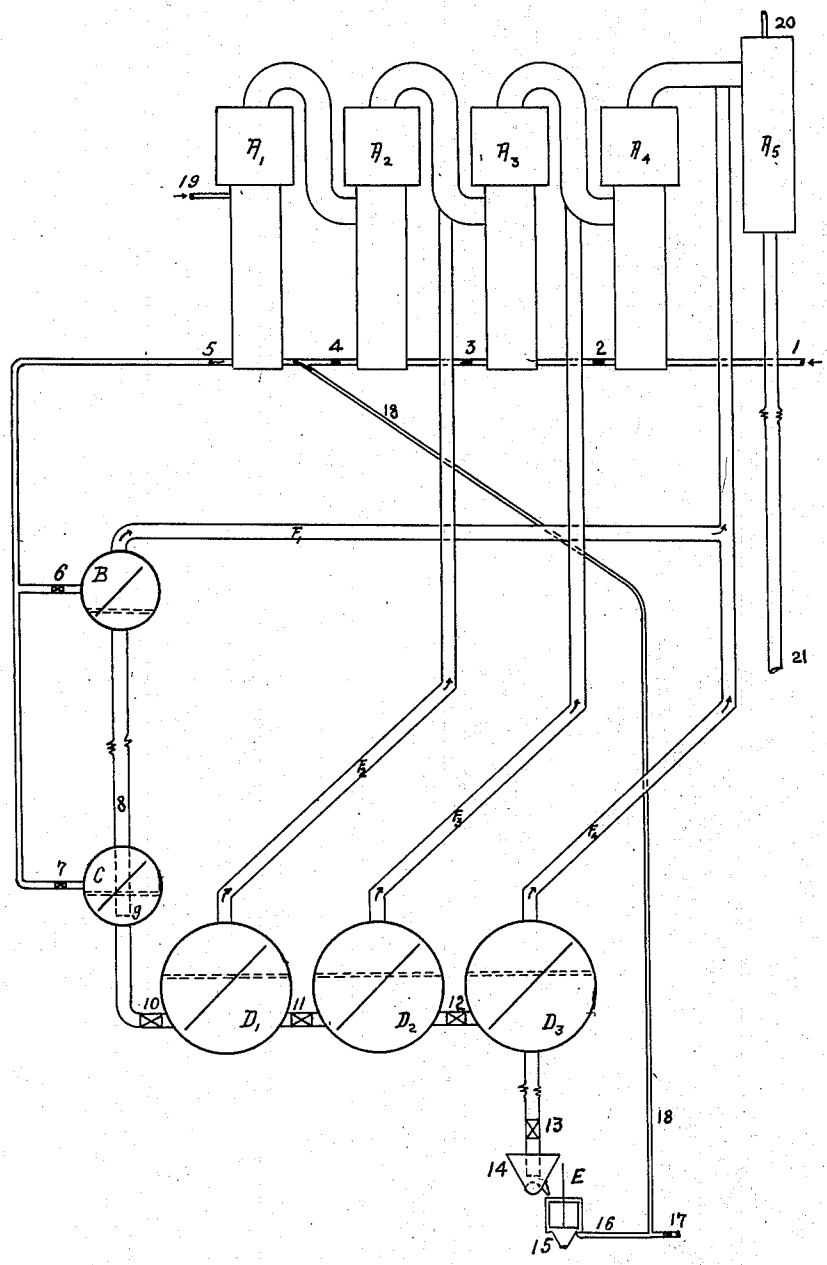
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PROCESS FOR THE RECOVERY AND PURIFICATION OF SUGAR

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INVENTOR.

Alfred M Thomsen

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PROCESS FOR THE RECOVERY AND PURIFICATION OF SUGAR

Alfred M. Thomsen, San Francisco, Calif.

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In the present state of the art sugar is chiefly recovered from the juice of the sugar beet and sugar cane. In the former case, the greater purity of the juice particularly as to invert sugar, permits of the production of a properly refined sugar directly at the sugar house; but in the latter case the bulk of the sugar reaches the ultimate consumer by way of the refinery.

A number of sugar houses do turn out a substantially white sugar from cane juice by the "double carbonatation" and other processes more or less patterned upon beet sugar practice, but to get the best results there seems as yet to be no substitute for the char filters of the refinery. It is therefore my purpose not to try to eliminate this time-honored method of removing impurities, but to so purify the sugar solutions before submitting them to the action of char that the expense of such treatment is greatly reduced.

