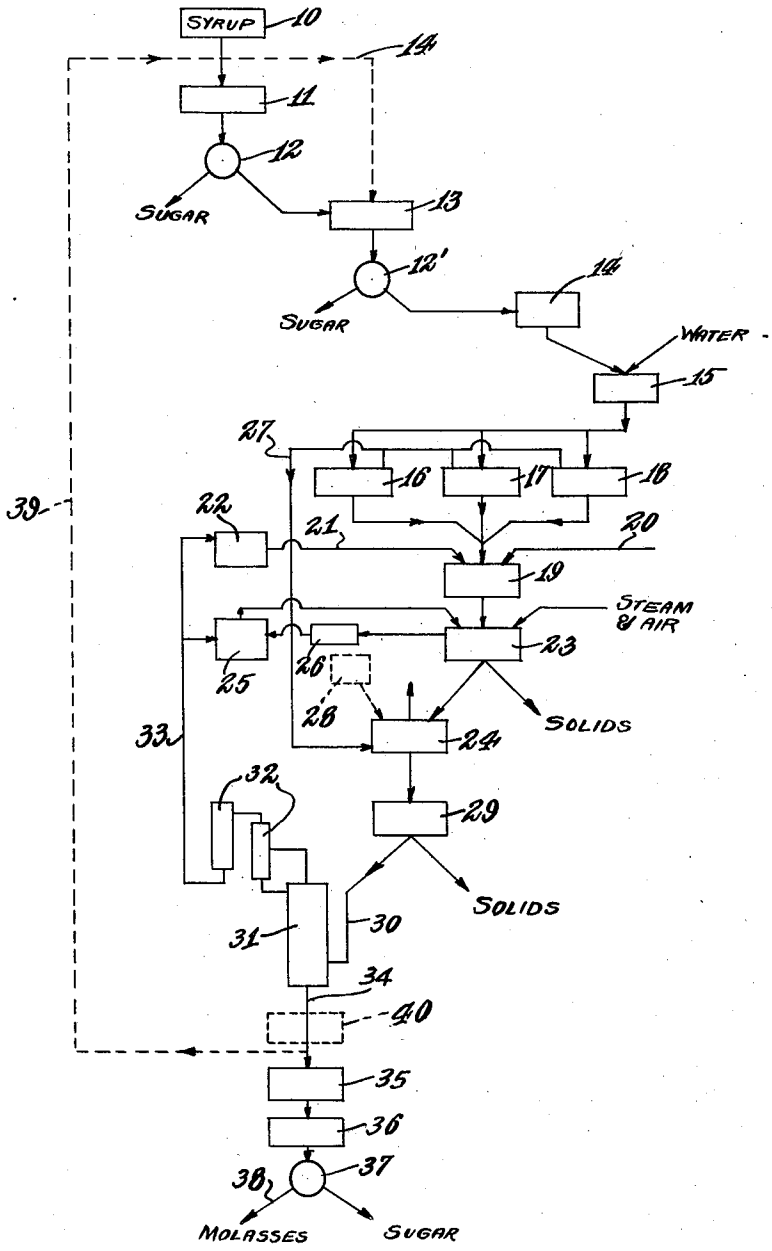
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PROCESS OF TREATING SUGAR SOLUTIONS

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PROCESS OF TREATING SUGAR SOLUTIONS

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This invention relates to an improved process of refining sugar and particularly to a process of recovering sucrose from impure sugar solutions.

The invention has for its general object the provision of a method or mode of operation whereby not only can the normal sugar refining process be improved but in addition the production of greater quantities of sugars of higher purity may be insured.

The process of this invention is not only applicable to the refining of raw sugar, but is also applicable to the production of what are known as unrefined cane sugars, said unrefined cane sugars forming a class intermediate between raw and refined sugars. A further object of this invention is to disclose and provide a method of refining sugar and whereby sugars of high purity and good keeping qualities may be more economically produced without material alteration of the normal plantation refining systems.

