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PURIFIED LIQUID SUGAR CONCENTRATE AND METHOD
OF MANUFACTURING SAME
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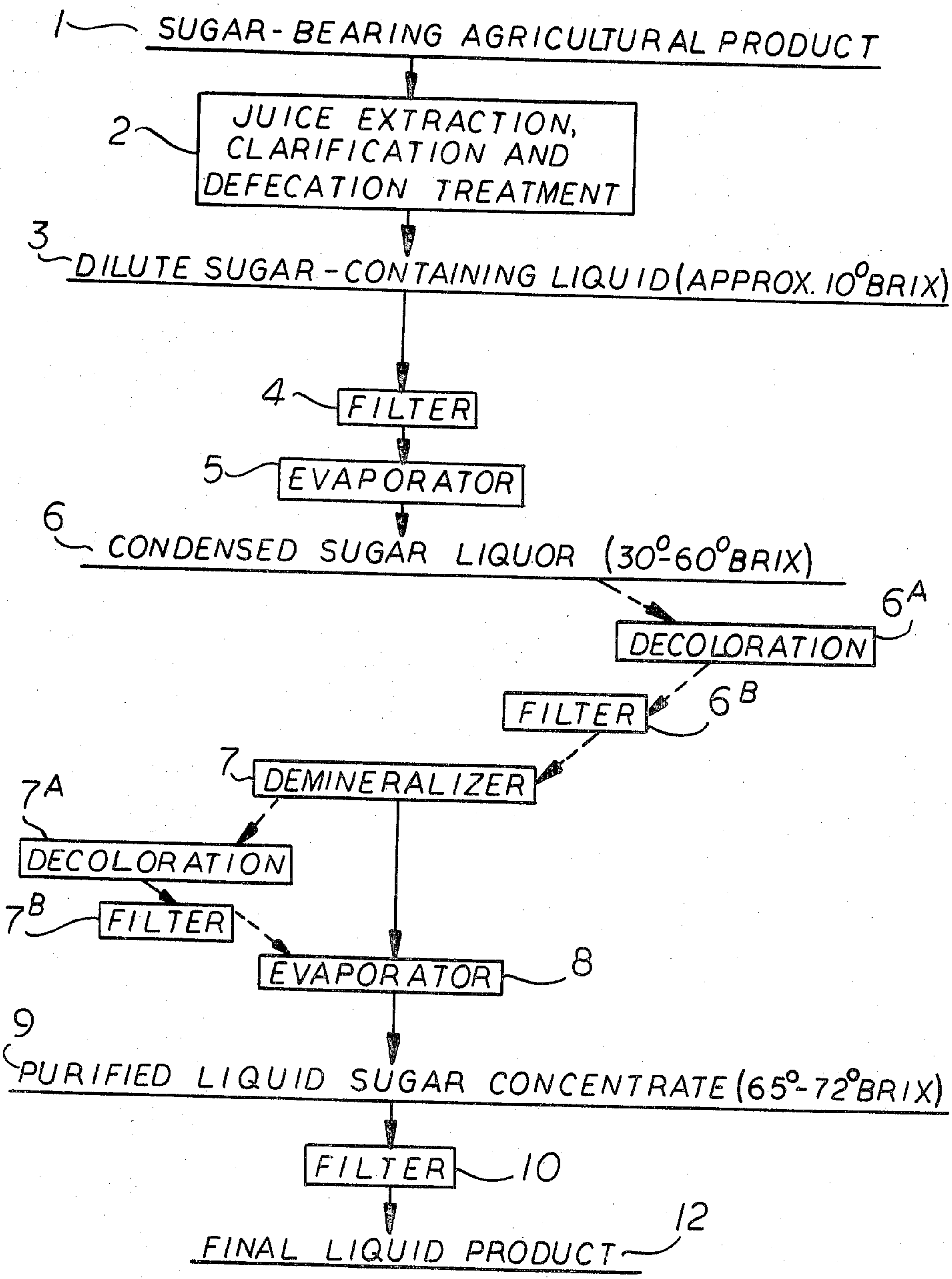


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**PURIFIED LIQUID SUGAR CONCENTRATE AND METHOD OF MANUFACTURING SAME**

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**U.S. Cl. 127—46** **2 Claims**


## ABSTRACT OF THE DISCLOSURE

A pure liquid sugar concentrate of at least 65° Brix is produced directly from a dilute sugar-containing liquid in a refining process which excludes crystallization as a required procedure in the method of purification. The refining includes concentration, demineralization in a mixed resin ion exchanger, further concentration and filtration.


