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## PROCESS OF PRODUCING DEXTROSE.

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The invention relates to a process of producing dextrose (grape sugar) from starch, and is more particularly concerned with the crystallization of the dextrose in the sugar liquor and the subsequent separation of the sugar crystals from the mother liquor.

In the manufacture of dextrose from starch, the starch is first converted into a sugar solution by hydrolysis, that is, by treating it with hydrochloric or sulphuric acid according to well-known methods. The sugar solution is then neutralized and filtered through bone-char. It is then introduced into a vacuum evaporator and brought to the proper consistency or density, usually 37° to 42° Baumé (the density being based upon the temperature of 100° Fahrenheit, as is usual in the art). The concentrated sugar liquor is dropped from the vacuum pan into a tank or crystallizer and slowly agitated. To effect a more speedy crystallization of the dextrose in the sugar liquor it is customary to place in the crystallizer fully developed dextrose crystals taken from a preceding run or batch. These are called seed crystals. In practice the seed crystals are usually left in the crystallizer. After the crystallization has been carried as far as practicable, the magma (consisting of the crystals and mother liquor) is fed into a centrifugal to separate the crystals from the mother liquor, or hydrol as it is termed. The sugar crystals may be further treated, as by washing, drying and otherwise, before they are placed on the market.

Dextrose crystallizes in two forms:—The hydrate crystals (containing water of crystallization) have a plate-like form and are known as "plate" crystals. The anhydrous crystals (free from water of crystallization) have a needle-like form and are known as "needle" crystals. The hydrate crystals develop at the lower temperatures (at about 105° F. and lower) and the anhydrous crystals develop at the higher temperatures (above 105° F.).

Considerable difficulty has heretofore been experienced in separating the crystals from the hydrol. The crystals frequently mat or agglutinate in the centrifugal, building up an impermeable layer around the interior of the basket and so preventing the passage of the hydrol therethrough. Many theories have been advanced in the literature on the art to account for this matting of the crystals in the centrifugal and many expedients suggested to obviate it. It seems to be quite universally agreed at the present time that the matting of the crystals could be effectively prevented if relatively large crystals of uniform size could be produced during the crystallizing step of the process. Up to the time of the present invention, however, so far as I am aware (and I am familiar with most, if not all, of the better known processes), no process has yet been proposed which uniformly produces crystals of such size that they can be easily and quickly separated from the hydrol. When the centrifuging is easily and quickly effected, the magma is called free spinning, and the magma is free spinning when the crystals are uniformly of proper size, the larger the individual crystals the better.

It is a commonly accepted theory at the present time that satisfactory (that is, complete) separation of the dextrose crystals from the hydrol can only be effected if the dextrose is in either its hydrate or anhydrous form. It is stated in so many words in the literature on this art that it is not possible to separate the dextrose from the hydrol if the dextrose has been crystallized into both its hydrate and anhydrous forms and both forms of crystals are present in the magma. I have found, however, contrary to this assertion, that a mixture of both hydrate and anhydrous crystals are easily and quickly separated from the hydrol if only the crystals are of proper size. In other words, the essential factor in producing a free spinning magma is the size of the crystals, regardless of whether they exist in the hydrate or anhydrous form, or as a mixture of both. It is not only essential to produce large crystals but it is also essential that the crystals be as nearly uniform in size as possible. If some of the crystals are large and others small, it is obvious that the smaller crystals will tend to fill in the interstices between the larger crystals and so form impermeable patches.

The object of the present invention is to provide an improved process of treating the sugar liquor so as to produce dextrose crystals of such size and uniformity that the centrifuging can be more uniformly easily and quickly performed than has been possible in the processes heretofore proposed or practiced. The construction and arrange-

ment of the apparatus employed in practicing the present process are as usual and need no particular illustration or description. The starch is converted by acid hydrolysis and the sugar solution (after neutralizing and filtering) is brought to the proper density, 37° to 42° Baumé. All these preliminary steps are performed in the usual and most approved manner. At this point the present process departs from the prior processes.