In all sugar refining processes, whether the process is designed to produce white refined sugars or plantation whites or raw sugar, large volumes of impure sugar solutions are handled and eventually accumulated, these impure sugar solutions being generally referred to as molasses. Hereinafter the term "molasses" will be applied to all impure sugar solutions whether such impure sugar solutions are actually molasses or not. In the manufacture of sugar from sugar cane, for example, a number of different grades of molasses are produced, these being generally known as first, second, third, fourth and final or black-strap molasses. The number of different kinds of molasses produced is optional, depending upon whether second, third or fourth massecuite system is used.

This invention concerns itself primarily with what is known as second, third or final molasses, all of these molasses having a polarization below about 60° Pol. These impure sugar solutions generally contain sucrose but in addition also contain proteins, albuminoids, amido acids, and other nitrogenous bodies, pentosans, coloring matters such as anthocyanin, mineral salts, and other substances which retard or prevent the crystallization of sucrose from the solutions.

An object of this invention is to disclose and provide a method of refining sugars whereby larger yields of sucrose may be obtained.

Another object is to disclose and provide a method of treating impure sugar solutions for the recovery of sucrose therefrom.

An object of this invention is to disclose and provide a method of refining sugar whereby the production of different grades of molasses is obviated and the extraction of sucrose therefrom made economically possible.

A further object of this invention is to disclose and provide a method of treating impure sugar solutions whereby the sucrose content thereof may be recovered.

A still further object of this invention is to disclose and provide a process of sugar refining which permits the production of large quantities of high purity sugars, the residual molasses containing very little glucose and consequently being well adapted for either the Steffens or Barium process by which the remaining sugar can be extracted.

These and other objects, uses and advantages of this invention will in part become obvious to those skilled in the art and will readily become apparent from a consideration of the detailed description of the invention given hereinafter. In describing the invention, reference will be had to the appended drawing which diagrammatically illustrates the process.

It is to be understood that this invention is applicable to the production of sugar, not only from sugar cane juices, syrups or molasses, but also from the treatment of sugar-containing solutions from various other sources such as, for example, sorghum, beets, etc. For purposes of lucidity, the process as described hereinafter will particularly refer to the adaptation of this invention to a cane sugar refinery.

As is well known, the sugar cane is first milled either by suitable revolving knives or shredders and then crushed. The bagasse may be sprayed with water between crushing operations so as to facilitate extraction. The juice is then strained to remove bagacillo and the strained juice clarified in any suitable manner. The clarified juice is then sent to evaporators and the sugar syrup thus produced.

As shown in the appended drawing, this syrup having a purity of between about 82 and 90, may be sent from the tank 10 to a suitable crystallizing pan or calandria 11 wherein crystallization is allowed to take place, giving rise to what is known as first massecuite. This massecuite is then sent to centrifugal separators 12 wherein the sugar is separated from the mother liquor, this mother liquor being generally termed first molasses. The purity of this first molasses may vary widely in practice from about 60° to 75° Pol. This first molasses may either be returned to the same crystallizing pan or to a second

crystallizing pan 13. A certain amount of syrup from the tank 10 may also be sent to the pan 13 as by line 14. The second strike of massecuite is again sent to the centrifugals 12' and the sugar separated from the mother liquor, this mother liquor being now termed second molasses. The sugar from the centrifugals 12' may be washed and bagged, being of a grade generally referred to as raw sugar.

The sugar from the centrifugals 12' is generally of slightly lower purity than the sugar from centrifugals 12 and furthermore is of darker color and does not have the keeping qualities of the sugar from centrifugals 12. The second molasses from the centrifugals 12' is generally treated so as to produce a third massecuite and so on, depending upon the number of strikes desired or capable of being made. This second molasses, however, generally has a purity of less than about 60° Pol. and in accordance with this invention is not again sent to crystallizing pans. Instead, such molasses (or any other similar impure sugar solution) is sent to a storage tank 14 from which it may be withdrawn as occasion warrants, into a dilution tank 15 wherein it may be diluted with water to any desired concentration. For example, the molasses or impure sugar solution may be diluted in the tank 15 in accordance with the teachings of Patent #1,788,628, to between about 35 and 65° Brix so as to allow the subsequent fermentation of invert sugar present in such impure solution to take place without the necessity of using special selectively fermenting yeasts. If desired, however, special yeasts may be employed, in accordance with the teachings of Patent #1,572,359 or Patent #1,401,433.

