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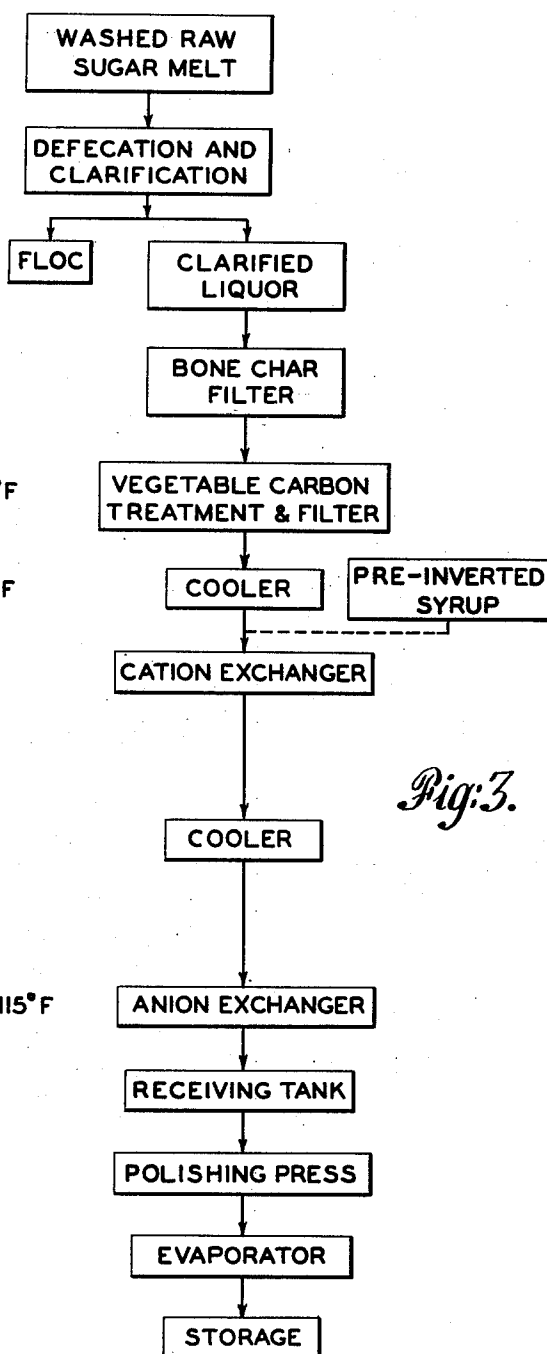
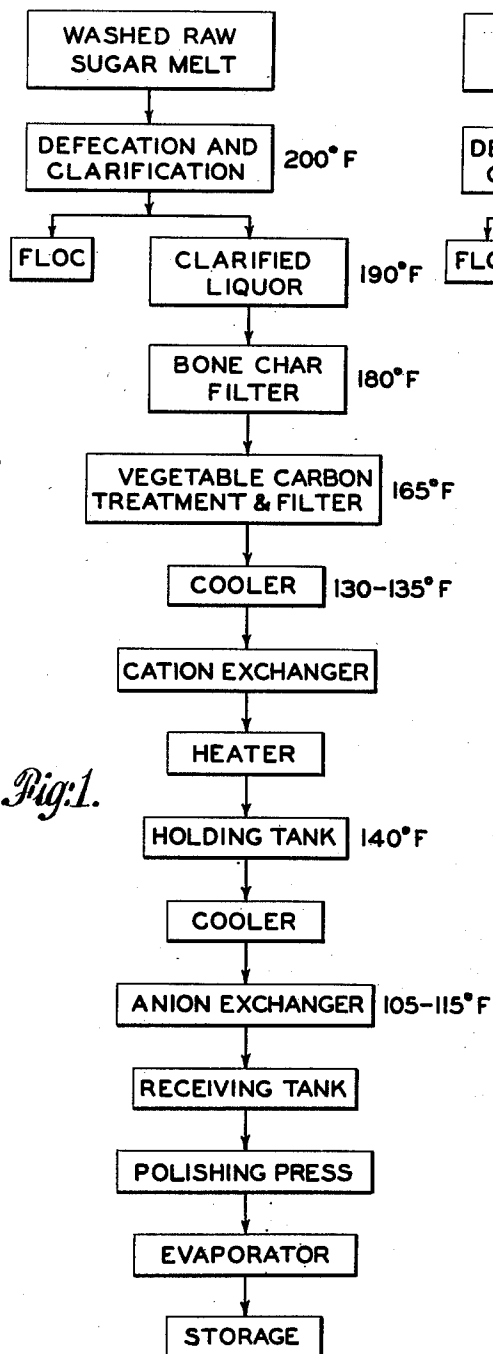
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2,594,440

