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V. E. HAURY

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SUGAR MANUFACTURE BY ALCOHOL EXTRACTION

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FIG. 1

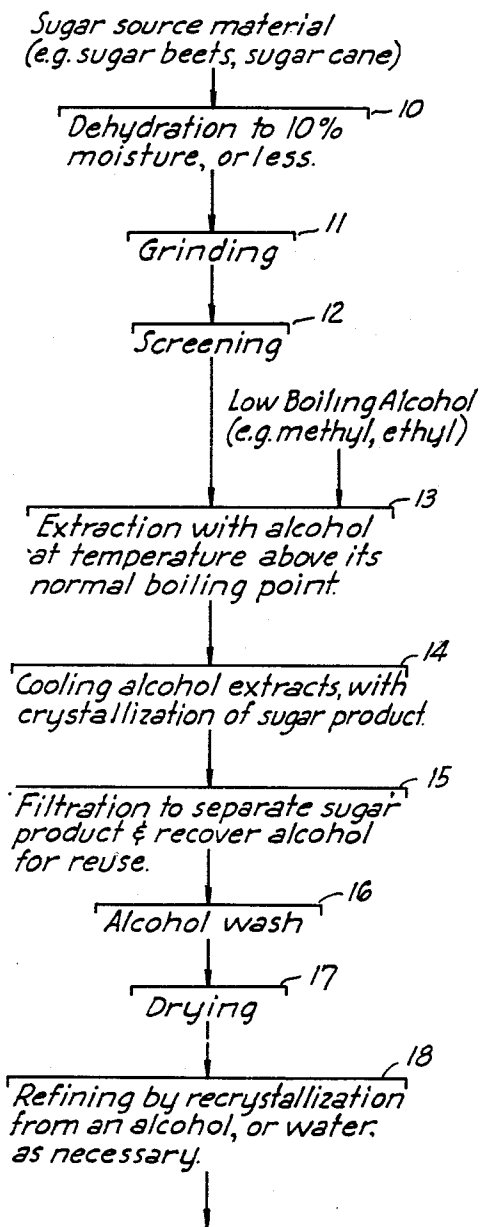
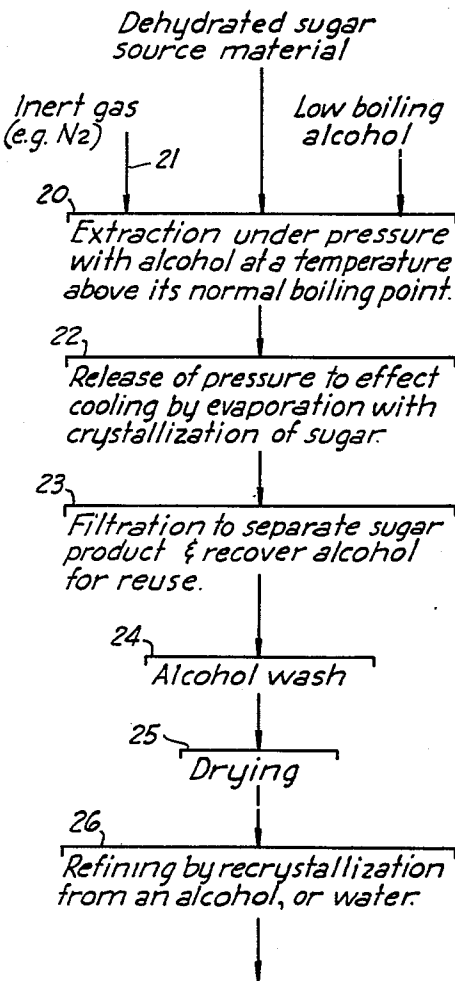


FIG. 2



INVENTOR.

Vernon E. Haury

BY

Fleishman Swain

ATTORNEYS

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2,943,004

SUGAR MANUFACTURE BY ALCOHOL EXTRACTION

Vernon E. Haury, Santa Susana, Calif., assignor of one-half to Simco Incorporated, San Francisco, Calif., a corporation of California

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17 Claims. (Cl. 127—58)

This invention relates generally to the manufacture of sugar, and its derivatives. More particularly, it pertains to improved methods for the extraction of sugar from raw sugar stock such as sugar beets, sugar cane and the like.

In conventional sugar manufacture, as in processing sugar beets, the fresh beets are washed and sliced into long narrow strips called "cossettes." These cossettes are dropped into large tanks or cells, with as many as twelve to fourteen being connected in series to form a "diffusion battery." The sugar is extracted in these diffusers with a counter current stream of hot water. The dark colored solution obtained from the diffusers (containing about 10 to 12% sucrose) is treated with various reagents such as lime, carbon dioxide, sulphur dioxide and decolorizing carbon in order to remove some of the impurities from the solution. The solution is then filtered and concentrated to about 55% sugar in multiple-effect evaporators. This concentration is necessary to subsequent graining or crystallization of the sugar in vacuum pans, and is generally quite prolonged as it is necessary to boil off all the water added in the extraction step as well as that originally present in the fresh beets. It is also wasteful of heat because of the high heat of vaporization of the water solvent employed.