In general the sugar house operating on cane produces a sugar which polarizes about 96°, and an exhausted molasses which contains the constituents of the juice not accounted for by the raw sugar. While not absolutely correct, raw sugar may therefore be considered as a mechanical mixture of pure sucrose and molasses, the latter not only coating the grains of sugar but also occurring as a constituent of the grains themselves. It may be quite true that the actual sucrose crystal is entirely free from molasses constituents and that the grains are made up of many such primary crystals occluding within their structure the undesirable impurities. Such considerations, however, are rather academic.

The means used by the sugar house comprise a rather simple chemical purification by means of lime, the so-called defecation, and then evaporation and crystallization whereby separation is made between a relatively pure form of sucrose, i. e., raw sugar, and the much more impure molasses which nevertheless unavoidably retain much sucrose, prevented from crystallizing by the impurities.

When the raw sugar reaches the refinery the first step is to remove as much of the adherent molasses as possible by the process called "affination." This is really a direct extension of the raw sugar work for it consists essentially in washing the crystals with a purging syrup which dilutes the adherent molasses and thus permits their removal in the centrifugals. The purging syrup is then boiled down for raw sugar and more exhausted molasses.

The production of molasses is thus seen to be an unavoidable necessity in present sugar practice and all that can be done is to "exhaust" the molasses as far as possible. Nevertheless in the best modern practice the loss in this undesirable by-product is not less than 5% of the total sugar and at times it may be double that amount.

A method whereby it becomes possible to recover this sugar is therefore eagerly awaited by the cane sugar manufacturer who ever looks towards the beet industry for his comparison. Thanks to Steffen's modification of the original attempt to de-sugar molasses by means of lime, the problem is now solved for the beet grower and the Steffen's house is regarded as an integral part of the manufacture of beet sugar. The method whereby this same object may be achieved for the cane industry is therefore included here as a definite step both in the recovery and in the refining of cane sugar.

It has already been shown that affination is a valued step in removing the grosser impurities from the sugar that is to be refined, and it thus diminishes to that extent the work done by the char filters. However, as a goodly share of the impurities are retained within the crystal these still remain and are removed in the subsequent steps. These consist of solution in water, filtration by some type of filter aid to remove suspended impurities and final treatment of the solution in the char filters. The water-white sugar solution yielded by the filters is then sent to the boiling pan where very pure sucrose crystallizes out and is subsequently separated out from a slightly impure syrup that is returned to the circuit.

In sugar refining, which will be dealt with first, my improvement starts with the production of a very much purer sugar than affination sugar as the intermediate step between raw sugar and the char filters. In this manner the molasses impurities are still further reduced in quantity before filtration of any type is resorted to. The means I employ are well known and are simply a further extension of the work of the sugar house to the refinery, namely evaporation and crystallization.

The tendency of the crystal to exclude impurities is one of the oldest and most reliable of the means employed in general chemical manufacture to purify substances, and the reason that it is not now universally used in sugar work to supplement the char filters is solely that it costs too much. Application of re-crystallization to the refining of sugar, therefore, depends upon the method used to effect it. If a far more efficient system than any now in use can be introduced, then re-crystallization has its place in sugar work, but otherwise it will be too expensive. This step depends upon the following facts.

If a saturated solution of sugar at 212° F. be cooled to 130° F., approximately 45% of the dissolved sugar will crystallize out and if it be cooled to 90° F. about 54% will be separated, that is, if enough time be permitted to elapse. At 212° F. the solution will contain about 83 parts of sugar to 17 parts of water. At 130° F. the ratio be-

comes 73 parts of sugar to 27 parts of water and at 90° F. the ratio drops to 69 parts of sugar for 31 parts of water. Re-crystallization can therefore be effected by merely dissolving the affination sugar in hot water to saturation, cooling to 90° F., separating the crystallized sugar, heating the residual sugar to 212° F. and saturating it once more with affination sugar. The entire cycle can then be repeated over and over again.

At first the re-crystallized sugar will be almost perfectly white, but as the syrup becomes more and more impure with each recurrent reuse the sugar will likewise become colored. At an appropriate point, therefore, it must be discarded and can then advantageously be used as purging syrup in the affination step from which it will finally emerge as exhausted molasses. The apparatus employed could conveniently be the ordinary type of crystallizer found in every sugar house and the yield is based upon definite, well established facts.

Unfortunately, however, there are excellent reasons why this simple procedure would result in a loss when compared with present practice. One objection would be that hot sugar solutions invert, though slowly, and to heat and cool such large volumes as would be needed would ultimately result in quite a loss. Another objection is based on the fact that quite a portion of the molasses-forming salts and organic non-sugars are removed by the char filters and, therefore, the introduction of re-crystallization will result in a greater production of molasses than present practice.