INVERT SIRUP PROCESS

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2 SHEETS—SHEET 1



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2 SHEETS—SHEET 2

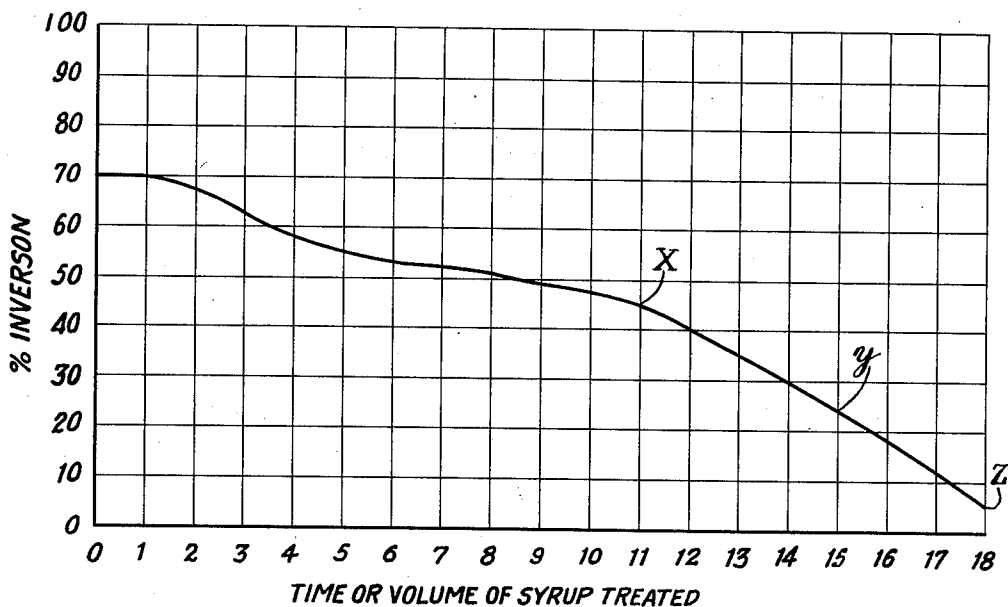


Fig. 2.

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INVERT SIRUP PROCESS

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This invention relates to the preparation of sugar syrups containing both sucrose and invert sugars.

For many uses it is desirable to produce syrups containing substantial proportions of both sucrose and invert sugar which are as water-clear or water-white in color as possible, and which remain stable both as to their sugar content and color upon storage and handling. Such a syrup containing sucrose, dextrose and levulose should be as free from iron as possible and as free from mineral salts as possible in order to have the best stability. Levulose is not particularly stable, and such a syrup containing levulose, particularly if it contains much ash, is apt to develop color upon standing.

Procedures that have been used heretofore for preparing such syrups involve the usual steps in the refining of raw sugar and at a certain stage in the refining process the addition of acid to convert the desired proportion of sucrose to dextrose and levulose. In spite of continued efforts over a period of a great many years, it has not been possible by this procedure to obtain a sucrose-dextrose-levulose syrup that is completely water-white, free from minerals and stable so far as color development is concerned upon subsequent storage and handling.