According to the present invention the sugar liquor dropped from the evaporator is cooled for a preliminary seeding to induce an incipient crystallization in the sugar liquor. For this purpose the sugar liquor is fed into a cooling tank provided with an agitator which serves to bring all parts of the liquor in contact with the cooling surfaces of the tank. The sugar liquor enters the cooler at a temperature ranging from about 132° F. to 140° F. The temperature is quickly brought down to 120° F. Economy of operation is furthered by performing as quickly as possible those steps of the process in which time is not essential for chemical or physical purposes. When the sugar liquor has a temperature of about 120° F., incipient crystallization is induced in the mass by introducing dry seed crystals derived from a previous run. It has been found that about 200 pounds of dry seed crystals are sufficient to induce incipient crystallization in about 11,800 pounds of sugar liquor. The dry seed consists of both hydrate and anhydrous crystals. Since, however, the hydrate form of crystal is understood not to develop at 120° F. in a liquor in which no crystallization has been induced, it is believed that only the anhydrous form of crystal grows at this stage of the process, although it will be obvious to those skilled in the art that this is difficult of exact determination and verification.

The dry seed crystals are added to the sugar liquor while the latter is at a temperature of approximately 120° F., because it has been found that if the sugar liquor is substantially hotter than 120° F., the seed crystals will go into solution so quickly as to be ineffective in inducing crystallization, whereas if the sugar liquor is substantially colder than 120° F., when the seed crystals are added, the seed crystals tend to agglomerate and so lose their effectiveness in inducing the growth of crystals in the liquor. Thus in order that the dry seed crystals may prove most effective in starting the formation of crystals in the sugar liquor the latter must be of the proper temperature, neither so hot as to completely dissolve the seed crystals, nor so cold as to cause them to agglomerate. After the dry seed crystals have been added the sugar liquor is gradually cooled down to 100° F., a temperature more favorable for the development of the hydrate crystals. It will be understood that when conditions are favorable the step of inducing incipient crystallization may be omitted, the temperature of the sugar liquor being reduced in the cooler without the addition of the dry seed crystals. However, I have found that more satisfactory final results are obtained by this preliminary crystallization step.

From the cooler the sugar liquor is piped to a crystallizer in which there is a quantity of crystals left from the preceding run or batch. These crystals are known as wet seed crystals, because they have not been separated from the hydrol. It is considered desirable in order to effect the most rapid crystallization to leave in the crystallizer from the preceding run an amount of crystals approximating from 40% to 50% of the incoming sugar liquor.

The crystallizer is provided with coils or other means whereby the temperature of the contents of the crystallizer may be regulated. In passing through the pipes from the cooler to the crystallizer the sugar liquor loses a few degrees of temperature and for that reason it is not considered desirable to reduce the temperature of the sugar liquor to much below 100° F. before the liquor leaves the cooler. When the sugar liquor has been conducted to the crystallizer its temperature is still further reduced and is brought to the point most favorable for the rapid growth of the crystals. This temperature has been found to range from about 90° F. to 95° F. The sugar liquor, or magma as it may now be termed, is maintained at this temperature for about 16 hours, during which time crystallization has progressed to such an extent that the liquor is permeated with the larger seed crystals and the smaller new crystals, whose growth has been induced and hastened by the presence of the seed crystals.

In order to hasten still further the growth of the crystals, the crystallizer is provided with a slowly revolving agitator by which the magma is stirred up and the seed crystals evenly distributed throughout the mass. By distributing the seed crystals uniformly throughout the liquor the uncrystallized portions thereof are brought into intimate contact with the seed crystals and thus the growth of the new crystals is more quickly induced. Moreover, by slowly stirring the magma all parts thereof are brought to a uniform temperature by coming in contact with the surfaces of the crystallizer by means of which its temperature is regulated.