## SUMMARY OF THE INVENTION

This invention relates to a process for the purification of dilute sugar-containing liquids, such as sugar juice, obtained from any sugar-bearing agricultural plant. More particularly, it relates to a simplified process for manufacturing a purified liquid sugar concentrate containing at least 65 weight percent of sugar solids which process excludes the step of crystallization.

In the manufacture of refined sugar solutions it is conventional practice to purify or defecate raw sugar juice by first treating the solutions with, for example, calcium hydroxide and carbon dioxide. Sugar solutions which have been pre-treated in this manner are however, not sufficiently pure and therefore have to be subjected to further refining or defecating treatment, particularly for the purpose of removing undesirable alkaline earth metal ions such as calcium and magnesium ions and various color bodies.

The present invention is concerned with the further purification of pre-treated dilute sugar solutions, i.e., sugar solutions or juices which have been subjected to the customary lime-carbon dioxide defecation treatment referred to or to similar equivalent pre-treating procedures.

Heretofore, pre-treated sugar solutions have been purified by removing the ash components by chemical precipitation or by ion-exchange, followed by subsequent boiling and crystallization operations. Ion-exchange resins employed in sugar purification processes are those having high de-ashing capacity, i.e., the ability to remove ionic impurities and also having high refining capacity for aqueous solutions, i.e., the ability to remove organic impurities present in sugar liquors for example, color bodies and materials giving rise to color bodies, colloidal material and the like. As these desirable properties are not possessed by any single exchanger system available it has been necessary therefore to accept some compromise between quality of sugar liquor effluent from the ion-exchangl system and economical operation. In order to meet the problem, it has been necessary to utilize crystallization as a separation procedure in order to manufacture a sugar product of satisfactory purity from sugar solutions produced by conventional pretreating and ion-exchange methods. Crystallization procedures, however, are time



consuming, require expensive equipment and are inherently inefficient from the point of view of percentage of product recovered. Further, with the rising demand in industry for liquid sugar products, the requirement that the product to be sold as an aqueous solution be first separated in crystal form has become a particularly burdensome problem. Yet so far as is known, no prior practical process capable of industrial utilization has been scccessfully employed in the manufacture of a purified liquid sugar concentrate, which did not require crystallization as an essential step in the purification of the final product.

Accordingly, among the objects of the present invention are to provide a novel purification method; to provide such a method which is particularly adapted for the treatment of dilute sugar-containing solutions; to provide such a method which can be utilized in the treatment of sugar solutions obtained by the extraction of sugar by means of water from any sugar-bearing agricultural plant; and to provide such a method by which a high purity juice or solution can be obtained with optimum efficiency which eliminates crystallization as an essential step of the process.

Briefly, the present invention provides a process for manufacturing a purified liquid sugar concentrate of at least 65° Brix directly from a dilute sugar containing liquid which process excludes the step of crystallization. In accordance with the novel process a defecated dilute sugar-containing liquid is condensed to produce a sugar liquor of from about 30° to 60° Brix. The condensed sugar liquor is thereafter decolorized and contacted with an ion-exchange material to remove dissolved solids and organic impurities. Thereafter the ion-exchange treated sugar liquor is further condensed to produce a liquid sugar product containing at least 65 weight percent of sugar solids.

Additional objects and advantages of the present invention will become more apparent from a consideration of the following specification and claims, when taken in conjunction with the accompanying drawing, wherein:

The single figure illustrates diagrammatically an illustrative process embodying the present invention.

It is to be understood that there is no intention to limit the invention to he specific forms and examples disclosed, but on the contrary, the invention covers all modifications, alternatives, equivalents and uses falling within the spirit and scope of the invention as expressed in the appended claims.

The dilute sugar-containing liquid which is treated in accordance with the present invention, can be obtained through the use of conventional extraction processes from any sugar-bearing agricultural plant such as, for example, sorghum, can, corn, beet, pineapple, or the like. The raw product is cleaned, as by washing and trash separation, then cut or chopped into relatively thin slices by sets of rotating knives or the like. In such processes, as illustrated in the flow diagram, the raw product **1** whether chopped fresh, dry shredded or a mixture of these, is subjected to pretreatment utilizing an extractor **2** comprising a crushing system or a diffusion system, or a combination of both, from which is obtained a raw sugar juice **3** containing about 10% sugar solids by weight, e.g., approximately 10° Brix.