One object of this invention is to produce such a syrup containing the desired proportion of invert sugar, that has as little color as possible and that is practically free from minerals, particularly iron.

A further object of this invention is the provision of an economical method of accomplishing these results in which the proportion of invert sugar can be controlled at will and the finished syrup is stabilized against the development of color.

Another object of the invention is the provision of an invert sugar syrup that is relatively free from minerals or ash, that has as little color as possible, and that does not increase in color appreciably after storage and handling.

In the drawings, Figures 1 and 3 are flow diagrams illustrating two procedures in accordance with our invention by which a high quality partly inverted sucrose syrup may be made.

Figure 2 is a graphic illustration of the proportion of sucrose inverted by a hydrogen ion exchanger during one cycle of operation.

It has been recognized for some time that ion exchange materials could be used to remove most of the mineral salts from water and various dilute solutions. It has also been proposed to treat

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sugar juices by such ion exchange materials to remove melassigenic salts and increase the recovery of crystallizable sugars. In this connection, it has also been recognized that the treatment of sugar juice with a cation exchange material containing exchangeable hydrogen, particularly when carried out at somewhat elevated temperatures, produces some inversion of the sucrose. Accordingly, on its face, it would seem to be a simple matter to produce a partly inverted sucrose solution free of minerals by the use of ion exchange materials. This has not proved to be the case for various reasons.

In the first place, the proportion of sucrose converted to invert sugar by the passage of a hot sucrose solution through a hydrogen ion exchanger is not uniform throughout a cycle of operation. The ion exchange material appears to act as a catalyst in producing inversion, and Figure 2 illustrates the change in amount of inversion from the time the flow of syrup starts through a bed of freshly regenerated hydrogen ion exchange material until the material reaches the point of exhaustion for all practical purposes. While the actual amounts inverted may vary with different temperatures of treatment and different flow rates, the proportion inverted follows such a curve in each cycle of operation of the exchange material. Other problems in such a process are the treatment by ion exchange of fairly concentrated sugar liquors, and the preparation of a finished syrup that initially is water-white and that remains water-white upon storage and handling.

We have found that, contrary to prevailing views, it is essential in a practical process to decolorize the sugar liquor with carbon or its decolorizing equivalent, and preferably with both bone char and vegetable carbon, before the ion exchange treatment if a stable syrup is to be obtained that contains as little color as possible. By the process of this invention, a water-white syrup can be obtained containing any desired controlled proportion of invert sugar, which syrup is stable as to sugar content and does not develop color appreciably upon subsequent storage or handling.

Our invention is based on the treatment throughout of a high density sugar solution or liquor which is first prepared as a washed raw sugar melt and subjected to the usual defecation, clarification and decolorizing, preferably with both bone char and vegetable carbons. The decolorized liquor is then treated with cation and anion exchange materials in such a manner as to

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control the proportion of sucrose inverted, to produce this inversion without increasing the color of the syrup, to remove minerals from the liquor, and to utilize to best advantage the capacities of the exchange materials. A stable invert syrup practically free from color and ash or minerals can thus be produced which requires a minimum of further processing.

One embodiment of our process, shown in flow sheet form in Figure 1, can be carried out as follows:

A washed raw sugar melt of high density is prepared and subjected to the customary defecation, clarification and decolorizing with carbon, such as by the use of bone char filters and the addition of vegetable carbon. Such a procedure is customarily carried out with the liquor at a temperature of the order of 180-190° F. After the liquor is thus clarified and decolorized with carbon, it is flowed through a bed of cation exchange material regenerated with acid so that it contains exchangeable hydrogen ions, the temperature of the liquor during this treatment being maintained as high as possible without serious injury to the exchange material. For example, it has been found that the liquor can be treated at about 160° F. in the cation exchanger although a temperature of the order of 130-135° F. is safer and therefore preferred.

