

UNITED STATES PATENT OFFICE

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PURIFICATION OF WOOD SUGAR

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Wood sugar, which is obtained by the hydrolysis of wood with concentrated hydrochloric acid, may be freed, to a very large extent, from the excess hydrochloric acid if the wood sugar solution in a finely atomized state is brought into contact with a hot current of air (see U. S. A. patent specification No. 1,678,819). The so-called "dry-sugar" obtained in this way still possesses two undesirable properties; it is a light, voluminous powder consisting of particles of irregular shapes and large surfaces, which is therefore, ill-suited for transport and further working up, and also it contains resinous impurities and always remnants of hydrochloric acid, which are detrimental for many purposes. The removal of both of these disadvantages in a technically advantageous manner is the object of the invention.

It might be contemplated to wash out the impurities of the dry sugar with organic solvents, such as alcohol, acetic anhydride and the like. The dry sugar, however, holds firmly in its pores large quantities of added nonaqueous liquids. Thus, 95% alcohol must be added in a quantity up to four times the weight of the product before any of it drains off. Moreover, even when still larger quantities of alcohol are used, the alcohol draining off contains only very little of the hydrochloric acid which is present in the crude product to be purified. Only by washing out the alcohol retained by the mass can considerable quantities of hydrochloric acid be brought into solution. However, it is not economical to work in this manner; the loss in sugar is considerable, the waste of alcohol is too great and its recovery too costly.

According to the present invention, over 80% of the hydrochloric acid present in the dry sugar can be separated by means of certain organic solvents by mere decantation of the same from the sugar and simultaneously the sugar is converted into a syrup of high specific gravity. The organic solvents suitable for this purpose are organic liquids containing oxygen which dissolve hydrochloric acid readily and sugar with difficulty. They are soluble in water. The separation of sugar and hydrochloric acid may,

for instance, be attained by gently heating the mixture of sugar and solvent. The loose sugar thereby sinters together and the solvent used may then be easily and almost completely poured off. The degree of heating necessary depends on the nature of the solvent and on its water content.

A mixture of 95% alcohol with benzene in the ratio of 5:1 has proved to be particularly suitable. It is then sufficient to heat the mixture to 50-60° in order to produce the desired sintering. The presence of benzene depresses the solubility of the sugar in alcohol and raises its absorption capability for the resinous extraneous constituents of the dry sugar.

The separation of sugar and solvent and the simultaneous conversion of the sugar into a syrup form may also be attained by adding a small quantity of water to the mixture of dry sugar and alcohol-benzene. If the dry sugar were first to be taken up with water and the syrup obtained treated with alcohol-benzene mixture, the original large surface of the dry sugar, otherwise firmly retaining the organic solvent, would indeed be destroyed; however, at the same time, the partition coefficient of the hydrogen chloride between the water in the syrup and the organic solvent would be displaced in an unfavorable direction so that much sugar would go into solution with but little hydrochloric acid. It would not be practical therefore to work in this manner.

When the dry sugar contains chlorides besides free hydrochloric acid, it is of great value to remove these chlorine ions also. This may be effected by adding fairly a small quantity of concentrated sulphuric acid to the solvent.

Examples

1. 1000 gms. of dry sugar obtained by hydrolyzing wood with hydrochloric acid are stirred up at ordinary temperature with 3 litres of a mixture containing 1 part of 95% alcohol and 0.2 part of benzene together with 10 gms. of concentrated sulphuric acid; after standing a short while the whole is heated to 55° and then the solvent is poured off. The latter contains 30 gms. HCl and 31 gms. of

sugar, the sugar syrup contains about 68% sugar and 0.3% HCl (all the chloride ion present being calculated as hydrochloric acid), besides about 13% of water and 18.7% of alcohol mixed with benzene. Thus, about 2.8 litres of the solvent mixture used have been poured off.

2. 1000 gms. of dry sugar obtained by hydrolyzing wood with hydrochloric acid are stirred up with 4 litres of the above mixture. After a short time, about 200 cc. of water are added in a finely divided state, whereupon the previously pulverized sugar goes over into syrup. The solvent contains 29 gms. HCl and 40 gms. of sugar, and the syrup, 62% sugar and 0.3% HCl, besides about 20% of water and 17.7% of alcohol-benzene. Thus, about 3.7 litres of the alcohol-benzene mixture have been poured off.

What I claim is:—

1. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, removing the hydrochloric acid by treating the resulting crude powder with a solvent comprising an alcohol, converting the crude product into a syrup, and removing the solvent.

2. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, removing the hydrochloric acid by treating the resulting crude powder with a solvent comprising an alcohol, sintering the crude product by gently heating the mixture, and removing the solvent.

3. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, treating the loose crude powder obtained with a mixture of alcohol and benzene, converting the crude product into a syrup, and removing the solvent.

4. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, treating the loose crude powder obtained with a mixture of alcohol and benzene, sintering the crude product by gently heating the mixture, and removing the solvent.

5. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, treating the loose crude powder obtained with a mixture of 5 parts of 95% alcohol and 1 part of benzene, converting the crude product into a syrup, and removing the solvent.

6. A process for purifying wood sugar ob-

tained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, treating the loose crude powder obtained with a mixture of 5 parts of 95% alcohol and 1 part of benzene, sintering the crude product by gently heating the mixture, and removing the solvent.

7. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, treating the loose crude powder obtained with a mixture of 5 parts of 95% alcohol and 1 part of benzene, sintering the crude produce by heating the mixture to 50–60° C., and removing the solvent.

8. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, treating the loose crude powder obtained with organic solvents containing oxygen which are capable of readily dissolving hydrochloric acid and which dissolve wood sugar with difficulty, and sintering the crude product and separating the solvent with the impurities by adding a small quantity of water to the mixture.

9. A process for purifying wood sugar obtained by hydrolyzing wood and other substances containing cellulose with hydrochloric acid, consisting in drying the wood sugar by atomization, treating the loose crude powder obtained with a mixture of alcohol and benzene, and sintering the crude product and separating the solvent with the impurities by adding a small quantity of water to the mixture.

10. A process as claimed in claim 1 and in which a small quantity of concentrated sulphuric acid is added to the solvent.

In testimony whereof I have signed my name to this specification.

EDUARD FÄRBER.