Accordingly, the term "dilute sugar-containing liquid" as used herein, is defined as meaning raw juice containing about 10% sugar solids, i.e. surcrose, levulose (fructose), dextrose (glucose) or mixtures thereof, which have been extracted from a sugar-bearing agricultural product. It is to be understood however, that the concentration of the preferred starting material in the present process has been selected primarily due to economic considerations taking into account the type of refining equipment currently available, and that this invention can be utilized with equal efficacy to produce a purified liquid sugar concentrate from sugar-containing liquids in which the concentration of sugar solids is greater or less than 10%.

In accordance with the present invention as exemplified by the foregoing objects, the dilute sugar-containing liquid **3** is first pretreated using conventional clarification and defecation process apparatus **2** to remove a substantial portion of the impurities contained therein. Impurities carried over with the sugar juice coming from the extraction apparatus **2** include soluble inorganic salts and various organic compounds as well as insoluble compounds and constituents contained in the raw agricultural product and in the extraneous material not removed in the cleaning step, which impurities appear as suspended solids in the dilute aqueous solution of sugar. The kind and quantity of ash components found in an extracted sugar juice will vary depending upon the raw product used, the washing method and the type of extraction apparatus employed. Typically, for example, in a conventional diffusion process the water which is passed through the chopped raw product to dissolve out the sugar is heated to about 180° to 200° F. and tends to dissolve out or carry out compounds or constituents contained in the raw product which are less likely to be removed in a crushing roll system.

Gross de-ashing is accomplished by treating the dilute sugar-containing liquid with, for example, lime and a carbonate-forming compound such as carbon dioxide, followed by filtration. In some instances where desirable, floculating agents such as "Separan" manufactured by the Dow Chemical Company can be employed effectively. Alternatively, a phosphating process using phosphoric acid and lime can be substituted in place of the carbonatation method.

As the final procedure in the clarification and defecation of the crude sugar solution, the thin juice lime-slurry at a pH slightly on the alkaline side is passed at a temperature selected to minimize temperature change, generally about 180° to 190° F., through a filter **4** to remove all solid products. Advantageously, direct pressure filtration apparatus of the type disclosed in U.S. Pat. No. 3,310,171, patented Mar. 21, 1967 by Henry Schmidt, Jr., et al. can be employed in this purification step. In a leaf-filter structure of this type the filter cake is removed periodically from the filter leaves, in situ, primarily by the energy of fluid impingement, which energy creates secondary and harmonic forces because of being opposed by relatively fixed and movable leaf supports acting on the several filter leaves at spaced-apart points. By this method the quantity of fluid used for sluicing is maintained at a practical minimum while effecting maximum cake removal function in an automatically controlled operation. Such apparatus is particularly useful in the present process where a relatively heavy cake build-up occurs. Cake removal is accomplished by using high velocity, high impact type nozzles that are disposed between leaves on a rotating variable flow sluice header. Because of the bottom outlet configuration on the leaves themselves, it is a simple matter to blow the heel from the vessel and refill the vessel with a leaching liquid so that the cake may be leached in place if desired. Alternatively, or subsequently, the virgin or leached cake can be blown dry with air if the discharge of a dry cake is desired. In this case, the entire leaf assembly can be rolled out and the cake then discharged into an awaiting pan, trough or truck. Dry discharge is generally accomplished by means of actuation of a pneumatic vibrator which can also be used when the filter elements themselves are to be washed in place. It is a feature of the present invention that all defecation products produced in the process are cascaded to the first



filtration station, which is a dry discharge station, so that all such products are available at about 50% moisture for simple hauling away with the result that pollution problems are greatly simplified.

It has also been found that by lowering the extraction temperature in the diffusion step to a temperature in the range of about from 100° to 120° F. the amount of ash and color in the juice extracted from the raw product can be markedly reduced thereby reducing substantially the operating costs of further refining the product. Suitable cold extraction apparatus is disclosed in copending patent application Ser. No. 718,609 filed Apr. 3, 1968.

Following defecation, the pretreated dilute sugar-containing solution is passed to an evaporator **5** where it is condensed to remove a substantial portion of the water contained therein prior to further purification treatment for the removal of the remaining ash components. Preferably, in accordance with this invention, the pretreated solution is evaporated to produce a sugar liquid **6** having a concentration in the range of from about 30° to 60° Brix. Advantageously, the condensation can be carried out using a multi-effect steam heated evaporator of the type generally employed in conventional sugar refining processes. Thereafter, the sugar liquor from the evaporator of from about 30° to 60° Brix and more preferably in the range of from about 40° to 45° Brix, is demineralized by means of suitable ion-exchange materials in conventional demineralizer equipment **7**.