The liquor coming from the cation exchanger is flowed into one or more holding tanks in which the elevated temperature of the liquor is maintained or increased somewhat. Heat exchangers may be used for this purpose if necessary. The liquor in these holding tanks is practically free of mineral salts but contains the acids corresponding to the salts in the liquor prior to the cation exchange treatment. Consequently, the proportion of invert sugar in the liquor in the holding tanks gradually increases due to the presence of the acids and the elevated temperature. Because the sugar solution in these holding tanks is substantially free of minerals, the inversion of the sucrose in this manner does not increase appreciably the color of the liquor as happens when such an inversion process takes place in the presence of a substantial amount of mineral salts.

As the liquor in each holding tank reaches the desired proportion of invert sugar, the liquor from such tank is cooled and flowed through a suitable bed of anion exchange material to remove free acids from the liquor. By this procedure a constant flow of sugar liquor through both the cation exchange bed and the anion exchange bed can be maintained once the process is started.

Suitable plumbing connections and valves between the cation exchanger, the holding tanks and the anion exchanger may be used to switch the flow to and from the various holding tanks as needed. If necessary, the degree of inversion of the liquor in each tank can be determined by the operator from time to time by any suitable means such as a saccharimeter provided with an observation tube connected by suitable pipes to each tank and arranged so that liquor from each tank can be pumped successively through this observation tube for frequent determination of the degree of inversion in the tanks.

We have also found that the cation exchange material can be utilized advantageously in a fully continuous process by varying the composition of the sugar liquor passed through the cation exchange bed as each cycle of operation progresses. In the curve shown in Figure 2, the proportion of sucrose converted to invert sugar by the cation

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exchange material remains fairly constant at a high level for roughly two-thirds of the cycle, or until the point X on the curve is reached. Thereafter in the cycle as more liquor is passed through the bed, the effluent shows a gradually decreasing amount of invert sugar with a corresponding increase in pH, and gradually increasing amounts of metallic cations until the point Z on the curve is reached when the cation exchange material becomes exhausted for all practical purposes and no longer produces any appreciable inversion of sucrose or removal of minerals.

Another embodiment of our process, therefore, is to utilize the entire cycle which may be accomplished by running the carbon decolorized hot sugar liquor through a cation exchange unit until the point X is reached. At this stage the influent or raw liquor is mixed or blended before entering the exchangers with a quantity of pre-inverted syrup having a substantially higher proportion of invert sugar than is needed in the final product. For example, 2 parts of the raw liquor may at this point be mixed or blended with 1 part of a syrup containing 90% invert sugar and the mixture passed through the exchanger until the point Y on the curve is reached when the proportion of pre-inverted syrup is increased to say 50%. The exact proportions for blending will depend obviously on a number of factors such as the temperature of treatment, flow rate used, composition of the pre-inverted syrup, and composition desired in the finished syrup. The percentages of invert sugar and ash referred to herein are calculated, in all cases, on the basis of the solids content of the sugar liquor or syrup mentioned.

The pre-inverted syrup may be prepared separately in any desired manner as by a conventional treatment with acid or it may be some of the first run of liquor from the cation exchanger that is drawn off and held at an elevated temperature to increase the amount of inversion.

In this procedure also, the liquor from the cation exchanger or exchangers is passed immediately through the anion exchanger without any holding period and after such intermediate cooling as may be necessary to avoid injury to the anion exchange material. Thus, the full capacity of the cation exchange material is utilized in spite of the drop in its catalytic effect to invert sucrose near the end of each run, and the entire treatment is carried out on a fully continuous basis.

In either of the foregoing procedures, the effluent from the cation exchanger will be substantially mineral free up to about the point X on the curve shown in Figure 2 and for the balance of each cycle will begin to show increasing amounts of unconverted mineral salts. If desired, the liquor processed during the first part of each cation exchange cycle can be maintained and handled separately from the liquor processed during the latter part of each cycle, and the two products sold as different grades of invert syrup.

