(No Model.)

C. STEFFEN.

PROCESS OF OBTAINING SUGAR.

No. 489,879. Patented Jan. 10, 1893.

Fig. 2.

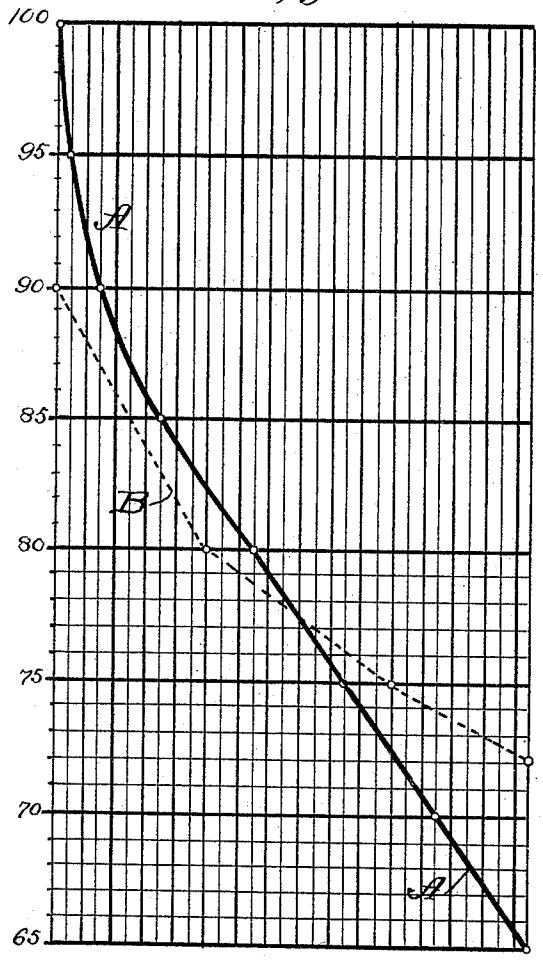
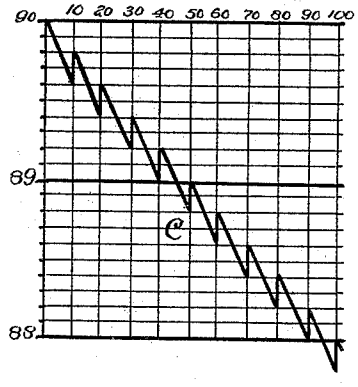


Fig. 1.



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PROCESS OF OBTAINING SUGAR.

SPECIFICATION forming part of Letters Patent No. 489,879, dated January 10, 1893.

Application filed November 20, 1890. Serial No. 372,110. (No specimens.) Patented in Germany February 27, 1890, No. 58,191; in France March 4, 1890, No. 204,121; in England March 6, 1890, No. 3,589; in Belgium March 6, 1890, No. 89,737, and in Spain March 10, 1890, No. 10,595.

To all whom it may concern:

Be it known that I, CARL STEFFEN, engineer, a subject of the Emperor of Austria-Hungary, and a resident of Vienna, Austria-Hungary, have invented certain Improvements in the Art of Extracting Crystallizable Sugar from Saccharine Substances, (for which I have been granted patents in England, No. 3,589, dated March 6, 1890; in France, No. 204,121, dated March 4, 1890; in Belgium, No. 89,737, dated March 6, 1890; in Spain, No. 10,595, dated March 10, 1890; and in Germany No. 58,191, dated February 27, 1890;) of which the following is a specification.

My invention relates to an improved process for obtaining from saccharine juices of plants, and from other more or less pure saccharine solutions the crystallizable sugar which they may contain, by first using such juices or solutions, to wash raw sugar or "masse-cuite," and afterward subjecting them to a novel method of "boiling" in order to produce sugar crystals. In the present and customary method of "boiling" such juices and solutions, the "liquor" is from time to time introduced into the pan as the evaporation proceeds, and the portion of liquor introduced is usually of the same degree of purity, from the commencement to the close of the operation. As the sugar separates a portion is deposited on the crystals first formed thus determining their growth, and another portion gives rise to the formation of new crystals. During the evaporation, therefore, the sugar assumes a solid or crystalline form while the impurities accumulate in the mother liquor which thus consequently decreases in "purity" or amount of crystallizable sugar. The decrease is not. however, a steady or continuous one, as each time a fresh quantity of "liquor" is fed into the pan the purity of its liquid contents is, for the time being increased. It results in consequence, that in this method of boiling, mother-liquor is not able to give up its maximum of crystallizable sugar as rapidly as it should do, nor is the energy of crystallization for a given period of time able to reach a maximum.