While the impurities which make up the ash components vary with the nature of the raw sugar-bearing agricultural product as well as with the areas in which such products are grown, some of the more common ash components which must be removed are sodium, potassium, calcium or magnesium salts, silica, traces of iron and certain organic compounds. The cation exchange material exchanges $H^+$ for $Na^+$, $K^+$, $Ca^{++}$, $Mg^{++}$ and the like, the acid thus formed being retained in the juice and the exchange ion remaining with the cation exchange material. The anion exchange material has the opposite chemical property and tends to take up or absorb the acid, exchanging an $OH^-$ ion therefore. When the anion exchange material exchanges $OH^-$ for the acid radical such as $Cl^-$, $SO_4^{--}$ and various organic radicals, such impurity will have been removed from the juice, since the $H^+$ of the acid combines with the $OH^-$ of the anion exchange material to form water and the acid radical remains with the anion exchange material. In the application of the ion-exchange process to sugar factories or mills, it has been customary to use several series of ion-exchangers, each series using a cation exchange bed and an ion exchange bed. Further, generally it has been necessary to provide several sets of beds so that one set can be utilized for ion-exchange while the other sets are being regenerated.

In the ion-exchange system of the present invention, preferably the condensed sugar liquor is passed through a mixed bed of anion and cation resins wherein the ash components are converted to hydrogen and hydroxide which in turn combine to form water. When the resins become exhausted they must be regenerated. The cation resin is regenerated with an acid such as hydrochloric or sulfuric acid and the anion resin is regenerated with a base such as sodium hydroxide or ammonium hydroxide. A suitable ion-exchange system for use in the present invention is disclosed in co-pending applications Ser. No. 396,840 filed Sept. 16, 1964 by Clay W. Riley et al. and Ser. No. 431,131 filed Feb. 8, 1965 by James F. Zievers et al.

Generally, it will be desirable to subject the condensed sugar liquor **6** to a decoloration treatment using granular carbon or decolorizing resin to remove the remaining organic material. In accordance with this invention the condensed sugar liquor can be decolorized before (step *6a*) or after (step *7a*) demineralization as desired.

Granular carbon has been used as an adsorbent for color, taste and odor for many years. Conventional systems generally are of the fixed bed, down-flow type, either in single or multiple column station, with the individual columns used in parallel or in series depending upon the requirements of the process. Granular activated carbon is particularly well suited to the present process because it can be regenerated and reused. Most advantageously the granular carbon treatment (step **6a** or **7a**) will be carried out in a single column with counter-current flow of materials. In the present invention the liquid either before or after demineralization, preferably will be fed into the columns at the bottom and will be taken off at the top. Periodically the column is pulsed. A predetermined quantity of exhausted carbon is removed from the bottom of the column and a like amount of regenerated carbon is added at the top. The result of such a processing is a more efficient use of the adsorbing powers of the carbon, lower total inventory of carbon and the production of less sweet water than would be produced in a fixed columnar processing system. Suitable apparatus for use in this process is disclosed in co-pending application Ser. No. 384,060 filed July 21, 1964 by James F. Zievers now U.S. Pat. No. 3,356,220.

The carbon treated condensed sugar liquor is then passed through a suitable filtration apparatus **6b** or **7b**, to remove the entrained solids suspended therein. Generally, it will be desirable to recycle the filtrate for clarity in accordance with standard decoloration practice. Suitable tubular filter apparatus is disclosed in United States Patent No. 3,244,286 patented Apr. 5, 1966 entitled "Filtration Apparatus" by H. Schmidt, Jr. et al.

The demineralized and decolorized sugar liquor having a concentration preferably not exceeding 60° Brix is thereafter passed to evaporator **8** and condensed to produce a purified liquid sugar concentrate product **9** having a concentration of at least 65° and preferably of 65°–72° Brix. The liquid product **9** is then polished in a final filter **10** using powdered activated carbon to produce final product **12**.

The following specific example is given to illustrate the extent of ash removal that can be accomplished by treating a dilute sugar-containing solution in accordance with the present invention by the elimination of crystallization as an essential step of the purification process.