The answer to the first objection is found by substituting a more rapid method of cooling, namely, in the application of a vacuum to the crystallizer. In this manner the cooling to 130° F. is made very rapid, the sensible heat of the magma being removed as latent heat of the escaping steam. For each 100 lbs. of magma acted upon some 5 lbs. of water will thus be removed. Not only is the reduction in temperature made highly efficient by this procedure but in addition the sugar held in solution by the water will also crystallize out thus giving a greatly increased yield. For each 100 lbs. of magma this latter item will amount to about 22 lbs. of sugar.

The use of a vacuum as the cooling medium will therefore increase the yield of sugar from 45% to 70% at 130° F. and from 54% to 80% at 90° F.

Instead of operating on a batch basis on a single crystallizer it will be evident that a series of vessels can be employed each one of which is maintained under a higher vacuum than the preceding one. Hot saturated syrup will then enter the first vessel and gradually flow through all of them becoming more and more crystallized as it passes from vessel to vessel until it finally reaches a temperature of around 130° F. To reach a lower temperature than that, such as 90° F., it will, of course, be necessary to employ cooling water in the crystallizer jacket and thus abstract the remaining sensible heat.

Another improvement now appears as a corollary to the series system. If each vacuum vessel be equipped with its own condenser then the cool syrup may be pre-heated by using it as the cooling medium in the condensers. To the last syrup is added enough water to compensate for the evaporation and all the sugar said water will dissolve at the lower temperature. It is then sent through the lowest temperature condenser where its mass is sufficient to absorb and again render sensible the latent heat liberated in the corresponding vacuum vessel. The syrup is then passed through the remaining condensers, becoming hotter with each pass until in the last one it approaches the temperature of the heating vessel. As this progressive heating increases its power of dissolving sugar, additional affination sugar may be added at any desired point in the transit. The hot syrup finally enters the heating vessel where very little additional heat is needed to bring it once more to the initial temperature. The final addition of affination sugar is then made and the cycle repeated.

So far the description has been along rather academic lines, stressing the principle rather than its specific application. Just how this will appear in practice is best studied by referring to the drawing, which represents diagrammatically a combination of a multiple effect evaporator and a continuous vacuum crystallizer with the flow of the sugar bearing solution plainly indicated so that it may be followed by means of the reference numerals.

In the attached drawing, A_1, A_2, A_3, A_4, A_5 represents a conventional quadruple effect evaporator and barometric condenser. B is a relatively small crystallizer directly connected to the highest vacuum by the pipe F_1. C is a mixer under atmospheric pressure. D_1, D_2, D_3 represents a series of crystallizers, connected to one another, and connected to the respective vapor-spaces of the evaporator by the vapor pipes F_2, F_3, F_4. E is the conventional centrifuge assembly.

The purified juice is seen entering at 1 in the direction of the arrow to the liquor space of A_4, and is then advanced by the valved pipes 3—4—5 in progressively concentrated form until it leaves the first or high temperature effect A_1 in a supersaturated condition at 5 also in the direction of the arrow. For the sake of clarity the pumps for such transfer have been omitted in the drawing. To effect this concentration steam is seen entering at 19, is condensed by water entering at 20 into the condenser A_5 and finally issues as water at the base of the barometric column at 21.

The concentrated syrup from 5 goes in part to the crystallizer B and in part to the mixer C at the points 6—7 being again commingled in C as B discharges into C by means of the water leg 8 which enters below the liquor level in C at the point marked 9, thus forming a hydraulic seal. The syrup, now containing the desired amount of seed crystals, then enters the crystallizers proper through the valved pipe 10. The crystallizing magma is then seen progressively advanced to higher and higher conditions of vacuum as it passes by the valved pipes 11—12 into the respective higher vacuums and cooler temperatures of D_2 and D_3, until it is finally discharged by the water leg 13 into the supply tank 14 of the centrifugal E.

This latter device discharges its crystals at 15 and a mixture of syrup and purging water at 16. This mixture is then returned in part to the first effect of the evaporator A_1 by the pipe 18, to undergo once more crystallization after adequate evaporation, or is diverted for the purpose of making a lower grade sugar, or as exhausted molasses, by the valved pipe 17. By appropriate control, the purging water can, of course, be sent exclusively back to A_1 with its proper quota of syrup, while syrup only and

no purging water be discharged at 17. This manipulation would be desirable in the event that such syrup were to be discarded entirely from the sugar cycle.