It is well known that the distillation of a low boiling alcohol, such as ethyl alcohol or methyl alcohol, requires less than half the heat required to distill an equal volume of water. Accordingly, if either ethyl alcohol or methyl alcohol could be used to extract the sugar instead of water, a considerable saving in heat requirements could be effected. On the other hand, I have found that extraction at atmospheric pressure even with boiling alcohol yields a solution containing only a small amount of sugar.

In general it is an object of the present invention to provide a novel method for the manufacture of sugar in crystalline form, employing low boiling alcohols such as methyl or ethyl alcohol.

Another object of the invention is to provide a method of the above character by which improved yields as well as simplified recovery of crystalline sugar can be attained, with reduction of cost.

Other objects and advantages of the present invention will appear from the following description and from the drawings in which:

Figure 1 is a flow sheet illustrating a general step employed in carrying out my invention; and

Figure 2 is a flow sheet illustrating a modified version of the method.

In accordance with my invention, sugar is manufactured by first substantially dehydrating sugar source material, preferably to a moisture content of no greater than about 10% by weight, and then extracting the dehydrated source material with an alcohol at a temperature at least 10° above its normal boiling point, but below temperatures that will tend to darken the result-

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ing sugar product. Preferably the extraction step is carried out in a closed system under autogenous pressure and within a temperature range of from about 100° to 150° C., and in no event at temperatures higher than about 200° C. I also prefer to carry out the extraction step with a proportion of alcohol about two to ten times the weight of the dehydrated sugar source material (optimum about 5:1). Thereafter the alcohol extract is cooled at a rate determined by the desired crystal size of the resulting sugar product. The crystalline product can be recovered by filtration, washed with additional alcohol, and dried to a final product.

When using a preferred proportion of about five parts by weight of alcohol to one part of dehydrated sugar source material, a maximum sugar content in the extraction step is reached in about five minutes within the preferred temperature range. The extraction solution is light colored and produces a practically white final crystalline product. Longer heating and higher temperatures tend to yield extraction solutions of darker color. A practically complete removal of sugar can be accomplished with about three extractions. The resulting solutions are very easily filtered, and the flow of solution through the filter is not retarded by the presence of fines.

While it is possible to use various source materials as will be presently explained, the process of the present invention is particularly adaptable of the manufacture of sugar from sugar beets. Assuming the use of sugar beets, the method can be carried out substantially as illustrated in Figure 1. Step 10 illustrates the dehydration of the washed fresh beets, for example in a continuous rotary dryer. Complete dehydration is not essential but I find that my process is most efficient when the water content is reduced to about 10% or less. For rapid extraction the dehydrated beets may be ground as at 11 to provide a desired initial particle size as determined in step 12. For example, screening to provide material capable of passing through an 8-mesh screen has proved advantageous.

In step 13, the ground dried beets are extracted by heating with an alcohol at a temperature above the normal boiling point of the alcohols added. The alcohol and dried beets can be heated in a closed system, such as an autoclave, and under their own pressure, to a temperature between about 90 to 200° C. for ethyl alcohol, and between about 75° to 200° C. for methyl alcohol. As previously indicated, a preferred temperature range for both alcohols is about 100–150° C. As will appear, the pressure within the closed system can advantageously be increased by the addition of an inert gas.

After the extraction step, the alcohol extracts are cooled to effect crystallization of the sugar, as indicated at 14. Most of the sugar separates rapidly as the solution is cooled, with the size of the crystals being determined by the rate of cooling. Rapid cooling produces small crystals whereas larger crystals are obtained by slower cooling.

After cooling, the alcohol extracts can be subjected to filtration in stage 15 to recover the sugar product. Conventional filtration equipment can be employed. Thereafter the crystalline sugar product can be washed with additional alcohol, as at stage 16, and dried at 17 to produce a substantially white final product. This product can be used without further treatment for many industrial uses, for example, in the preparation of non-ionic detergents, or it can be further refined as in step 18. One means of refining is by recrystallizing from an alcohol or, if desired, the sugar may be recrystallized from water.

The processing in accordance with Figure 1 is subject to a number of variations. For example, the inclusion

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of decolorizing carbon in the extract mixture will yield a somewhat lighter solution. Decolorizing carbon can also be used in the refining step 18. It is also desirable to carry out the extraction step 13 in an acid-free solution. Thus it is contemplated that lime can be added to neutralize the solution. Moreover, it has been determined that the solution will have a slightly lighter color if about one part of lime is added to about a thousand parts of dried source material before extraction, although increased amounts of lime may result in a dark colored solution.