Sugar juice containing 0.864 lb. of sugar solids per gal. (approximately 10° Brix) consisting of approximately 90% sucrose and 10% glucose-fructose mixture, and having an ash component content of 5%, extracted from corn is clarified and defecated by treating it in a suitable mixing chamber. In this example a thin juice lime-slurry is produced on a continuous basis utilizing calcium hydroxide and phosphoric acid in a single stage mixing chamber through which the sugar juice is passed. The juice pump discharges into a vertical cylindrical chamber having two spaced impellers axially mounted on and driven by a single shaft. Inlets are provided adjacent each impeller for the treating chemicals. The hot juice to be treated enters near the bottom of the mixing chamber, passes upwardly through the chamber where it is mixed with the lime-slurry and phosphoric acid and is discharged near the top. A suitable lime-slurry of sweet water containing approximately 1 lb. of calcium hydroxide per gal. is pumped into upper portion of the mixing chamber where it is mixed thoroughly by the top impeller. An aqueous solution of $H_3PO_4$ having a pH of about 2 is injected through the lower inlet into the chamber and mixed thoroughly by the bottom impeller. The mixed thin juice lime-slurry is treated as need be with calcium hydroxide to adjust the pH to the slightly alkaline side, e.g. pH=7.2 and pumped to a surge tank and preparatory to filtering out all solid products. Suitable apparatus for the clarification and defecation treatment described herein is disclosed in co-pending application Ser. No. 463,230 filed June 11, 1965 by James F. Zievers now U.S. Pat. No. 3,391,789 which describes such apparatus and its method of use in removing ionic impurities from an aqueous solution in a continuous process.

Preferably, the thin juice lime-slurry will be circulated through the pressure filtration apparatus to build up cake on the filter leaves using as a filter-aid the final filter sluicings recovered during the filtration of the final liquid product. The defecated dilute sugar-containing solution recovered from the pressure filtration apparatus will have a concentration of approximately 10% Brix and an ash content in the range of from about 4 to 4.5%.

Following defecation, the pretreated sugar solution in this example is evaporated in any suitable manner to produce a sugar liquor having a concentration of from about 40° to 45° Brix.

The condensed sugar liquor is at this point decolorized using granular activated carbon in accordance with the method referred to above. The activated carbon is supplied through a top opening on a single column and thereafter the condensed sugar solution is introduced for filtering upwardly through the carbon which removes the melassigenic constituents and certain other foreign materials contained therein. A commercially available activated carbon which is suitable for the present process is Darco granules. The carbon treated condensed sugar liquor is then passed through a filtration apparatus of the type referred to above to remove the entrained solids. The filtrate is then recycled for clarity.

The sugar solution at this point is introduced into an ion exchange device wherein the ash components of the sugar solution are passed in contact with ion exchange resins in a mixed resin bed. The cation resin can be a monofunctional sulfonated copolymer of styrene and divinylbenzene. A suitable cation resin is sold by Rohm & Haas under the trade name Amberlite IR–120. It has a specific gravity of about 1.3. The anion resin can be a styrene type anion exchanger produced from styrene and divinylbenzene containing quaternary ammonium groups. A suitable anion resin is sold by Rohm & Haas under the trade name Amberlite IRA–401S. The specific gravity of such anion resin is generally about 1.1. The substantial differences in densities between the two types of ion exchangers permit their ready separation either by means of a liquid having a density intermediate thereto, so that one will float and the other will sink or by passing water upwards through a bed of mixed exchangers at such a rate that the quaternary ammonium resin is carried out of the columns whereas the cation exchange resin is not. After demineralization in the manner described above the effluent leaving the column has a pH of 7.0 and an ash content of less than 1 percent. Thereafter, the demineralized and decolorized sugar liquor is condensed by evaporation to produce a purified liquid sugar concentrate product having a concentration of at least 65° Brix and an ash content of less than 1 percent. This concentrate is then treated with powdered activated carbon to control the development of any thermophylic bacteria and colloidal suspensions and the final product separated by conventional filtration techniques.

What is claimed is:

**1.** A process for manufacturing a purified uncrystallized liquid sugar concentrate, consisting of the following sequential steps:

(i) condensing a dilute sugar-containing liquid by evaporation to adjust the concentration of sugar solids in the resultant condensed sugar liquor to a value in the range of from about 30 to 60 weight percent;

(ii) demineralizing said condensed sugar liquor by passing it through a mixed-resin bed;

(iii) condensing the demineralized sugar liquor by evaporation until the concentration of sugar solids is at least 65 weight percent;

(iv) filtering the concentrated liquor; and

(v) separating a liquid sugar product containing at least 65 weight percent of sugar solids having an ash content of less than 1 percent by weight.

2. The process in accordance with claim 1 wherein the process includes as an additional step, decolorizing the sugar liquor while it is at a concentration of from 30 to 60 weight percent sugar solids.

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