The best mode of operation, however, is to use two or more cation exchange units which may be connected in series so that the raw liquor is passed successively through at least two separate units. By using suitable piping connections, the exchange units may be operated with either one first in the series, or with either one cut out of the circuit for regeneration.

With two or more freshly regenerated cation exchangers connected in series, the first bed or unit would be doing most or all of the inverting of sucrose and conversion of salts to acids dur-

ing the first part of the liquor run. When the first bed or unit becomes partially exhausted, the "breakthrough" point would be reached as evidenced by the presence in the effluent from the first bed of traces of unconverted mineral salts. The first bed would still be capable of doing useful work, however, so that the flow of liquor would be continued beyond this breakthrough point of the first bed for a suitable time, for example as long as the exchange material continues to convert to acids a substantial proportion such as 30-50% of the mineral salts in the raw syrup. Of course, during this phase of operation any subsequent cation exchange unit in the series would produce additional inversion of sucrose and remove any minerals left in the syrup by the partially exhausted unit.

At some such point, however, the first unit would be cut out of the sugar liquor line and be regenerated while the flow of raw liquor is continued to the subsequent exchange unit or units in the series. The freshly regenerated unit would then be connected back in the liquor line as the last unit in the series. Such a procedure can be followed with only two cation exchange units although it will be apparent to those skilled in the art that it would provide a greater factor of safety to use three or more such units in such a series. If a large enough number of cation exchange units are used in series so that at least one unit is always operating in the first portion of the cycle as shown by the curve in Fig. 2, it is possible to produce an invert syrup that has a fairly constant and sufficiently high proportion of invert sugar. In this manner a top grade invert syrup can be produced by a wholly continuous process utilizing most of the capacity of the cation exchange material during each cycle, or in other words reducing the frequency and number of regenerations.

If only one or two cation exchangers are used in series, it may be desirable to increase and control the proportion of invert sugar in the finished syrup by either the holding tank or pre-invert blending procedures described in connection with Figs. 1 and 3. In fact, by using holding tanks between the cation and anion exchange treatments and also adding pre-inverted syrup to the raw sugar liquor going into the cation exchanger, greater flexibility is obtained in the control of the proportion of invert sugar in the finished syrup.

It is also desirable to employ two or more anion exchange units and to operate them in series on the same principle.

In any of the foregoing procedures, the effluent syrup from the anion exchanger is preferably adjusted in pH, passed through a polishing press and then evaporated to the desired high concentration before being pumped to storage.

The liquor passing through the anion exchanger may be at a temperature of the order of 110° F. and does not have to be reheated but may be passed through a filter, such as a polishing press, and then concentrated in a vacuum evaporator, if necessary, without any further heating.

The density of the sugar liquor thus treated is not particularly critical although the higher it is, the less work will have to be done at the last stage in the evaporator. On the other hand, the liquor is more easily processed in the ion exchangers if it is not too concentrated. As a compromise, a sugar liquor having a density of about 60° Brix has been found suitable for treatment by the procedures of this invention.

It is desirable to adjust the pH of the syrup promptly after it comes from the anion exchanger in order to improve its stability. The first portion of the effluent from the anion exchanger, particularly if that material is regenerated with a solution of caustic soda, or a similar strongly alkaline reagent, is apt to be distinctly alkaline. If the syrup is left in this condition, the levulose in the syrup is affected adversely. Subsequent portions of the anion exchange effluent have a lower pH and may be blended with the first portion if this can be done without too much delay and produces a sufficiently low pH in the blend. Otherwise, it is desirable to stabilize the syrup by adding a small amount of acid, such as hydrochloric acid, to reduce the pH to 6.5 or less. A pH of about 3 to 6 is satisfactory. This may be done while the syrup is in the receiving tank referred to in the flow diagrams in Figures 1 and 3. If the finished syrup is not made acid enough, the levulose is not properly stabilized, and the color of the finished syrup is affected adversely if such a pH adjustment is not made. On the other hand, an invert syrup of the purity obtained by the procedure of our invention even with its pH lowered to 4 to 5 is a substantially unbuffered system and may be handled and stored in iron equipment without adverse effects.