The diluted impure sugar solution may then be sent to fermenting tanks 16, 17, 18, etc., wherein fermentation is allowed to take place so as to convert the invert sugar into alcohol without substantial inversion of the sucrose. The fermented molasses, generally containing from 2% to 10% of alcohol (caused by the fermentation) may then be sent to the tank 19 and a small quantity of alkaline earth oxide or hydroxide added as by the line 20. Preferably, before the addition of the alkaline earth oxide or hydroxide (such as milk of lime), a quantity of alcohol is added to the fermented molasses, this alcohol being added by line 21 from an alcohol storage tank 22. It has been found that the presence of alcohol during the precipitation of organic impurities by lime in the tank 19 is highly desirable, the alcohol apparently having three functions: first, it stops the fermentation; second, it aids the alkaline earth oxide or hydroxide in precipitating organic impurities, coloring matter, etc.; third, it renders subsequent separation of the precipitated substances from the liquid easy. If the lime were to be added to a fermented molasses containing only 3% or 4% alcohol, not only would the precipitation of organic impurities be incomplete, but in addition, the separation of the precipitate from the liquid by filtration would be almost impossible because of the character, consistency and slimy nature of the precipitate. It has been found desirable to add a volume of alcohol equal to the volume of the fermented molasses before precipitating the organic impurities and coloring matter with lime.

When an equal volume of alcohol is thus employed, 5% to 6% by volume of a 20% milk of lime solution may be added to insure complete precipitation of the organic impurities. It is to be understood, however, that the lime may be added to the fermented molasses before the addition of the alcohol. The amount of lime added to the tank 19 for the purpose of precipitating organic impurities and coloring matter without precipitating the sucrose, will vary with the amount of alcohol present. In all events, a quantity of lime insufficient to precipitate the sucrose should be added.

The solution may then be filtered as in the filter 23 or the precipitate separated from the liquid in any other suitable manner. The filtered sugar solution may be discharged into a tank 24. The filter press may be washed with alcohol from a storage tank 25 and then blown with steam or steam and air, so as to recover the alcohol, the steam and air passing into a condenser 26 and the alcohol condensed therein returned to the storage tank 25. The filter press cake resulting from this operation may be discarded.

The sugar solution collected in the tank 24 is alkaline in character, containing a mixture of calcium and potassium hydroxide, the alkalinity usually running about 0.2% calculated as CaO. This slightly alkaline solution is then preferably neutralized in any suitable manner.

For example, carbon dioxide collected from the fermenting tanks 16, 17 and 18, may be conveyed by line 27 and passed through the solution in tank 24 so as to precipitate the calcium and potassium salts which are substantially insoluble in the alcoholic solution in the tank 24. Instead of using carbon dioxide, sulfuric, phosphoric, or any other acid which will form substantially insoluble calcium and potassium salts, may be mixed with the solution in tank 24, such acid being supplied from a suitable storage indicated at 28.

The neutralization is preferably carried on to a pH of between about 6.6 and 7.2. Ordinary temperatures are recommended during this neutralization step so as to prevent loss of alcohol but if provision is made therefor, higher temperatures may be used satisfactorily.

The neutralized sugar solution may then be filtered in a filter 29 and the cake consisting of the precipitated bodies discarded. The alcoholic sugar solution may then be sent through line 30 to a suitable still or evaporator 31 in which the alcohol is separated from the sugar solution, the alcoholic vapors passing through suitable condensers 32 and the alcohol being returned by line 33 to the alcohol storage tanks 25 and 22.

The de-alcoholized sugar solution or syrup may then be sent by line 34 to any suitable evaporator 35 and then to a crystallizing pan 36 from which the mass may be discharged into a centrifuge 37 for the purpose of separating the crystallized sugar from the residual molasses. The molasses discharged at 38 contains very little glucose and no gums and consequently is well adapted for either the Steffens or Barium process by which the remaining sugar can be extracted. If desired, the sugar solution from line 34 may be sent as by line 39 to the initial refining operations, becoming either a part of the syrup fed to the first pan 11, or to the second pan 13.

