UNITED STATES PATENT OFFICE.

HERMANN REICHARDT AND CARL HEYER, OF DESSAU, ANHALT, GERMANY.

PROCESS OF MANUFACTURING SUGAR.

SPECIFICATION forming part of Letters Patent No. 344,923, dated July 6, 1886.

Application filed February 11, 1885. Serial No. 155,624. (No specimens.)

To all whom it may concern:

Be it known that we, HERMANN REICHARDT and CARL HEYER, both doctors of philosophy, chemists, and residents of the city of Dessau, in the Duchy of Anhalt, and German Empire, have invented certain Improvements in the Process for the Manufacture of Sugar, of which the following is a specification.

The present invention relates to improvements in a method for the extracting or the separating of sugar from strongly-concentrated impure solutions.

All methods of extracting or separating sugar known as yet and based on the formation of earth alkali saccharates render the separation of sugar only possible from diluted solutions, whether it be that either the solution of sugar from which the extraction or separation is to be performed may have only a low degree of concentration or that the agent for the reaction must be used in the shape of hot hydrated lye, (baryta and strontia lye,) or mixed with water, (lime-water, lime-paste.)

Every method for extracting or separating sugar that requires the use of diluted solutions has naturally the one disadvantage that the quantity of non-saccharíferous lye is very great, and thus, even if the quantity of sugar contained in it be small, the loss of sugar is considerable, and the use or application of non-saccharíferous lye (residuum) is therefore combined with great expense. These deficiences and a series of other disadvantages common to all the methods just mentioned, and more or less evident in the one than in the other, are avoided by the present new method, which allows of an extraction or separation of sugar with as small a use as possible of water, and thus proves to be a method of extracting or separating sugar from strongly-concentrated solutions. This method is based on an observation made in a series of experiments, that sugar in strongly-concentrated solutions, in which its molecules are closer together has a far greater affinity to the hydrates of the earth alkalies, and a much greater inclination to form saccharates than when in diluted solutions.

The following method, in which the application of strontia is described, requires only a simple mixture of the sugar in a strongly-concentrated solution—such as is found in molasses or the filling mass—with the base in a solid or compact form. It has been ascertained that the contact of the dry strontia with the strongly-concentrated sugar solution produces even at the ordinary temperature a very quick formation of saccharates which is not to be attained when solutions are employed.

Among the forms in which strontia can possibly be applied, (pulverized oxide, of strontia, hydrate of strontia, and crystallized strontia,) the product which arises from slaking heated strontia, the dihydrate of strontia deserves the preference. This dihydrate of strontia is produced in the following manner: Caustic strontia in lumps as it comes from the furnace is exposed to a fine drizzle of water or of vapor from water. The porous glowed mass sucks up the water eagerly, turns white, and, developing a quantity of steam, crumbles into a white crystalline powder. All superfluous water that has been added evaporates, and when too great an abundance of water has been avoided, there remains only so much water in the strontia as is in keeping with the formula $SrO2(H_2O.)$ The hydrate of strontia $SrO(H_2O)$ obtained in the well-known ways, or the crystals of the nine-times hydrated strontia $SrO9(H_2O)$ can likewise be used advantageously, whereas with pulverized non-hydrated caustic strontia or with hot concentrated lye of caustic strontia (quite apart from the fact that the production of the last-named is much more circumstantial than that of the dihydrate) after the present method only with difficulty, and after a long time a corresponding reaction can be produced, for heating ensues in this case, and the complete formation of the saccharates is consequently retarded. Owing to the considerable quantity of water contained in the strontia lye, and to the heating produced by the high temperature of the concentrated lye, or by the hydration of the caustic strontia, the mass also gets so watery that it fails to have that consistence which is requisite for a quick and complete formation of saccharates. A certain paste-like consistence of the mixture, together with a strong energetic mixing up of it, is extremely material for the success of making strontia sugar after this method, for else the strontia, owing

