UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING INVERT SUGAR.

SPECIFICATION forming part of Letters Patent No. 498,000, dated May 23, 1893.

Application filed April 22, 1891. Serial No. 390,072. (No specimens.) Patented in France October 18, 1889, and November 15, 1889, No. 201,406; in Belgium October 19, 1889, No. 88,121, and November 16, 1889, No. 88,481; in England October 19, 1889, No. 16,540; in Italy October 21, 1889, LIV, 188, and November 16, 1889, LIV, 191; in Luxemburg December 8, 1889, No. 1,217; in Spain July 12, 1890, No. 10,731, and in Austria-Hungary August 16, 1890, No. 49,445 and No. 30,255.

To all whom it may concern:

Be it known that we, ALFRED WOHL and ALEXANDER KOLLREPP, doctors of philosophy, subjects of the King of Prussia, and residing in Berlin, Kingdom of Prussia, German Empire, have invented new and useful Improvements in the Production of Invert Sugar, (for which we have obtained patents in Belgium October 19, 1889, No. 88,121, and November 16, 1889, No. 88,481; in France October 18, 1889, and November 15, 1889, No. 201,406; in Great Britain October 19, 1889, No. 16,540; in Italy October 21, 1889, No. 188, Vol. LIV, and November 16, 1889, No. 191, Vol. LIV; in Austria-Hungary August 16, 1890, No. 49,445 and No. 30,255; in Luxemburg December 8, 1889, No. 1,217, and in Spain July 12, 1890, No. 10,731,) of which the following is a specification.

Prior to this invention the production of invert sugar has been performed as follows:—Cane sugar is dissolved in water to form a diluted solution; this solution is mixed with acid, and the mixture subjected to heat; after heating, the acid is neutralized by means of alkalies (carbonates or hydrates) and the neutralized solution carefully evaporated in a vacuum pan at the lowest temperature admissible in order to obtain the product in a merchantable form ready for use. No process, however, is known, which permits the inversion of cane sugar sirup of the same density as required for the sirup, *i. e.* without the necessity of concentration subsequent to inversion.

Now, we have invented a process by means of which sugar solutions of any degree of concentration, *i. e.*, up to ninety per cent., may be completely inverted at any temperature up to 110° centigrade without causing formation of caramel or coloration of the sirups.

The new process consists in simply melting together cane sugar and water in the proportion required by the degree of concentration of the sirup to be prepared, and digesting the mixture together with a very small quantity of acid, for instance hydrochloric acid, dependent upon the ashes contained in the sugar.

The process comprises the following successive operations: first, estimation of the ashes of the sugar to be transformed into invert sugar sirup; second, melting the mixture of sugar and water; third, digesting the same with acid; fourth, neutralizing the acid; fifth, filtering the sirup.

First. The estimation of the ashes is carried out in the usual manner, viz: by heating a sample of the sugar to be inverted together with sulphuric acid. In case the quantity of resulting ashes is less than 0.03 per cent. of the weight of the sugar, to each one hundred kiligrams of sugar is added a watery solution containing ten grams of hydrochloric acid, (HCl,) free from hydrate. In case of a greater proportion of ashes, sufficient hydrochoric acid has to be added in such excess to the above quantity that the proportion of hydrochloric acid free from hydrate (HCl) is equal to 0.4 of the weight of the ashes.

Second. The melting of the sugar is operated in an open pan provided with a jacketed bottom to receive steam. In case more than two thousand kilograms of sugar are to be transformed at a time, the said pan should moreover be provided with a stirrer, so as to facilitate mechanical stirring. To each one hundred parts by weight of sugar are added ten to fifty parts of water according to the desired degree of concentration of the invert sugar sirup to be produced. The mixture is heated to 70° to 110° centigrade under constant stirring. While the proportion of water here given gives the best results, and a less amount of water will probably not be used in practice, we do not limit ourselves to these exact figures, as it is possible that a slightly greater amount of water than fifty parts might be used under some circumstances without seriously prejudicing the result. The saccharine solution formed by this mixing of sugar and water must contain, however, sixty per cent. or more of sugar.

Third. As soon as the mass of sugar and water is heated to the desired degree of temperature, whereby it is reduced to a partly molten state, acid is added in quantity calculated as stated, and the acidulated mass is subjected

to digestion. This procedure is continued for one-fourth hour with a digesting temperature of 100° to 110° centigrade; one-half hour with a digesting temperature of 90° to 100° centigrade; one hour with a digesting temperature of 80° to 90° centigrade; two hours with a digesting temperature of 70° to 80° centigrade. Within this lapse of time the sugar will completely be transformed into invert sugar without any formation of caramel or coloration of the sirup. The proportions of acid are given giving the best results, but we do not limit ourselves to the exact figures, as a small excess of acid especially of the weaker acids may be used without seriously prejudicing the result.