By this procedure, an invert syrup containing at least about 60% solids and 30 to 75% of the sugars present as invert sugar can be prepared with much less color and ash than has been commercially possible heretofore. The ash content may easily be held down to a maximum of 0.08% of the total solids in the syrup and preferably does not exceed 0.06% of the total solids.

The color, as determined on a standard scale which is referred to herein as the "caramel scale," may be easily held down to a maximum of 0.6, and preferably does not exceed 0.4. This color of the syrup is determined by comparing a sample in a 2 oz. French, square cross section ($1\frac{7}{8}$ " x $1\frac{7}{8}$ " outside dimensions) water sample bottle made of colorless glass, with a set of color standards ranging in value from 0.1 to 10 or higher. These standards are prepared as follows: The basic or number 10 standard is prepared by making up a caramel solution of such strength that 10 ml. of solution diluted with distilled water to 100 ml. matches the combined 30 yellow and 6.2 red color glasses of the well known Lovibond scale when superimposed on each other and held against a water sample bottle containing distilled water. The other standards from 0.1 up to 10 are then made up by diluting smaller amounts of the same caramel solution to 100 ml. with distilled water. Thus, the 0.4 color standard contains 0.4 ml. of the caramel solution diluted to 100 ml., the #1 standard contains 1 ml. of the caramel solution, etc. The color of a liquid determined on this scale is calculated on the basis of solids content.

By way of example, it has been found that the process of this invention makes it possible to produce on a commercial scale an invert syrup containing about 76% solids with around 50% invert sugar based on total solids that consistently has a color on the above scale of not more than 0.2 or 0.3 and an ash content of 0.002% or in any event less than 0.01% of the total solids in the syrup.

It has been found that various cation exchange materials, as long as they are organic in nature and capable of being regenerated with acid, can be used in the cation exchanger. It is also desir-

able, of course, to employ a cation exchange material that is not deteriorated by or sensitive to the elevated temperatures of the liquor passing through the cation exchanger.

Various anion exchange materials may also be used. While such materials have been referred to as anion exchange materials, it is not clear whether they operate by producing a true exchange of hydroxyl ions for the ions of the acids in the liquor, or whether they operate by absorbing the entire acid molecules from the liquor. In either case, however, the net result is the removal of free acids from the sugar solution of liquor.

It has been found that it is most important to carry out the ion exchange treatment after the sugar liquor has been clarified and decolorized with bone char and/or vegetable carbon, or equivalent decolorizing material, if a stable clear water-white syrup of the best quality is to be obtained. An ion exchange demineralizing and inversion process applied to sugar liquor before the color is removed with the carbon produces a much darker syrup. By the treatment of a "decolorized" or "carbon decolorized" sugar liquor is meant a liquor which has had its color reduced to the order of 0.8 to 1 on the caramel scale before the liquor is subjected to the ion exchange treatment.

Also, a particular advantage of the process of this invention is that the inversion takes place while the syrup is practically free from cations. When inversion is carried out in the presence of substantial quantities of minerals, for example, around 300 P. P. M. of inorganic salts, enough color is produced in the finished syrup to be very noticeable. Also, of course, the anion exchange treatment avoids the necessity of adding alkali to neutralize some of the acid used in the conventional method of producing an invert syrup. The addition of substantial amounts of such alkali in the usual process has the effect of increasing color in the syrup, apparently due to some type of indicator action and decomposition of levulose at high pH. By the process of our invention the sugar solution is not held at elevated temperatures for any substantial period of time after inversion which also is important in avoiding development of color.

By the foregoing procedures, not only is a substantially mineral free syrup produced, but the relative proportions of sucrose and invert sugars can be controlled at will and maintained constant within reasonable limits. In addition, such a syrup can be produced which for the first time is water-white, or free of objectionable color, and is stable under ordinary conditions of storage and handling.