to its specific gravity, would fall to the bottom, and thus be removed from the reaction. Under the conditions thus stated the strontia saccharate is formed by the method hereinafter described in so short a time (one-fourth to one-half hour) as could not be attained if solutions and equal small quantities of strontia were used. The factory-like working of this method would be, according to what has just been said, as follows: The thick and tough liquid solution of sugar, (molasses, filling mass, &c.,) containing forty to sixty per cent of sugar, is well mixed up with pulverized strontia for the purpose of forming the saccharate. Apparatus is used for this similar to that employed in the manufacture of sugar for cutting up firm or compact filling masses in general, having strongly-constructed stirring-machines with strong arms for cutting and mixing up the thick and tough liquid mixture. However, any apparatus that causes a strong motion and stir can be used for this purpose. The amount of strontia that is added is regulated by the amount of sugar contained, so that for each one hundred kilograms of sugar forty kilograms of hydrate of strontia are added, or for each equivalent of sugar one equivalent of oxide of strontia (SrO) is employed. According to the concentration of the sugar solution, and to the energy of stirring, the formation of the saccharate ensues within the limits stated more or less quick, but is mostly, especially if dihydrate is applied, completed within a quarter of an hour, which is manifested by the clear almost white color and homogeneous thick paste-like condition of the mass. When this appearance shows itself, the mass is mixed in the same apparatus with so much water, or so much of the fluid gained from the washing out of the saccharate, that it becomes liquid enough to be forced by means of a pump into the strongly-constructed filter-presses, into which the mass is admitted from the mixing apparatus in this state. The impure saccharate that remains of the side of the filter-press after the expressing is clarified with pure water or water containing strontia of the remainder of the non-saccchariferous parts. The wash-water thus obtained can always be used for washing fresh quantities of the saccharate. The first non-saccchariferous lye that runs off can if it is not concentrated enough be used in a similar manner; but after being used a second time, and when the strontia is removed, it has generally a specific weight of forty to forty-five Brix, and therefore represents a highly-concentrated residuum. The washed-out saccharate, extracted in the manner just described, and which contains about one equivalent of sugar to one equivalent of strontia, is easily soluble in hot water, and even more so in hot saccchariferous water, and this solubility is taken advantage of to separate the indissoluble impurities, (particles of slag, carbonate nitrate, and phosphate of strontia.) The best way of doing this is to let the hot solution of saccharate settle in high vessels, and, after decantation of the clear solution, to boil the indissoluble parts lying at the bottom once more in water, and separate them from the liquid by means of filter-presses, suction-filters, &c. The clear solution of saccharate thus obtained is disengaged from the strontia by introducing carbonic acid, and the carbonate of strontia thus detached is worked up again into caustic strontia after the well-known methods. By this means, and especially if solutions of sugar are used for dissolving the saccharate, a tolerably concentrated liquor is obtained, which contains twenty to thirty per cent. of sugar of excellent purity. After this method more than ninety per cent. of the sugar contained in the molasses, filling mass, &c., are extracted if the unclean lye (residuum) is used once more for mashing saccharates. The lye that remains after the extraction of the saccharate contains, beside all the so-called non-saccchariferous parts, according to the materials used, a greater or smaller quantity of sugar, glucose, strontia, lime, and a little free ammonia. These substances can under circumstances be neglected and these lyes (residuum) may be worked up direct in the well-known way into coal or manure; but in most cases it will be advantageous to separate and extract one or the other of the substances just named. The most advisable way of doing this is to add a portion of spirit to the cold residuum and in such a measure that a fifty per cent. solution of spirit is formed. By this means if there is a sufficient quantity of strontia or lime the saccharates and glucose parts, which do not dissolve easily in spirit, are precipitated. Any superfluous or additional strontia is likewise in so far separated by the addition of spirit that at the most one-fifth to one-tenth per cent. SrO remains in the solution. The precipitate can be separated by decantation, filter-presses, and suction-filters from the fluid, and in so far as it consists of sugar or strontia is used again in the way described in the process of extraction. If the precipitate contains large quantities of glucose, which will show itself regularly whenever molasses containing glucose, particularly osmosed molasses, refined or cane sugar molasses are used, the glucose is extracted separately by decomposing the glucosate by the introduction of carbonic acid. The residuum containing spirit is freed from spirit and ammonia by distillation in the well-known manner, and then used for making coal, manure, &c. When strontia containing lime or molasses rich in lime compounds are used, the whole amount of lime contained in them is found again in the residuum lye. If these lyes have taken up so much lime that the carbonate of strontia extracted from them by saturation or the saccharate precipitated by spirit are too strongly charged with lime and accordingly furnish a strontia preparation

overmixed with lime, which, owing to the great solubility of sugar-lime, would lessen the extract of sugar, it is advisable to proceed to a separation of the lime from the strontia. This can be performed either by amalgamating the non-sacchariferous lye containing lime before treating it with spirit or by amalgamating the hydrated solution of the precipitate precipitated by spirit with hydrate of alkali, (caustic residuum, coal solution,) whereby hydrate of strontia alone is crystallized; or, again, the two solutions just obtained can be subjected to a treatment with insufficient quantities of carbonic acid, which gives rise to a precipitate consisting, chiefly, of carbonate of potassa with very little strontia, whereas in the saturation that follows a carbonate of strontia tolerably free of lime is precipitated.

The following advantages may be claimed for the method just described: First, the simple method of proceeding combined with the short time of operation till the pure solution of sugar is obtained; second, the perfect manner in which the related strontia is turned to account, which substance, when mixed with sugar, dissolves easier than it does with water alone; third, the avoidance of lixiviation apparatuses and crystallization-vessels for the production of strontia crystals; fourth, the avoidance of all heating and artificial cooling; fifth, the extraction of refuse lye in a highly-concentrated form; sixth, the extraction of all valuable substances else left back in the residuum, especially of strontia in the shape of caustic strontia.

Having now described our invention, and also the manner how it may be performed, what we claim, and desire to secure by Letters Patent, is—

1. The process of extracting sugar from strongly-concentrated and impure sugar solutions, such as filling mass or molasses, which consists in thoroughly mixing them with hydrate of strontia in a solid form and maintaining for a time energetic agitation of the pasty mass, substantially as specified.

2. The dissolution of the extracted and washed-out saccharate in hot water or sacchariferous water in order to remove the insoluble impurities, substantially as specified.

3. The process of mixing the mother-lye, resulting from the separation of the saccharate, with alcohol in order to precipitate the strontia, glucose, and sugar contained therein, substantially as specified.

4. The separation of the lime contained in the non-sugar lye from the strontia by mixing the lye with caustic alkali or by fractionated saturation of the lye with carbonic acid, substantially as specified.

5. The application of the dihydrate of strontia for separating sugar from impure solutions, substantially as specified.

6. The application of alcohol for gaining the sugar glucose and strontia still contained in the mother-lye, substantially as specified.

In testimony whereof we have signed our names to this specification in the presence of two subscribing witnesses.

HERMANN REICHARDT.
CARL HEYER.

Witnesses:
MARC M. ROTTEN,
B. ROI.