A further modification is illustrated in Figure 2. In this modified process, sugar source material is dehydrated as before, and is added to the extraction step 20. Extraction is carried out under positive pressure as determined by the introduction of an inert gas, such as nitrogen, at stage 21. Cooling is then accomplished at 22 by release of pressure to effect cooling by evaporation, and consequent crystallization of the sugar. As in the process of Figure 1, crystal size depends upon the rate of cooling, which in this instance is determined by the rate of vapor release. The released alcohol vapors can be liquefied by compression and returned to the extraction process as desired. Further processing of the sugar in steps 23 to 26 can be substantially as in the process of Figure 1.

In either of the processing procedures described above, the alcohol extract from the filtration step can be recycled, that is, it can be used again without distillation for further extractions. The alcohol can be recycled in this manner several times. In fact, it appears that the extraction efficiency is actually greater on the first recycle than during the initial extraction, and no apparent decrease in efficiency is noted even after the second recycle. After repeated recycling, the concentration of colored materials including molasses increases somewhat. Although these colored impurities can be substantially removed by treating the discolored solvent with discolorizing carbon, it is preferred to eventually distill the alcohol for reuse.

It is to be noted that the processes as described above have related primarily to extraction with a single low boiling alcohol. It should be understood however that the invention is not limited to the use of a single alcohol in the extraction steps, and that a mixture of two or more alcohols (such as a mixture of methyl and ethyl alcohols) can be employed without material variance from the principles of the invention. Also, the lower boiling alcohols can be diluted with up to about an equal volume of other alcohols such as the propyl and butyl alcohols, ethylene glycol and the like. Other diluents such as acetone, benzene, ethyl acetate and carbon tetrachloride can also be used. These diluents generally decrease the solubility of sugar in the mixed solvent but in some cases result in a more complete separation of the dissolved sugar on cooling.

The processing in accordance with the invention has many advantages. For example, the dehydration of the beets and cane before extraction permits substantial economies in both processing and transportation as a result of the volume and weight reduction made possible. In conventional refinery areas, the dehydrated granular materials can be transported to the extraction stages by pneumatic conveyors or similar means. Moreover, the dried beets and cane can be stored for a considerable time without deterioration, making possible year round operation of the plant instead of the normal seasonal operation. In addition, new-type plants can be constructed to operate economically on a much smaller scale than conventional plants. When combined with the water and fuel saving, this fact can permit such plants to be built in the tropics right alongside the raw sugar mills, and eliminate the costs normally arising from sacking of the raw sugar and transporting it to the refinery.

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Of even greater importance is the fact that the manufacture of the sugar itself can be carried on in a much simplified manner that is not only more economical but also more efficient. By using alcohol as a solvent instead of water, heat requirements for distillation can be cut by at least 50%. Finally the processing can be carried out more rapidly with alcohol as a solvent than with water as a solvent.

The many advantages of the invention are exemplified in the following specific examples of its practice:

EXAMPLE 1

A series of sugar beet extractions were carried out at varying temperatures, and for different periods of time, as indicated in Table I below. For each of these extractions, one hundred parts by weight of beets were first prepared by drying at 110° C. and then grinding to pass through an 8-mesh screen. This quantity of dehydrated, screened beet stock was then extracted with five hundred parts by weight of methyl alcohol under autogenous pressure in an autoclave, and at the temperatures and times shown in the table. The hot alcohol and extracted sugar were then withdrawn from the autoclave through a stainless steel filter screen and passed through a water cooled condenser. The cooled solution was maintained at a temperature of about 15 to 20° C. for about two hours. Most of the sugar crystallized immediately. Thereafter the crystallized sugar was filtered, washed with methyl alcohol, and dried. Parts by weight of sugar recovered in each of the extractions is set forth in the table.

Table I

Extraction temperature, ° C.	Time at extraction temperature, min.	Sugar yield, parts by weight
65	30	less than 2.
70-75	30	13.
90	5	29.
90	20	37.
110	5	37.
130	5	39.
150	5	37.

EXAMPLE 2

One hundred parts by weight of oven dried beets were prepared as in Example 1, but without grinding or screening. This dehydrated beet stock was heated in an autoclave to 110-115° C. for five minutes in the presence of five hundred parts by weight of 95% ethyl alcohol. The hot alcohol and extracted sugar was separated from the beet pulp and cooled, as in Example 1. The yield by this process was 28 parts of crystalline sugar.

