UNITED STATES PATENT OFFICE.

ALFRED WOHL, OF CHARLOTTENBURG, AND ALEXANDER KOLLREPP, OF BERLIN, GERMANY.

PROCESS OF RECOVERING SUGAR.

SPECIFICATION forming part of Letters Patent No. 695,702, dated March 18, 1902.

Application filed October 20, 1900. Serial No. 33,774. (No specimens.)

To all whom it may concern:

Be it known that we, ALFRED WOHL, Ph. D., residing in Charlottenburg, near Berlin, and ALEXANDER KOLLREPP, Ph. D., residing in Berlin, Germany, citizens of the Empire of Germany, have invented certain new and useful Improvements in Processes of Recovering Sugar, of which the following is a specification.

This invention relates to improvements in that particular method of sugar-manufacture in which is employed for separating the sugar out of its impure solutions the property of certain chemical compounds of combining with the sugar, forming a sucrate, said sucrate being then removed from the solution and further treated for obtaining the sugar therefrom. Lead oxid and lead carbonate are substances commonly employed for this purpose. Recovery of the sugar from the lead sucrate is commonly effected by what is known as the "saturation" method, in which the lead sucrate in aqueous solution is subjected to contact with carbonic-acid gas, whereby lead carbonate is formed and sugar set free. When for carrying on the process of sugar-manufacturing according to this method lead carbonate is employed, said lead carbonate is not commonly employed alone, but in connection with an alkali solution or lye, such as a solution of potassium hydrate, (caustic potash.) The basic carbonate of lead is usually employed. Basic carbonate of lead also is the product of the saturation stage, and it is common to use the carbonate produced by the saturation stage of a preceding run for mixing with the sugar solution for a subsequent run, thus forming a cycle process. Owing to other features of the process, it is desirable that this alkali-lye should be dilute, and it is common to use a lye so dilute that although binding of almost all the sugar takes place, still all the sugar is not entirely bound—*i. e.*, converted into lead sucrate. This difficulty may be overcome, however, by employing instead of the basic carbonate a carbonate more highly (preferably completely) saturated with carbonic acid—for example, the normal carbonate. When this substance is used, the binding of the sugar is complete, even though the alkali-lye be of somewhat greater than the customary dilution. It is possible to produce this normal carbonate in the saturation stage instead of producing the basic carbonate, which is the usual result. This is attained by continuing the saturation with carbonic-acid gas for a much longer time than usual—in other words, saturating until the precipitate shows an acid reaction. This, however, is very deleterious to the sugar, for the reason that non-saccharine matters, in particular alkali salts in the form of lead double salts, are formed, which dissolve in the sugar solution, rendering it impure and also much more difficult to filter; also, the carbonate of lead produced by this method is not as pure as is desirable, and the impurities accumulated in the same after a few runs render it unfit for use in the cycle process.

The object of this invention is to provide a process by which normal carbonate of lead and sugar, both containing a less quantity of impurities, may be obtained from the lead-sucrate solution; and for this purpose the invention consists in the steps of expressing the lye from a mixture of alkali-lye and lead sucrate, treating the sucrate with a warm wash-lye, washing the sucrate, and saturating the same with carbonic acid.

The following example will indicate clearly the steps of the new process. Into a suitable vessel provided with an agitator are introduced five hundred kilograms molasses or other sugar solution from which it is desired to separate the sugar in a pure state, five hundred kilograms strong normal carbonate of lead containing about twenty-five per cent. water, and one hundred and twenty liters crude potash-lye of 1.5 per cent. strength. Twelve hundred liters crude lead sucrate are also introduced, and the mass is agitated for about fifteen minutes and then allowed to stand for half an hour. Five hundred kilograms molasses and eight hundred liters lye are then added, agitation is continued for fifteen minutes, and the mass again left undisturbed for half an hour. Twelve hundred and eighty kilograms normal carbonate of lead and three thousand five hundred liters lye are added in three portions within the next hour. After mixing the mass is allowed to stand and crystallization of lead sucrate ensues, the en-