Minor modifications also suggest themselves. As the initial start of a sucrose crystal is quite pure even from impure syrup, it will not be necessary to commence the cycle with a true solution. Instead, an excess of sugar may be added so that the grains will only partly dissolve and the magma entering the first vacuum chamber will then consist of a syrup in which many fine crystals of relatively pure sucrose are suspended. As soon as additional evaporation and reduced temperature causes further quantities of sugar to separate, such sugar will deposit on the suspended crystals which will thereby grow in size and no false grain will be formed. In this manner the output of re-crystallized sugar may be notably increased without seriously detracting from its purity.

It is apparent that the sequence of these steps can to a certain extent be varied at will. Thus the affination step may be dispensed with and the raw sugar used directly. Of necessity, the resultant sugar will not be as pure as if the affination step were retained but it will be immensely improved.

It is also evident that the precise degree of saturation is immaterial; but, of course, the more concentrated the solution the greater is the percentage of sugar that will crystallize. I have, therefore, used the expression "substantially saturated" as indicating the empirical strength of solution as elected by the operator whether it be below saturation, supersaturated, or even if it contain suspended crystals.

An analysis of the preceding information shows plainly that this process for the refining of sugar is built around my improvements in crystallization and evaporation. These improvements can manifestly be used in other industries as well as in sugar refining and sugar recovery, but in order to make the disclosure of my process sufficiently plain it has been necessary to describe them in detail. I, therefore, wish it understood that I claim these steps by themselves and not merely their application to the sugar industry.

In the version of sugar refining as here given and as illustrated on the flow sheets, I have represented these steps in an advantageous manner; but I wish to reiterate that this is only a preferred illustration and that I do not confine myself to it but claim as well all the modifications herein suggested.

I claim:

1. The method of evaporating and crystallizing which comprises: Passing the solution undergoing treatment through a multiple effect evaporator in a direction counter-current to the passage of the heating steam at such a rate that a super-saturated product issues from the high temperature effect; next passing said super-saturated product, in the direction of the increasing vacuum, through a series of heat insulated crystallizers each one of which is maintained at a progressively higher vacuum than the preceding crystallizer by connecting it with the appropriate vacuum phase of the before mentioned multiple effect evaporator, thus obtaining a crystallized magma from the last crystallizer in the series while the vapor evolved during the cooling process is employed in performing useful evaporation in the multiple effect evaporator.

2. The method of evaporating and crystallizing which comprises: Passing the solution undergoing treatment through a multiple effect evaporator in a direction counter-current to that of the heating steam at such a rate that a supersaturated product issues from the high temperature effect; dividing said supersaturated product into two portions, a minor and a major portion; subjecting the minor portion to the effect of a high vacuum thus producing a copious formation of fine crystals; commingling the minor portion thus treated with the major portion; then passing the mixture of both, in the direction of the increasing vacuum, through a series of heat insulated crystallizers each one of which is maintained at a progressively higher vacuum than the preceding one by connecting it with the appropriate vacuum phase of the before mentioned multiple effect evaporator, thus obtaining a crystallized magma with enhanced size of crystals when compared with the entering seed crystals while the vapor evolved during the cooling process is employed in performing useful evaporation in the multiple effect evaporator; separating the crystals in said magma from their mother liquor and returning said mother liquor once more to the multiple effect evaporator.

3. The method of recovering sugar from sugar containing plant juices which comprises: Passing the suitably purified juice through a multiple effect evaporator in a direction counter-current to the passage of the heating steam at such a rate that a supersaturated syrup issues from the high temperature effect; next passing said supersaturated syrup, in the direction of the increasing vacuum, through a series of heat insulated crystallizers each one of which is maintained at a progressively higher vacuum than the preceding crystallizer by connecting it with the appropriate vacuum phase of the before mentioned multiple effect evaporator, thus obtaining a mobile massecuite from the last crystallizer in the series while the vapor evolved during the cooling process is employed in performing useful evaporation in the multiple effect evaporator.

4. The method of recovering sugar from sugar containing plant juices which comprises: Passing the suitably purified juice through a multiple effect evaporator in a direction counter-current to the heating steam at such a rate that a supersaturated syrup issues from the high temperature effect; dividing said super-saturated syrup into two portions, a minor and a major portion; subjecting the minor portion to the effect of a high vacuum thus producing a copious formation of fine crystals; commingling the minor portion thus treated with the major portion; then passing the mixture of both, in the direction of the increasing vacuum, through a series of heat insulated crystallizers each one of which is maintained at a progressively higher vacuum than the preceding one by connecting it with the appropriate vacuum phase of the before mentioned multiple effect evaporator thus obtaining a substantially caramel-free mobile massecuite while the vapor evolved in cooling is employed in performing useful work in the aforesaid multiple effect evaporator.

ALFRED M. THOMSEN.