As a modification of the process described hereinabove, the alkaline sugar-containing filtrate from the filter 23 may be sent directly to a still such as the still 31, for the removal of alcohol, thus eliminating the neutralizing operation 24 and the filtration operation 29. When alkaline sugar-containing filtrate from filter 23 is sent directly to still 31, the alkalinity of the filtrate is used up by the organic bodies remain-

ing in the liquid and the de-alcoholized syrup comes out in substantially neutral form but with a dark precipitate which can be removed by a subsequent filtration as, for example, in the filter 40.

A still further modification of the process may consist in precipitating the interfering organic impurities in tank 19, filtering the solution at 23, neutralizing the filtrate in the tank 24, and then discharging the neutralized filtrate directly to the still 30 for de-alcoholization. The sugar solution discharged from the still under these conditions would contain solid impurities and for this reason, such solution would have to be filtered as at 40 before being sent to the evaporators and crystallizing pan.

Those skilled in the art will observe from the description given hereinabove that an improved process of refining sugar has been disclosed. In order to specifically refer to the results attained by this process, reference will be had to the following example: 1 ton of second molasses of about 80° Brix, containing 19.51% glucose, 44.77% sucrose, 5.09% ash and 8.22% organic non-sugars, was diluted to 48° Brix, a yeast culture added, and the molasses fermented. After dilution but before fermentation, the molasses or impure sugar solution consisted of 340 gallons containing 11.68% glucose, 26.81% sucrose, 3.05% ash, 4.92% organic non-sugars, 46.46% solids, and a real purity of about 57.71. After fermentation, this molasses solution contained only 2.2% glucose and 26.76% sucrose. Its real purity had been increased to 64.83. This fermented molasses was mixed with about 300 gallons of alcohol and about 40 gallons of 20% milk of lime, for the purpose of precipitating the organic impurities and coloring matter. After filtration, it was found that the filter cake weighed about 300 pounds, contained about 0.65% P_2O_5, 43.15% ash, other than P_2O_5, 10.60% protein, and 39.24% organic matter other than protein. The alkaline sugar solution or filtrate was then neutralized with dilute sulfuric acid and filtered. The filtrate contained only 0.84% glucose, 26.16% sucrose, 1.87% ash, and 3.75% organic non-sugars. It had a real purity of 80.18 and upon being de-alcoholized, passed through a crystallizing pan and then centrifuged to give rise to about 716 pounds of sugar of 96° Pol. and about 537 pounds of final molasses. The filter press cake from the filtration after neutralization with dilute sulfuric acid, consisted of about 40 pounds, of which about 54% consisted of calcium sulfate and 34% consisted of potassium sulfate.

These specific figures clearly show that the ash content of the molasses is materially reduced, the loss in sucrose content is negligible, and that a sugar of relatively high purity can be made economically. It is to be observed that the repeated reboiling of successive grades of molasses, with accompanying cost of power and labor, is eliminated to a great extent by the method of this invention. The cost of the sugar is thus reduced not only by reason of the increased yield and higher purity, but also by reason of the reduction in power and labor costs. The loss of alcohol in the process is negligible and instead alcohol is actually produced as a by-product. The investment in equipment (other than that found normally in plantation refineries) is very low, it being understood that instead of using a number of separate units (as, for example, filters 23, 29 and 40 or centrifugals 12, 12' and 37) the same units can be employed at different times for the different operations.

In a further modification of the process (particularly adapted to treatment of molasses of low glucose content) the step of fermenting molasses may be eliminated. In this modification, alcohol and lime are added to the unfermented molasses and the process carried out as described hereinbefore.

Although the use of alkaline earth oxides and hydroxides for the precipitation of organic and coloring impurities has been specifically stated, it is to be understood that other substances may also be employed for this purpose, for example, aluminum hydroxide or aluminum cream.

Certain modifications have been described in detail in the description given hereinabove but other modifications may be made in the process without departing from the scope of the invention.

All such changes and modifications as come within the scope of the appended claims are embraced thereby.

I claim:

1. In a process of refining sugar, the steps of withdrawing molasses having a polarization of below about 60° Pol., from a sugar refining system, diluting such molasses with water, adding yeast to such molasses, fermenting invert sugar present in said molasses without inversion of sucrose, adding alcohol to the fermented molasses in the substantial absence of invert sugar, adding an alkaline earth oxide or hydroxide to the alcoholic sucrose solution in amount sufficient to precipitate organic impurities but insufficient to precipitate sucrose, separating the precipitated impurities from the alcoholic sucrose solution, neutralizing the alcoholic sucrose solution with an acid capable of forming substantially insoluble calcium and potassium salts, separating the precipitated salts from the substantially neutral alcoholic sucrose solution, said solution being substantially free from invert sugar, removing alcohol from the solution, and then concentrating the solution and crystallizing sugar therefrom.