EXAMPLE 3

A quantity of beets were dried at 110° C. and ground substantially as in Example 1. This beet stock was then divided for a series of tests in which a portion A was used in the ground dehydrated state, and a portion B was moistened with about 11% by weight of water. Thereafter identical tests were made on the separate portions of the beet stock. The first extract was made with distilled methyl alcohol in the proportion of 500 parts by weight of alcohol to 100 parts by weight (on a dry basis) of beet stock. After cooling and filtering the alcohol extract to separate the crystallized sugar, the filtrate was made up to the original volume of methyl alcohol with fresh alcohol. This filtrate was then recycled for use as a solvent for the extraction of another sample of the dried beets A and moistened beets B. The filtrate from this series of extractions was recycled again in similar fashion. The weights of sugar recovered by cooling and filtering these extracts are shown in

Table II below:

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Table II

	Sugar yield, parts by weight	
	Series A 100 parts of dried sugar beets	Series B 111 parts of dried sugar beets con- taining about 10% water
First extract.....	40	31
First recycle.....	43	36
Second recycle.....	39	34

EXAMPLE 4

Crystalline sugar obtained from extractions such as described in the foregoing examples was recrystallized by dissolving 50 parts of the sugar in 500 parts of methyl alcohol at 110° C. Upon cooling the solution, an average of 43 parts of white sugar crystallized readily. Upon filtering the recrystallized sugar from the alcohol extract solution, additional sugar was recovered by distillation to further concentrate certain of the solutions. In other instances the filtrate was used in the initial extraction of sugar from dried sugar beets with recoveries similar to those obtained in Example 1.

EXAMPLE 5

To demonstrate the use of mixed solvents, sugar was extracted with a mixture of 200 parts of methyl alcohol and 200 parts of benzene at 120° C. On cooling the solution 12 parts of crystalline sugar was separated. The residue after evaporation of the solvent was less than one part.

Particular reference has previously been made to sugar beets as the principal source material for manufacturing sugar in accordance with the invention. It should be understood that good results may be obtained using almost any sugar source material. For example, sugar cane, sorghum plant, or other source material may be used. It should also be understood that the present invention is not limited to a batch process such as described, but may be carried out in continuous or semi-continuous manner.

I claim:

1. In a process for the manufacture of sugar from sugar beets, sugar cane and the like, the steps of substantially dehydrating sugar source material, extracting said dehydrated source material with a low boiling alcohol at a temperature at least 10° C. higher than the normal boiling point of such alcohol but below about 200° C., cooling the alcohol to effect crystallization of sugar, and separating the crystallized product.

2. The process of claim 1 wherein said alcohol is ethyl alcohol.

3. The process of claim 1 wherein said alcohol is methyl alcohol.

4. The process of claim 1 wherein said dehydrated source material is ground and screened prior to the extraction step.

5. The process of claim 1 wherein the extraction with

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alcohol is carried out at a temperature between about 100 and 150° C.

6. The process of claim 1 wherein the extraction with alcohol is carried out under autogenous pressure in a closed system.

7. The process of claim 1 wherein said extraction with alcohol is carried out at a pressure greater than atmospheric.

8. The process of claim 1 wherein said extraction with alcohol is carried out in the presence of decolorizing carbon.

9. The process of claim 1 wherein said extraction with alcohol is carried out under slightly alkaline conditions.

10. The process of claim 1 wherein said cooling is accomplished by release of pressure to effect cooling by evaporation.

11. The process of claim 1 wherein said crystallized product is separated by filtration.

12. The process of claim 10 wherein said crystallized product is refined by recrystallization from a solvent for such product.

13. The process of claim 10 wherein the alcohol filtrate is recovered and re-used in the extraction step.

14. In a process for the manufacture of sugar from sugar beets, sugar cane and the like, the steps of dehydrating sugar source material to a moisture content of no greater than about 10% by weight, contacting the dehydrated source material with from 2 to 10 times its weight of a low boiling alcohol, heating said alcohol and sugar source material in a closed system to a temperature at least 10° C. higher than the normal boiling point of such alcohol but below about 200° C., cooling the alcohol to effect crystallization of extracted sugar, filtering the crystallized sugar from the alcohol extract, washing said crystallized sugar with alcohol, and drying the crystallized sugar to a final product.

15. In a process for the manufacture of sugar from sugar beets, sugar cane and the like, the steps of substantially dehydrating sugar source material, extracting said dehydrated source material with a mixture of solvents including a low boiling alcohol and at a temperature at least 10° C. higher than the normal boiling point of such alcohol but below about 200° C., cooling the mixture of solvents to effect crystallization of sugar, and separating the crystallized product.

16. The process of claim 15 wherein said alcohol is ethyl alcohol.

17. The process of claim 15 wherein said alcohol is methyl alcohol.

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