In the accompanying drawings:—Figure 1, is a diagram, is given to illustrate the progress of crystallization and evaporation, in the method of extracting crystallizable sugar, heretofore in use. Fig. 2, is a similar diagram, in like manner illustrating the progressive action and conditions at different periods in the process of reduction, according to my improved method.

In Fig. 1, the vertical lines or ordinates are equally divided by the horizontal lines representing abscissas.

The diagonal line in Fig. 1, shows the condition of the liquid contents of the vacuum pan during the progress of evaporation and crystallization. The ordinates represent the "coefficient of purity" of the liquor, and the abscissas the time, the "boiling" has been in progress. The abscissas are divided in spaces representing ten minutes each. (The term "coefficient of purity" is here used in the sense in which it is generally employed: thus the statement that a "liquor" has a coefficient of purity ninety, is equivalent to saying that ninety per cent. of the total solid matter present in solution is crystallizable sugar. This form of expression deals only with the quality of the solution, and gives no information regarding its strength or in other words as to the proportion of water present.)

The "curves" C, show the periodical increase in "purity" of the mother liquor brought about by the introduction from time to time of fresh liquor, in the usual way. This mother liquor owing to the periodical improvement, is unable to reach in a given period of time its lowest possible degree of purity by the separation of sugar. To reach this lowest possible degree of purity is the object and ultimatum of the boiling process.

In the process of boiling it is necessary that the fluid character of the mass be not diminished by evaporation beyond the point at which the particles of crystallizable sugar move easily in it to form crystals or to attach themselves to crystals already formed, or below the point at which the contents of the vacuum pan may be easily withdrawn. As sugar boiling is now practiced this limit of evaporation is reached while yet a large pro-

portion of crystallizable sugar has not yet been crystallized. To get this it is common to reboil the molasses, which indeed is not true molasses because of this undue amount of sugar. The boiling of such mother-liquor or sirups as they are technically called, is however, frequently a matter of considerable difficulty; and before the available sugar can be completely extracted the operation may have to be repeated several times.

Boiling in vacuum premises that the liquor or sirup shall possess such crystallizing capacity that grains of sugar can be produced; but the ease and rapidity with which the crystals are formed depends largely upon the character and purity of the solution to be evaporated. The separation of crystals having however once commenced, takes place subsequently with greater ease; as those first formed promote crystallization by their presence, by the influence which they exert upon the sugar still in solution. Consequently, the larger the number of crystals present, the more energetic will be the crystallization. Very impure solutions cannot be boiled to a grain under ordinary circumstances, because the grain refuses to form at first and the conditions then rapidly become less favorable for crystallization owing to the increase in viscosity of the solution. In considering this question, it should therefore be borne in mind that the evaporation of saturated sugar solutions is not of necessity accompanied by crystallization, and this is especially the case with very impure sugar solutions; these readily become supersaturated. It has, however, long been known that if such impure solutions are brought in contact with a sufficiently large number of crystals, a very effective crystallization can be brought about in the vacuum pan; and this knowledge has been made practical use of in sugar factories by the addition of raw sugar crystals to juices which could otherwise only be boiled with great difficulty. Similarly it is sometimes customary in sugar refineries, when very small crystals are desired, to bring the liquor to the crystallizing point, and then by the introduction of a quantity of finely pulverized sugar to start energetic crystallization, thus insuring the formation of small crystals by shortening the time of boiling and consequently that given to the crystals in which to grow.

It is to be inferred from an inspection of the curve C, in Fig. 1, that the process of boiling would be more correctly carried out, and would also be more effective, if mother-liquor were not subject to periodical improvement. If on the other hand the liquor drawn from time to time into the pan were to correspond in purity with that of the liquor contents of the pan for the time being, a mother liquor of much greater impurity and consequently much more exhausted of its sugar would be obtained in a given time. The object among others, of the process forming the subject matter of this application, is to produce from liquor, or sugar solutions in general, in the manner given below, a series of solutions ranging from a very low to a very high degree of purity. The total quantity of liquor thus operated upon should be about sufficient for a pan of sugar.

