

UNITED STATES PATENT OFFICE.

OTTO ALEXANDER BIELMANN, OF NEW ORLEANS, LOUISIANA.

PROCESS OF REFINING SUGAR.

SPECIFICATION forming part of Letters Patent No. 557,643, dated April 7, 1896.

Application filed March 29, 1895. Serial No. 543,708. (No specimens.)

To all whom it may concern:

Be it known that I, OTTO ALEXANDER BIELMANN, a subject of the Emperor of Germany, residing at New Orleans, in the parish of Orleans and State of Louisiana, have invented new and useful Improvements in Processes of Refining Sugars, of which the following is a specification.

My invention relates to the art of manufacturing sugar from cane, beet, and all sugar-containing plants, the purpose of my invention being to improve upon the methods of manufacturing sugar in common use at this time, and to obtain more sugar of better quality which shall be ready for direct consumption, the same being produced in one continuous operation. The following is a full description of my process and will serve to make it understood by all who are familiar with the manufacture of sugar.

It is a well-known fact that the method of clarifying raw-sugar solutions (I refer mostly to raw juice obtained from either sorghum or sugar-cane) is an empirical one and removes and alters certain classes of the none-sugars present in said solution to a very limited extent. These are known as "albuminoids," such as arabinose or pectinose, &c., on one side and glucose on the other side, which are eliminated, as stated hereinbefore, only to a very limited extent by the methods in common use. While neither phosphoric acid (H_3PO_4) nor pyrophosphoric acid ($H_4P_2O_7$) is able to eliminate these organic none-sugars named, metaphosphoric acid (HPO_3) and its salts eliminate the greater part of those impurities by coagulation, and thus purify the sugar solutions to a great extent. Therefore I apply a small amount of this acid compound to said sugar-containing solution in any suitable stage of the sugar manufacture—that is to say, I use said acid by mingling the same with the raw juice as soon as the latter is obtained (either by the crushing-mill or by the diffusion process) or I may use said acid after the raw juice is more or less evaporated or at any point where said juice may be termed "syrup." I may also apply it in the vacuum-pan, mixer, or centrifugal machine wherever circumstances make it preferable to do so. I prefer to use said acid by mixing it with the sugar-containing solution after the raw

juice is first extracted either by the crushing-mill or by the diffusion or other process, and before any lime is added, because of the fact that it only shows the peculiarity of coagulating the above-named compounds when the acid is used in a free state. Wherever this clarifying agent may be used, however, I prefer to remove the coagulations which are formed as soon as possible, which may be done either by skimming, filtering, or decanting.

Owing to the fact that in the course of clarifying, evaporating, and reboiling some organic compounds, not fully explored and described as yet, show a greater tendency to coagulate than they show in the first stage of manufacture, I apply a very small amount even to after products, or to first, second, and third molasses, in order to restore the grain-ing capacity to the highest possible point, as more fully described in my application for Letters Patent filed upon the 21st day of July, 1894, Serial No. 518,202.

There are two ways to render the glucose present in saturated or supersaturated sugar solutions crystallizable, the first being to form crystallizable anhydrous glucose crystals by adding a few crystals of anhydrous glucose itself, the second being to form a compound known as "sodium-chlorid" glucose (also termed "glucosate of sodium chlorid") having (after Liebig) the formula $NaCl_2C_{12}H_{12}O_{12} \cdot 2H_2O$. As both crystalline compounds are formed only in saturated or supersaturated solutions, it naturally has to be done in the vacuum-pan, and to do this I use in both cases a few crystals of both anhydrous glucose or sodium-chlorid glucose, either compound being used, as preferred. The introduction of said compounds may be made in various ways, but I prefer to mix said crystals with sugar crystals of a suitable size and add the mixture to the sugar solution, the purpose being in order to start crystallization of both glucose and sucrose at the same time. This course is also desirable because of the fact that being mixed thoroughly in this manner the proportion between the sugar crystals and the crystals of glucose or sodium-chlorid glucose will always be substantially in the desired proportion. If it should be desirable to form the sodium-chlorid glucose compound, I first determine by analysis the

amount of glucose, sodium, and chlorin that may be present in a certain lot of syrup, sufficient in quantity to make a "strike" of first masse-cuite. I prepare, in accordance with the analysis, enough of sodium and chlorin (it being sufficient, under normal conditions, to use sodium chlorid for said purpose) to bind all the glucose present in a given quantity of syrup by forming sodium-chlorid glucose in the manner stated above. Said solution, which must be as highly concentrated as possible, is also mixed with the sugar crystals and the crystals added thereto of sodium-chlorid glucose already mentioned. After this has been done the whole mixture is drawn into the vacuum-pan in the usual way.

In order to get the best possible crystallization of both sugar and sodium-chlorid glucose crystals, it is advisable to boil the strike down to a moisture test of between five per cent. and seven per cent. and, after the finished masse-cuite is dropped in the mixer, to add a suitable amount of a sugar solution of about from five to ten per cent. of the masse-cuite, showing about the same purity as the uncrystallizable part of said strike, or, in other words, the first molasses will show this added sugar solution having a density of about 36° or 38° Baumé, will be mixed with the masse-cuite quickly, and will in the process of centrifugal drying carry through the meshes of the centrifugal sieve the much smaller crystals of sodium-chlorid glucose, where they may be separated either by filtration or decantation.