Fourth. After digestion the acid is neutralized by means of sodic or potassic carbonates or hydrates applied in calculated quantity.

Fifth. The sirup thus obtained is passed through a fine sieve in order to remove any mechanical impurities, and cooled down, when it is ready for use.

Example: One thousand kilograms of white sugar containing 0.025 per cent. of ashes, are mixed with two to four hectoliters of water and the mixture is heated until it shows a temperature of about 95° centigrade, when the steam is cut off and the hot mass acidulated with 0.264 kilograms equal to 0.222 liters of hydrochloric acid having the specific gravity 1.188 (*i. e.* containing one hundred grams HCl), this acid having previously been diluted with 10 liters of water. The acidulated mass is exposed to the action of a temperature of 90° to 80° centigrade for one-half hour, when the sirup is removed from the pan and subjected to rapid cooling. In this manner a clear, colorless and thick sirup is obtained having eighty-five per cent. A quantity of this sirup corresponding to half the normal weight of cane sugar and dissolved in as much water as to obtain one hundred cubic centimeters, will, on observation at 20° centigrade, show a levorotation equal to twelve degrees of the saccharometer. The sirup contains 0.008 per cent. of free hydrochloric acid which is neutralized by the addition of 0.23 kilograms of bicarbonate of soda so that the sirup finally contains 0.013 per cent. of chloride of sodium.

The described process of producing invert sugar sirup may be modified as follows:

First. In order to allow the inversion to be finished in one-half hour at a lower temperature than 90° to 100° centigrade, the proportion of hydrochloric acid has to be increased as follows:—at 80° to 90° centigrade twenty grams of hydrochloric acid per one hundred kilograms of sugar and 0.425 of the ashes; at 70° to 80° centigrade, forty grams of hydrochloric acid per one hundred kilograms of sugar and 0.45 of the ashes.

Second. In order to carry out the inversion in one-half hour at 100° to 110° centigrade, the proportion of hydrochloric acid has to be reduced to five grams HCl per one hundred kilograms of sugar and 0.4 of the ashes.

Third. In lieu of HCl other acids may be made use of, such as HBr., HNO_3, H_2SO_4, HFl, $C_4H_6O_4$ (tartaric acid), $C_6H_6O_7$ (citric acid), H_3PO_4 or H_2SO_3, and in such proportion that there are employed, for each ten grams of hydrochloric acid twenty grams HBr, twenty grams HNO_3, thirty grams H_2SO_4, fifty grams HFl, ninety grams tartaric acid, one hundred and twenty grams citric acid, one hundred and fifty grams H_3PO_4, two hundred grams H_2SO_3, and in respect to the ashes: in lieu of 0.4 to 0.45 hydrochloric acid 0.7 to 0.8 H_2SO_4, 0.45 to 0.5 HFl, 0.95 to 1.1 H_3PO_4, 0.9 to 1.0 H_2SO_3, 0.7 to 0.8 HNO_3, 1.75 to 2.0 tartaric acid, 2.0 to 2.4 citric acid. In all cases the neutralization of the acid has to be performed by the equivalent quantity of an alkali (carbonate or hydrate).

Fourth. The same process may be utilized for producing highly concentrated non-crystallizable sugar-solutions by inverting only half the quantity of sugar contained in the original solution. To this end the described process is so modified as to reduce to one-third either the time of digestion or the proportion of acid which would be necessary for completely inverting all the sugar present in the solution.

We claim—

1. The process of producing concentrated invert sugar sirup which consists in mixing saccharine solutions containing sixty or more per cent. of sugar with dilute acid in proportion determinated as described, and heating, substantially as and for the purpose specified.

2. The process of producing concentrated invert sugar sirup which consists in mixing concentrated saccharine solutions containing sixty or more per cent. of sugar with dilute acid in proportion determinated as described, heating the mixture and neutralizing the acid, substantially as and for the purpose described.

In testimony whereof we have hereunto set our hands in the presence of two subscribing witnesses.

ALFRED WOHL.
ALEXANDER KOLLREPP.

Witnesses:
HENRY SPRINGMANN,
WILHELM LINDMAR.