In my method, when the liquor has been prepared in portions of increasing purity, the boiling operation is commenced with the best or purest portion. This, in consequence of its purity, possesses great crystallizing power; and it results that in the shortest possible time, and under the most favorable possible conditions sugar crystals are formed in great number. As the evaporation proceeds the remaining portions of liquor are drawn into the pan in order of their purity, the most impure being consequently left to the last. It is clear from what has been already said, that the crystals first formed are able to promote the separation of crystals from the less pure portions of liquor subsequently admitted to the pan and consequently the last and very impure portions, which would under other circumstances crystallize with the greatest of difficulty, yield their sugar readily under the influence of the large number of sugar crystals present. It is moreover evident that, as each successive portion of liquor is drawn into the pan, the quantity of sugar present in a crystalline condition has increased, and consequently the influence it exerts upon each succeeding portion increases also; so that the constantly increasing effect of the greater impurity of the last portions in retarding crystallization is counterbalanced by the constantly increasing quantity of the sugar present which promotes it. In consequence of the attractive action thus exerted by the crystals the whole of the sugar capable of crystallizing is obtained in a solid form, and the mother-liquor remaining is true molasses.

The process has still other advantages. The quality of the mother-liquor is at no time suddenly improved, as the portions of liquor successively introduced into the pan are approximately the same in quality as the mother-liquor surrounding the crystals at the time of such introduction. The addition therefore, of each succeeding portion merely increases the quantity of the mother-liquor without materially affecting its quality. It results that the liquor is more completely under the control of the boiler, who, under other circumstances frequently has difficulty in preventing the formation of crystals he does not desire. It is moreover often a matter of considerable importance that the crystals should be regular in form, as well as in size, and this end can be best attained by preventing any sudden variation in the character of the mother-liquor. The formation of small crystals toward the end of the operation is also entirely obviated for the reason that owing to the last portions of liquor being very impure, they possess but a very small tendency to de-

posit new crystals. The separation of sugar as does occur being essentially brought about by the influence of crystals already present, with the result that the sugar is deposited upon them, merely increasing their size but not their number. In Fig. 2, the same system of lines is shown representing ordinates and abscissas with difference only in number, the degrees of purity ranging from sixty-five to one hundred.

In Fig. 2, the curve B, represents the condition of the mother-liquor during the course of evaporation and crystallization in vacuum, when the liquor fed into the pan is of the same degree of purity throughout the operation. The ordinates give the "quotient of purity" of the mother-liquor, the abscissas the time of duration of the boiling. The curve A, in the same figure shows the condition of another mother-liquor also during crystallization and evaporation, but in this case the liquor fed into the pan is divided into portions according to quality. The portions form collectively a series of sirups increasing gradually from a very low to a very high degree of purity; and are taken in proper order into the pan commencing with the solution of highest value, and ending with that of greatest impurity. The ordinated in this case also denote the quotient of purity of the mother-liquor, and the abscissas the time of duration of the boiling.

The numerical figures given for the co-ordinates are those obtained by actual experiment; they show that in the method of boiling last described a mother-liquor of considerably less purity can be withdrawn from the crystallizing process in a given time than when the method of boiling first given is followed. This is due to two principal causes:—First. By commencing the boiling operation with a liquor of great purity a large quantity of crystals is formed in the shortest possible time, and these crystals promote the separation of sugar from the liquor of less purity which follow. Second. The liquor drawn into the pan does not materially improve the quality of mother-liquor but merely increases its volume, and upon evaporation more sugar is deposited, and a mother liquor of lower quality produced, which is in turn increased in quantity by the addition of liquor of like value.

Having now fully described in the foregoing my new method of boiling liquor after the same has been prepared in portions of ascending purity, I will proceed to discuss the preparation of such portions of liquor from the juices of plants and from sugar solutions in general. The liquor obtained by the partial evaporation of juices of plants &c., is not (as has been heretofore customary) taken directly to the vacuum pan for crystallization, but is merely sufficiently concentrated to furnish a solution of sugar which will be saturated at the temperature at which it is to be subsequently employed; and which will consequently be unable to dissolve any sugar when brought in contact with it at that temperature. The liquor thus prepared is then employed as a lixiviating fluid to displace the molasses from a "masse-cuite" obtained in a preceding boiling operation, and when the molasses has been completely expelled, the liquor adhering to the crystals is in turn displaced by the use of steam, water, or a more or less completely saturated solution of pure sugar.

