UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING LEAD SUCRATE.

SPECIFICATION forming part of Letters Patent No. 726,130, dated April 21, 1903.

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To all whom it may concern:

Be it known that I, ALFRED WOHL, a citizen of the Empire of Germany, residing in Charlottenburg, near Berlin, Germany, have invented certain new and useful Improvements in the Art of Sugar Manufacture, of which the following is a specification.

This invention relates to improvements in the art of sugar manufacture, and more especially to improvements in that particular method of sugar manufacture in which is employed for recovering sugar from impure sugar-containing solutions the well-known property of certain chemical substances to unite with sugar, forming a sucrate, said sucrate being at a subsequent stage of the process converted into another compound, whereby the sugar is liberated in solution, from which it can be recovered in a pure state by crystrallization or other well-known method.

The impurities commonly present in sugar solutions are such mechanical particles as chalk, sand, and iron-rust, also the chemical known as "raffinose." The latter is very difficult to remove, yet on account of its many deleterious properties very necessary to be removed.

The object of this invention is to provide a process of the general type stated by which, however, a larger proportion of the raffinose can be eliminated, together with the mechanical impurities, than by prior processes. In other words, the object is to produce a sucrate containing a less proportion of raffinose. For this purpose the invention consists in adding to an impure alkaline sugar-containing solution a quantity of lead oxid, the addition being made in successive portions and not at once and at a rate below the rate of crystallization of the thus-formed bisucrate of lead and finally separating the crystallized bisucrate from the mass.

The invention consists, further, in subjecting the impure sugar-containing solution to a preliminary treatment, whereby the percentage of raffinose is still further reduced in the finished bisucrate, which preliminary treatment will be fully described, as also the other steps of the process, hereinafter.

In carrying out the process the first step is to add to a sugar-containing solution containing the usual impurities a quantity of alkali, so as to render the same alkaline. Simultaneously the first portion of lead oxid may be added. The proportions are approximately three or four parts sugar solution, such as molasses, one part yellow oxid of lead, and one-quarter part alkali, such as caustic potash. The mass is preferably agitated in a suitable stirring vessel during the mixing, so that the whole is thoroughly well mixed, and the mass is then allowed to stand. By the resulting reaction bisucrate of lead is formed, and the mass is allowed to stand until crystallization of this bisucrate takes place. It is essential that the quantity of lead oxid added shall not be sufficient to permit conversion at once into sucrate of all the sugar in the solution. It must be less than sufficient to cause conversion of the entire quantity of contained sugar into bisucrate. The molasses must be in excess. When crystallization has taken place, a small additional quantity of lead oxid is added to the solution. Owing to the fact that there are already in the solution crystals of sucrate the further crystallization upon addition of this small quantity of lead oxid does not consume so much time as the first. Further quantities of oxid are then added one after another until all the sugar in the solution is converted, sufficient time, however, being allowed between each addition of oxid for the complete crystallization of the sucrate formed by the preceding addition, so that the molasses remains in excess until the last. When all the sugar in the mass is thus converted, the crystals, which are pure white rough crystals of bisucrate of lead held in suspension in the mass, are removed. Their further treatment for recovery of the sugar is accomplished by the ordinary saturation process—*i. e.*, dissolving the crystals in water and treating the solution with carbonic acid, whereby lead carbonate is precipitated and the sugar remains in solution. Any other suitable process may be employed, however, for recovering the sugar from the bisucrate-crystals, the object of this invention being merely to obtain the bisucrate in as pure form as possible. As equivalents for lead oxid in the foregoing may be mentioned lead hydroxid or lead carbonate and alkali in such proportions, for example, as will hereinafter appear.

By the process described a bisucrate is obtained which upon saturation gives colorless solutions and contains only about one-third of the raffinose present according to usual methods.

The initial crystallization—*i. e.*, the crystallization after the initial quantity of lead oxid is mixed with the molasses and alkali—takes some time. The time may be shortened, however, by placing in the solution a small quantity of bisucrate-of-lead crystals. When this is done, it is only necessary to let the solution stand for a short time for crystallization.