In order to get rid of the coloring-matter, which, when present, forms a material feature in manufacturing high-grade sugar, I proceed as follows: Instead of using sulfuric-acid gas, as is done in almost all sugar-factories where they make this class of sugar, I may also use hyposulfurous acid, having a formula of H_2SO_2 , or its salts, in which is dissolved a very small quantity of indigo-blue, thus converting the indigo-blue to indigo-white by reduction. I not only bleach the sugar solution to a far better extent, when treated in this manner, than is obtainable by the use of sulfuric-acid gas alone, but I form certain compounds with the coloring-matter of the so-treated sugar-containing juice by the use of hydrosulfurous acid or its salts, insoluble in the sugar-containing solution, which can be removed in the usual way. I prefer to use this bleaching reagent in all the stages of the sugar manufacture where it may be applied profitably; but I prefer to use the same for the most part, as already set forth, on raw juice as soon as the same is separated from the sugar-containing plant, and as long as said juice is in a cold state, and to add a very small quantity of the compound again in the evaporation stage, and finally either in the vacuum-pan or mixer or centrifugal machine, together with the "wash," (generally water and discoloring compounds.)

In order to avoid melting of the sugar crystals, as is done by the use of water to a great extent, I prepare the wash out of sugar-syrup having a density of 25° Baumé and mix with about one-tenth of one per cent. of hyposulfurous acid. I apply said wash only after all molasses has separated from the sugar crystals. This wash, being of a high purity, may be caught separate in the general manner. The indigo-blue mixed with said acid is reduced to indigo-white, and, passing through the whole process of manufacture, will gradually be reconverted into indigo-blue by oxidation taking place wherever the sugar solution or masse-cuite or sugar crystals are exposed to the atmosphere, thus giving the sugar crystals the desired colorless appearance, which will stay and not fade or deteriorate after a very short time, as is the case in the use of any bluing now in common use.

It must be clearly understood that I may use indigo-blue or its equivalent for the purpose described in combination with any acid compound or chemical reagent which will effect similar results.

What I claim is—

1. In the manufacture of crystallizable sugar, the process hereinbefore described which consists in mingling with the sugar-containing solution, at any stage in the process of manufacture, a suitable proportion of metaphosphoric acid or its salts in a free state, whereby the elimination and removal of organic bodies is effected, substantially as described.

2. In the manufacture of sugar, the process herein described, for the elimination of glucose which consists in the crystallization of said glucose by adding to the sugar solution crystallized glucose, or a crystallized salt of glucose, such for instance, as sodium-chlorid glucose, substantially as described.

3. In the manufacture of sugar, the process hereinbefore described, which consists in boiling the strike down to a suitable density, and then adding a mixture of sugar crystals and anhydrous crystals of the type of sodium-chlorid glucose, whereby the crystallization of sucrose and glucose begins simultaneously, substantially as described.

4. In the manufacture of sugar, the process described consisting in the simultaneous promotion of crystallization both of sucrose and glucose, by adding to a saturated or supersaturated sugar solution containing glucose, while being boiled to masse-cuite in the vacuum-pan, a mixture containing sugar crystals of a suitable size, with crystals of anhydrous glucose or the type of sodium-chlorid glucose, substantially as described.

5. In the manufacture of sugar, the process herein described, which consists in adding a small percentage of indigo-white to the sugar-containing juice and again adding a further portion thereof to the sugar solution in the vacuum-pan, substantially as described.

6. In the manufacture of crystallizable

sugar, the process hereinbefore described for decoloring the sugar crystals, said process consisting in adding to the raw sugar-containing juice and to the sugar solutions subsequently derived therefrom, hyposulfurous acid (H_2SO_2) or a salt thereof, bleaching and crystallizing the coloring-matter thereby and separating the same from the solution in the form of an insoluble compound, substantially as described.

20 7. In the manufacture of sugar, the method described which consists in incorporating with a sugar solution hyposulfurous acid (H_2SO_2) or a salt of said acid in which is dissolved a small amount of a decoloring compound, such for example as indigo-white, in order to bleach the sugar-containing solution and to remove coloring-matter present in said solution by forming crystals, which are eliminated in the usual way at the same time by
25 the presence of the indigo-white in the sugar solutions before boiling into masse cuite, every grain of said solution thus containing some of said decoloring compound which will
30 improve the appearance of the dried sugar crystals and by being gradually oxidized to

indigo-blue by atmospheric oxygen, will preserve said color, substantially as described.

8. The process described for manufacturing sugar which consists in the following steps; 30 first mingling with the sugar-containing solution at any stage a suitable quantity of metaphosphoric acid, or its salts, to remove organic impurities, second eliminating the glucose by adding to the solution glucose crystals, or a 35 salt of glucose such as sodium-chlorid glucose, third adding to the sugar-containing juice a small quantity of indigo-white, fourth bleaching the sugar crystals by adding to the solution hyposulfurous acid (H_2SO_2), or a salt 40 thereof, and fifth converting the indigo-white into indigo-blue by the gradual oxidation of the crystals by the atmosphere, substantially as described.

In testimony whereof I have hereunto set 45 my hand in presence of two subscribing witnesses.

OTTO ALEXANDER BIELMANN.

Witnesses:

JAMES L. NORRIS,

THOS. A. GREEN.