The terms and expressions which have been employed herein are used as terms of description and not of limitation and there is no intention in the use of such terms and expressions of excluding any equivalents of the features shown and described or portions thereof, but it is recognized that various modifications are possible within the scope of invention claimed.

We claim:

1. A process of preparing a clear substantially water-white demineralized syrup containing sucrose and invert sugar which comprises subjecting a high density sugar liquor to clarification and carbon decolorizing, thereafter passing such liquor at an elevated temperature through an acid regenerated cation exchanger to convert salts therein to acids and to invert sucrose, cooling the liquor thus obtained, and passing the

cooled liquid through an anion exchanger to remove free acids.

2. A process of preparing a clear substantially water-white demineralized syrup containing sucrose and invert sugar which comprises subjecting a high density sugar liquor to clarification and carbon decolorizing, thereafter passing such liquor at an elevated temperature through an acid regenerated cation exchanger to convert salts therein to acids and to invert sucrose, cooling the liquor thus obtained, passing the cooled liquor through an anion exchanger to remove free acids, and adjusting the pH of the treated syrup to the range of about 3 to 6.5.

3. A process of preparing a clear substantially water-white demineralized syrup containing sucrose and invert sugar which comprises subjecting a clarified, carbon decolorized high density sucrose liquor to treatment at an elevated temperature with an acid regenerated cation exchange material to convert mineral salts in the liquor to their corresponding acids, holding the effluent liquor thus obtained at an elevated temperature until the desired proportion of sucrose has been converted to invert sugar, then cooling the liquor and immediately treating it with an anion exchange material to remove free acids therefrom.

4. A process of preparing a clear substantially water-white demineralized syrup containing sucrose and invert sugar which comprises flowing a clarified carbon decolorized sucrose liquor at an elevated temperature through a series of beds of cation exchange material charged with exchangeable hydrogen, collecting the liquor thus treated in a container, holding the liquor in the container at an elevated temperature until the desired proportion of sucrose has been converted to invert sugar, and then flowing the liquor from said container through a bed of anion exchange material to remove free acids therefrom.

5. A process as defined in claim 3 in which the liquor after treatment with the anion exchange material is adjusted to a pH of about 4 to about 6.

6. A process as defined in claim 4 in which the liquor after treatment by the anion exchange material is adjusted to a pH between about 4 and about 6.5.

7. A process of preparing a clear substantially water-white demineralized syrup containing sucrose and invert sugar which comprises passing a hot clarified, carbon decolorized sucrose liquor through a bed of acid regenerated cation exchange material, blending with the raw liquor to be thus passed a quantity of pre-inverted syrup when the exchange material becomes partly exhausted, cooling the effluent from the cation exchange bed, and passing said effluent through a bed of anion exchange material.

8. A process of preparing a water-white syrup containing sucrose, dextrose and levulose which comprises flowing a heated high density carbon decolorized sugar liquor through a bed of acid regenerated cation exchange material until the effluent shows a substantial drop in invert sugar content, then blending with the liquor to be flowed therethrough an appropriate quantity of pre-inverted syrup containing a high proportion of invert sugar to produce an effluent of the desired invert sugar content, holding the effluent from the cation exchange bed to further adjust the proportion of invert sugar, and passing said cation exchange bed effluent through an anion exchanger to remove free acids.

9. A process of preparing an invert sugar syrup which comprises subjecting a high density melt

of raw sugar to defecation, clarification and decolorizing with carbon, flowing the sugar liquor thus obtained through an acid regenerated cation exchange unit to convert salts in the liquor to their corresponding acids and to invert a portion of the sucrose in the liquor, adjusting the proportion of invert sugars in the effluent from said units as the flow proceeds in order to maintain the ultimate proportion substantially constant, and flowing the invert sugar liquor thus obtained through an anion exchange unit.

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