As before mentioned, the bisucrate obtained contains only a very small quantity of raffinose as compared with the product of prior processes. When, however, the molasses is subjected first to a suitable preliminary treatment, the bisucrate may be obtained entirely free from raffinose. This preliminary treatment consists in heating the molasses with alkali and lead sucrate (bisucrate) to approximately 90° centigrade. The solution is then filtered and the filtrate, cooled to 20° to 30° centigrade, is treated in the same manner as the molasses in the process first described. When this preliminary treatment is employed, it is not necessary to add to the filtrate a further quantity of bisucrate of lead, as proposed in the preceding paragraph, as the bisucrate added in this preliminary treatment charges the solution sufficiently so that the crystallization is accelerated. As an illustration involving this preliminary treatment, also the use of carbonate of lead and alkali instead of lead oxid, may be given the following example: To one thousand kilograms molasses are added seven hundred liters dilute alkali lye and two hundred kilograms bisucrate of lead having a water content of about fifty per cent. The whole is heated to about 90° centigrade, filtered, and the filtrate allowed to cool to 20° to 30° centigrade. To the cooled filtrate are added one thousand liters strong alkali lye and five hundred kilograms basic lead carbonate having a water content of about thirty per cent., the whole being thoroughly mixed in a suitable stirring vessel and then allowed to stand for a short time until the crystallization of bisucrate is complete. To two thousand five hundred liters strong alkali lye are added one thousand five hundred kilograms lead carbonate, the mass divided into four equal parts, and these parts added one after another to the partially-crystallized filtrate. Sufficient time is allowed to elapse between adding each successive portion to permit crystallization of the bisucrate formed from the previous addition, the whole operation of adding the four portions taking between one and two hours. The resulting mass of bisucrate-crystals is then removed by any suitable means from the solution for further treatment in the manufacture of sugar, as before referred to. Instead of adding the lead oxid or its equivalent in a number of independent portions the whole may be added by continuously running the same into the vessel. This operation is, however, limited by the fact that the speed of the inflow must in no case exceed the speed of crystallization of the bisucrate. It is the essential feature of the process that the lead oxid or equivalent is never in excess; but the sugar is always in excess until the last, the object being to avoid the formation in the solution of amorphous bisucrate. Upon the addition of the lead oxid in the manner hereinbefore described amorphous bisucrate, if formed at all, does not exist as such for any considerable time; but the bisucrate passes quickly or at once into the crystalline condition, from which visible crystals form more or less slowly, according to the quantity of crystals already existing in the solution and according to the other conditions of the solution, such as alkalinity, temperature, and concentration. By carrying out the described process the mechanical impurities and raffinose are left behind in the pulp remaining on removal of the crystals, and a superior quality of bisucrate is obtained.

Having thus described my invention, I claim as new and desire to secure by Letters Patent—

1. The herein-described improvement in the art of sugar manufacture, which consists in adding to an impure alkaline sugar-containing solution a quantity of lead oxid, at a rate below the rate of crystallization of the thus-formed bisucrate, and removing the crystallized bisucrate from the mass, substantially as set forth.

2. The herein-described improvement in the art of sugar manufacture, which consists in adding to an impure alkaline sugar-containing solution a quantity of lead oxid, in a plurality of portions, each following the preceding at a rate below the rate of crystallization of the bisucrate formed by said preceding portion, and removing the crystallized bisucrate from the mass, substantially as set forth.

3. The herein-described improvement in the art of sugar manufacture, which consists in adding to an impure alkaline sugar-containing solution a quantity of crystallized bisucrate of lead, adding to the mixture a quantity of lead oxid less than sufficient to cause crystallization of the entire quantity of contained sugar, allowing the thus-formed bisucrate to crystallize, gradually adding to the mixture further quantities of lead oxid at a rate below the rate of crystallization of the thus-formed bisucrate, and removing the crystallized bisucrate from the mass, substantially as set forth.

In witness whereof I have signed this specification in the presence of two witnesses.

ALFRED WOHL.

Witnesses:
ERNST VON NIESSÉN,
W. WERNSKIUCK.