tire mass of sugar in the molasses being completely converted into lead sucrate. By compression of the mass in a filter-press the lye is now expressed from the lead sucrate. Said lye is now heated to 50°–60° centigrade, subjected to contact with carbonic-acid gas, and filtered. The filtrate, which is free of lead, is evaporated and the residue burned into crude potash, which is suitable for use in repeating the process. The lead sucrate remains in the press in the form of a pulp. In order to free the same from the small quantity of alkali-lye not expressed, it is necessary to wash it. This lixiviating with water may be accomplished without intermediate treatment of the sucrate; but when this is done the filter-cloths become quickly clogged, due to the fact that the wash-water converts a small quantity of lead which is contained in the lye into lead carbonate, and this fills up the interstices of the cloths. This disadvantage is present whether lead carbonate or oxid be used as the desaccharification material. The difficulty may, however, be avoided by not lixiviating the sucrate at once, but first subjecting it to treatment with a weak alkali-lye—for example, the lye commonly known in this industry as "wash-lye." The pulpy sucrate mass is kneaded, mashed, or otherwise thoroughly mixed with a suitable quantity of this lye, preferably in a warm state, and when the mixing is complete is mixed with a further quantity of a still more dilute lye heated to a higher temperature. The heating of the lye causes the greater part of the lead in solution to precipitate in the form of a granular deposit. After the second mixing the sucrate mass may be lixiviated with water in the filter-press without fear of the cloths becoming clogged. After washing, the sucrate is saturated with carbonic acid, which is accomplished by passing carbonic-acid gas through an aqueous solution of the sucrate or otherwise bringing the solution into intimate contact with the gas, whereby the lead sucrate is decomposed, the lead uniting with the carbonic acid and forming basic lead carbonate and the sugar forming with the water a syrup. At the point when the liberation of the sugar is complete carbonation is interrupted and the entire mass filtered. The sugar goes off in a comparatively pure state in the solution and is afterward allowed to crystallize out or is separated out in any well-known manner. The precipitate remaining behind in the filter contains lead carbonate and impurities. To remove the latter, the precipitate is repeatedly washed. It is then mixed with water in abundance, in which it is held in suspension. The mixture is then subjected to saturation with carbonic-acid gas. The basic carbonate absorbs under this treatment a further quantity of carbonic acid and is converted into a comparatively pure normal carbonate suitable for use in a subsequent run of the process. It is desirable to add to the mixture of the precipitate and water a small quantity of a suitable acid—such, for example, as muriatic acid. This addition aids the reaction, and the acid itself acts to reduce the remaining insoluble impurities, such as carbonate of lime, while the highly-saturated carbonate of lead remains unchanged by the acid.

The process above described is also of advantage in that from the normal carbonate produced a superior grade of lead oxid can be obtained in case that substance is used as the desaccharification material in place of the carbonate itself.

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

1. The herein-described process of recovering sugar, which consists in expressing the lye from a mixture of alkali-lye and lead sucrate, treating the lead sucrate with warm wash-lye, washing the sucrate and saturating the sucrate with carbonic acid, substantially as set forth.

2. The herein-described process of recovering sugar, which consists in expressing the lye from a mixture of alkali-lye and lead sucrate, treating the lead sucrate with warm wash-lye, washing the sucrate, saturating the same with carbonic acid, interrupting the saturation when the sugar is entirely liberated, separating the sugar solution from the precipitate, and subjecting the precipitate to additional saturation with carbonic acid, substantially as set forth.

3. The herein-described process of recovering sugar, which consists in expressing the lye from a mixture of alkali-lye and lead sucrate, treating the lead sucrate with warm wash-lye, washing the sucrate, saturating the same with carbonic acid, interrupting the saturation when the sugar is entirely liberated, separating the sugar solution from the precipitate, and subjecting the precipitate in a dilute acid solution to additional saturation with carbonic acid, substantially as set forth.

In testimony that we claim the foregoing as our invention we have signed our names in presence of two subscribing witnesses.

ALFRED WOHL.
ALEXANDER KOLLREPP.

Witnesses:
ERNST VON NIESSEN,
WOLDEMAR HAUPT.