When the magma in the crystallizer has been maintained at a temperature of about 90° F. to 95° F. for about 16 hours so that the liquor is permeated with crystals, the temperature of the magma is raised to a

degree ranging from about 110° F. to 120° F. and is maintained at this elevated temperature for a period ranging from about 5 to 8 hours. By thus raising the temperature of the magma after crystallization has progressed to a considerable extent therein, the more minute crystals are caused to go into solution again. Incidentally the larger crystals are somewhat reduced in size. The degree to which the temperature of the magma is raised and the length of time it is maintained at this temperature will, of course, depend upon the character of the batch. The object of thus raising the temperature of the magma after crystallization has begun is to get rid of the more minute crystals and leave in the liquor the larger crystals so that they may become nuclei of still larger crystals when the magma is again reduced in temperature for the final crystallizing step of the process.

When the more minute of the new growth of crystals have been dissolved by raising and keeping the magma at the elevated temperature, the magma is again reduced to the temperature most favorable for the rapid growth of crystals, namely, 90° F. to 95° F. Reducing the temperature of the magma is accomplished gradually in about 3 to 5 hours. The undissolved crystals now tend to build up rapildy into relatively large crystals of uniform size. The time necessary for the recrystallization of the dextrose in the liquor is about 12 hours. It will thus be recognized by those skilled in the art that the recrystallization or final crystallizing step, as it may be called, is accomplished in a relatively short time. The crystals produced are of such magnitude and of such uniformity of size that the hydrol can be easlily and quickly separated from the crystals.

After recrystallization has been effected, two-thirds of the magma is drained off and passed into the centrifugal machine by which the crystals are separated from the hydrol. The remaining one-third of the magma serves as seed crystals for the incoming batch of sugar liquor from the cooler. When the crystals have been freed from the hydrol they are washed while still in the centrifugal by passing a stream of water through them which completely washes out any hydrol which may remain adhering to the crystals. After the crystals have been washed they are then passed through a dryer. The dried crystals discharged from the dryer are then packed for market in the usual manner.

The great desideratum in the present devolopment of the various processes of making crystallized dextrose is to produce a free spinning magma, that is, a magma in which the sugar crystals can be easily and quickly separated from the hydrol or mother liquor. The crystals produced by the practice of the present process are relatively large and of such uniformity of size that the crystals are quickly and easily separated from the hydrol when the magma is placed in a centrifugal machine. If the operator carefully follows the steps of the process outlined above, he is assured of uniformly producing a free spinning magma. The object of the invention being to produce large crystals of uniform size and it having been found necessary to employ both the elevated temperatures and the lower temperatures, the finished product will be found to consist of both hydrate and anhydrous crystals. This fact has been established by both microscopic examination (at 800 diameters) and by several independent, but corroborating chemical analyses. Moreover, the crystals range between 99.5% and 100% in pure dextrose, as determined by chemical analysis of each run, as is customary in the art.

Although the present invention has been described as particularly applicable to the process of producing crystallized dextrose from sugar liquors derived from converted starch, it will be understood that the invention is not to be restricted to this purpose, inasmuch as it is applicable to the production of crystallized dextrose from sugar liquors however derived.

Having thus described the invention what I claim as new is:

1. The process of producing crystallized dextrose from sugar liquor derived from converted starch which consists, in bringing the sugar liquor to a density of about 37° to 42° Baumé, reducing the temperature to substantially 120° F., adding dried seed crystals, reducing the temperature to about 100° F., introducing the liquor into a crystallizer containing wet seed crystals in amount equal to substantially one-half of the introduced liquor, slowly agitating, reducing the temperature to about 90° F. to 95° F., maintaining the liquor at this temperature until the mass is permeated with crystals, raising the temperature to about 110° F. to 120° F. to cause the finer crystals to go into solution, slowly cooling the magma to about 90° F. to 95° F. to cause the unmelted crystals to develop into relatively large crystals, maintaining the magma at this temperature for about twelve hours while slowly agitating, and centrifuging to separate the crystals from the mother liquor.