2. In a process of refining sugar, the steps of withdrawing an impure solution containing invert sugars and sucrose and having a polarization of below about 60° Pol., from a sugar refining system, diluting such impure solution with water, adding a yeast thereto, fermenting the diluted solution to remove invert sugars without inverting the sucrose, adding alcohol to the fermented solution, adding an alkaline earth oxide or hydroxide to the alcoholic sucrose solution in amount sufficient to precipitate organic impurities but insufficient to precipitate sucrose, separating the precipitated impurities from the alcoholic sucrose solution, neutralizing the alcoholic sucrose solution with an acid capable of forming calcium salts which are substantially insoluble in such alcoholic sucrose solution, removing alcohol from the solution, and then separating precipitated impurities from the de-alcoholized sucrose solution to form a sucrose solution substantially free from invert sugar whereby sucrose may be crystallized therefrom.

3. In a process of refining sugar, the steps of withdrawing an impure sugar solution containing invert sugars and sucrose and having a polarization of below about 60° Pol., from a sugar refining system, diluting such impure solution with water, adding a yeast thereto and fermenting the solution to remove invert sugar without inverting the sucrose, adding an alkaline earth

oxide or hydroxide to the alcoholic sucrose solution in amount sufficient to precipitate organic impurities but insufficient to precipitate sucrose, separating the precipitated impurities from the alcoholic sucrose solution, neutralizing the alcoholic sucrose solution with an acid capable of forming substantially insoluble calcium and potassium salts, removing alcohol from the solution by distillation, then separating precipitated impurities from the de-alcoholized sucrose solution, said solution being substantially free from invert sugar whereby sucrose may be crystallized therefrom.

4. In a method of recovering sucrose from impure sugar solutions, the steps of fermenting an impure solution containing invert sugar and sucrose to remove invert sugar without inverting the sucrose, adding alcohol to the fermented solution in volume approximating the volume of the fermented solution, adding an alkaline earth oxide or hydroxide to the alcoholic sucrose solution in amount sufficient to precipitate organic impurities but insufficient to precipitate sucrose, separating the precipitated organic impurities from the alcoholic sucrose solution, removing alcohol from the sucrose solution by distillation, and then separating precipitated impurities from the de-alcoholized sucrose solution to form a sucrose solution substantially free from invert sugar whereby sucrose may be crystallized therefrom.

5. In a method of recovering sucrose from impure sugar solutions, the steps of fermenting an impure solution containing invert sugar and sucrose to remove invert sugar without inverting the sucrose, adding alcohol to the fermented solution in volume approximating the volume of the fermented solution, adding an alkaline earth oxide or hydroxide to the alcoholic sucrose solution in amount sufficient to precipitate organic impurities but insufficient to precipitate sucrose, separating the precipitated impurities from the alcoholic sucrose solution, neutralizing the alcoholic sucrose solution to a pH of about 6.6–7.2 with an acid capable of forming calcium salts which are substantially insoluble in such alcoholic sucrose solution, separating the precipitated salts from the substantially neutral alcoholic sucrose solution, and then removing alcohol from the solution to form a sucrose solution substantially free from invert sugar whereby sucrose may be crystallized therefrom.

6. In a method of recovering sucrose from impure sugar solutions, the steps of fermenting an impure solution containing invert sugars and sucrose to remove invert sugar without inverting the sucrose, adding an alkaline earth oxide or hydroxide to the alcoholic sucrose solution in amount sufficient to precipitate organic impurities but insufficient to precipitate sucrose, separating the precipitated impurities from the alcoholic sucrose solution, neutralizing the alcoholic sucrose solution with an acid capable of forming calcium salts which are substantially insoluble in such alcoholic sucrose solution, separating the precipitated salts from the substantially neutral alcoholic sucrose solution, and removing alcohol from the solution to form a sucrose solution substantially free from invert sugar whereby sucrose may be crystallized therefrom.

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