By proceeding in the manner described, the liquid first flowing from the "masse-cuite" will consist essentially of molasses, which at the time of commencing the washing operation was surrounding the crystals of the "masse-cuite." The liquid following however will gradually increase in purity, owing to the admixture of a portion of the liquor used for lixiviating with a portion of the molasses, and this increase in purity will continue until the whole of the molasses has been expelled, when the liquor will flow from the sugar crystals in precisely the same condition as it was admitted to them. As the liquor is followed by a pure solution of sugar, the liquid flowing from the sugar crystals will gradually approach to, and finally reach, a condition of absolute purity.

In the case in which steam or water is employed to displace the liquor, some of the washed sugar crystals are dissolved, and thus a solution of pure sugar is formed which displaces the liquor adhering to the crystals remaining undissolved.

After discarding the first portions expelled from the "masse-cuite" which consists essentially of molasses, the solution is collected in portions of increasing value which range from a very low to a very high degree of purity. The liquor so collected is then subjected to the boiling process above described, and a "masse-cuite" is obtained, containing in a solid form the whole of the sugar capable of crystallizing originally present in the liquor.

By submitting the "masse-cuite" to the washing process in the manner described it can be entirely free from molasses and the whole of the sugar obtained in a condition of great purity. It is thus possible to obtain in a pure form, from a given liquor, the whole of the sugar capable of crystallizing by first using such liquor for washing a "masse-cuite" obtained in a preceding operation, and then subjecting the same divided into portions according to purity, to the boiling process in the manner described.

The process may be carried out in practice in the following manner:—The juices of plants, or solutions of sugars, are just sufficiently concentrated in the ordinary evaporating apparatus, to convert them into saturated solutions of sugar. If it is not desired to work warm, but at ordinary temperatures, the solutions may be cooled by the help of any of the appliances usually employed. "Masse-cuite" from a preceding boiling operation, either in a warm condition or after passing

through one of the refrigerators ordinarily used for the purpose, is filled in the suction vessels, subjected to suction, and lixiviated by means of a saturated solution prepared as above described. The molasses drawn off by means of a pump, through the perforated bottoms of the suction vessels, is discarded; and the lixiviating fluid is then delivered into a number of reservoirs which separate the same into portions of different quality. The flow of liquor to the sugar is continued until a fluid passes from the suction vessels of nearly the same degree of purity as that entering them. A saturated solution of pure sugar is then fed into the suction vessels until it is withdrawn from them by the pump almost unchanged. The crystals are not surrounded by a pure solution of sugar, and can be simply dried or "melted" and converted into any desired form of refined sugar. The entire washing fluid, which is almost equal in quantity to that from which the "masse-cuite" was produced, being now separated according to its quality in the reservoirs, is from there taken to the vacuum pan and crystallized in order of decreasing purity. The "masse-cuite" produced is then treated with a fresh quantity of saturated liquor so that it may be resolved into pure sugar on the one hand, and on the other into molasses absolutely free from sugar capable of crystallizing.

The separation of the molasses, the washing with liquor and the subsequent lixiviation with water or steam (if employed) can be accomplished in centrifugal machines; and in this case also, after the molasses has been discarded, the liquid discharge from the machines is separated in a sufficiently large number of portions according to its quality. It is recommended that not less than ten portions of liquor of varying quality be employed, and when the best effect is looked for it is even desirable that the number should be larger if possible.

If it is necessary to use the entire quantity of liquor required for one boiling operation, for the washing process, the surplus is to be added to that quantity of liquor having the same degree of purity as itself, and is not to be taken to the pan until all portions of liquor of greater purity have preceded it.

What I claim and desire to secure by Letters Patent is:—

1. The process of extracting sugar from the juices of plants and solutions of sugar in general, by the successive introduction of sugar solutions of constantly decreasing purity into the vacuum apparatus for evaporation and crystallization, such solutions being prepared by employing a saturated solution of more or less pure sugar to expel the sirup from a "masse-cuite" in the manner herein described, and collecting the liquid flowing therefrom into a number of portions of ascending purity.

2. The herein described process of extracting sugar from sugar solutions by systematic boiling, which consists in adding to a solution of sugar undergoing evaporation and crystallization, and consequently still in contact with the crystals it has deposited, solutions of sugar successively decreasing in purity, but of approximately the same quality as the liquor in process of crystallization at the time such addition is made.

In witness whereof I have hereunto signed my name in the presence of two subscribing witnesses.

CARL STEFFEN.

Witnesses:
W. B. MURPHY,
JOHN H. FORSTER.