2. The process of producing crystallized dextrose from sugar liquor derived from converted starch which consists, in concentrating the liquor to a density beyond which it would be too viscous, reducing the temperature to a degree favorable for the introduction of dry seed crystals, introducing dry seed crystals into the liquor, dropping the liquor into a crystallizer containing wet

seed crystals and slowly agitating at a temperature favorable for the growth of crystals until a substantial part of the magma has crystallized, raising the temperature until the finer crystals go into solution and again reducing the magma to a temperature favorable to the growth of crystals and maintaining it at the reduced temperature until crystallization has been effected, and centrifuging to separate the crystals from the mother liquor.

3. The process of producing crystallized dextrose from sugar liquor derived from converted starch which consists, in concentrating the liquor to a density beyond which it would be too viscous, reducing the temperature to a degree favorable for the introduction of dry seed crystals, introducing dry seed crystals into the liquor, introducing the liquor into a crystallizer containing wet seed crystals, and slowly agitating at a temperature favorable for the growth of crystals until a substantial part of the magma has crystallized, raising the temperature to cause the finer crystals to go into solution, reducing the temperature and maintaining the magma at the reduced temperature to cause the undissolved crystals to grow into relatively large crystals, and centrifuging to separate the crystals from the mother liquor.

4. The process of producing crystallized dextrose from sugar liquor which consists, in bringing the liquor to the desired density, reducing the temperature of the liquor to the degree most favorable for the introduction of seed crystals, introducing seed crystals into the liquor to induce incipient crystallization, maintaining the liquor at this temperature until incipient crystallization has begun, introducing the liquor into a crystallizer containing seed crystals, slowly agitating at a temperature favorable for the growth of crystals until a substantial part of the liquor has crystallized, raising the temperature of the mass until part of it becomes fluid, reducing the temperature again to a degree most favorable for the growth of crystals and maintaining the magma at this reduced temperature until crystallization has been effected, and centrifuging to separate the crystals from the mother liquor.

5. The process of producing crystallized dextrose from sugar liquor which consists, in concentrating the sugar liquor to substantially 37° to 42° Baumé, cooling to about 100° F., in the presence of seed crystals inducing incipient crystallization, introducing into a crystallizer containing wet seed crystals, further cooling to about 90° F. to 95° F., agitating, reheating to cause the finer crystals to go into solution, recooling to cause the unmelted crystals to grow larger, and centrifuging.

6. The process of producing crystallized dextrose from sugar liquor which consists, in concentrating to the desired density, reducing the temperature to substantially 120° F., adding seed crystals in amount sufficient to start incipient crystallization, introducing the liquor into a crystallizer containing seed crystals in amount equal to substantially one-half of the introduced liquor, slowly agitating, reducing the temperature to the degree most favorable for rapid crystallization, maintaining the liquor at this temperature until the mass is permeated with crystals, raising the temperature to cause the finer crystals to go into solution, slowly cooling the liquor to cause the unmelted crystals to develop into relatively large crystals, maintaining the mass at this temperature for about 12 hours, and centrifuging to separate the crystals from the mother liquor.

7. The process of producing crystallized dextrose from sugar liquor which consists, in concentrating the liquor, cooling it to a degree favorable for the introduction of seed crystals to start incipient crystallization, introducing such seed crystals into the liquor, dropping the liquor into a crystallizer containing wet seed crystals and slowly agitating it at a temperature favorable for the growth of crystals until the whole mass is permeated with crystals, raising the temperature of the mass until part of it becomes fluid, again lowering the temperature of the mass and maintaining it at the lowered temperature while agitating until crystallization has been effected, and separating the crystals from the mother liquor.

8. The process of producing crystallized dextrose from sugar liquor which consists, in concentrating the liquor, cooling it, seeding it for incipient crystallization, further reducing the temperature of the liquor in the presence of wet seed crystals while agitating it until substantially the whole mass is permeated with crystals, raising the temperature to cause the finer crystals to go into solution, again lowering the temperature to effect crystallization, and separating the crystals from the mother liquor.

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