PUBLISHED

MAY 11, 1943.

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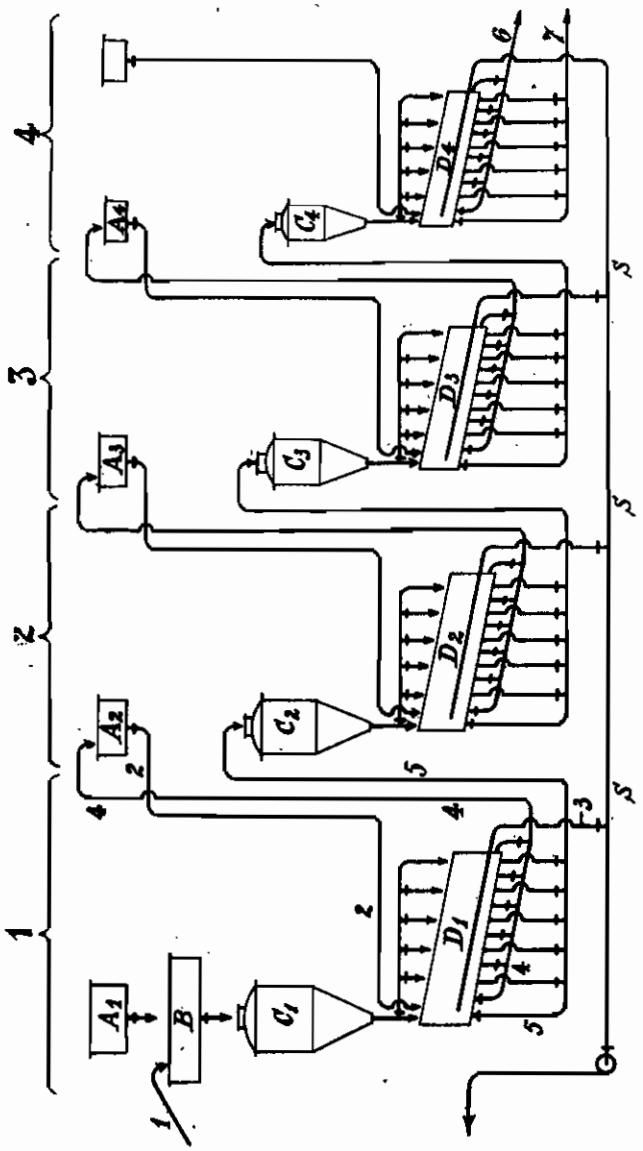
Filed April 18, 1942

Serial No. 439,604

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Published May 11, 1943 Serial No. 439,604

# ALIEN PROPERTY CUSTODIAN

## METHOD FOR SACCHARIFYING CELLULOSIC MATERIALS BY MEANS OF DILUTED MINERAL ACIDS

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Application filed April 18, 1942

Processes for saccharifying cellulose by reacting diluted mineral acids at a raised temperature with suitably divided starting material are already known. The necessity of withdrawing as soon as possible the sugar thus produced to subsequent detrimental action of acid and heat has been recognized by some authors.

As a matter of fact, it has been proposed a process wherein restricted amounts of diluted acid solutions (0.1 to 0.4 per cent) are intermittently passed through cellulosic material in a battery of "percolators" under pressure and at increasing temperature, in order periodically to expel from the reaction zone the sugar produced therein, the proportion being of 5 to 10 parts of acid solution for 1 part of cellulosic material to be treated.

While the foregoing process brings about substantial improvement over prior processes as concerns sugar destruction, it is far from solving the problem in a thoroughly satisfactory manner. As a matter of fact, it is well known that one washing of the material in one vessel cannot provide for thorough removal of the sugar contained therein. Hence, the sugar removal by intermittently adding diluted acid, while fresh sugar is produced in the meantime, can be only partial and a considerable proportion of sugar remains in the reaction vessel wherein it is obviously decomposed during subsequent heating in the presence of further amounts of acid.

It is a primary object of this invention to do away with the aforesaid difficulty and thus to obtain higher sugar yields than by practizing prior processes. A characteristic feature of this invention is that between two successive acid hydrolysis treatments, the starting material is subjected to methodical diffusion in a conventional battery, the liquid used as a vehicle being preferably that diluted acid solution which will be employed for the next hydrolysis. The whole amount of sugar produced in each step is thus removed and a starting material thoroughly freed from sugar is forwarded to the next cooking step.

Another feature of this invention is as follows: the successive hydrolysis treatments to which starting material is subjected are performed at increasing temperatures and with decreasing acidities.

A still further feature of this invention comprises subjecting to hydrolysis a cellulosic material containing only that amount of acid which has been absorbed by its pores, i. e. without any covering or coating liquid. It is thus possible to obtain less diluted sugar solutions than according to other known processes.

Broadly speaking, four successive hydrolysis with intervening methodical diffusion as above set forth will be sufficient for exhausting the starting material but it is obviously within the ambit of this invention to subject cellulosic material to a larger number of hydrolysis.

The concentration of acid liquor and the cooking temperature in each one of the successive treatments may vary within fairly broad limits according to the nature of wood under consideration. By way of merely indicative example, acidities of 6 to 2 grams or 10 to 3 grams per litre may be employed according as hydrochloric acid or sulphuric acid is resorted to; as to temperature, it may vary from 150 to 200° C.

It has been found advantageous to deacidify the material remaining after the last hydrolysis and consisting of entirely exhausted lignin by methodically washing it with hot or cold water. Deacidification is favourable in the respect of further use of lignin, particularly for the manufacture therefrom of a high grade charcoal adapted to be employed in automobile vehicle gas producers.

According to the species of wood, the alcohol yield varies from 220 to 280 litres per 1,000 kg of dry wood. The acid consumption ranges from about 8.5 kg to about 16.5 kg per 100 litres of alcohol according as the case may be.

In spite of the use of diffusion batteries between successive hydrolysis, the apparatus is relatively simple and inexpensive as a large number of its component parts particularly the diffusion elements can be made of wood.

The following example which is not limitative will show with reference to the accompanying drawing, the manner in which this invention may be carried out into practice.

The apparatus shown on the drawing comprises four units 1, 2, 3, 4 each of which includes an acidulated water tank A, a cooking vessel C and a diffusion battery D. The first unit further comprises a humidifying tank B in which acid solution impregnates the starting material.

Wood comminuted into chips is fed through pipe 1 to tank B wherein it is acted upon by said acid solution at a temperature of 90° C. from tank $A_1$, the solution being an aqueous solution containing 5 grams of hydrochloric acid per litre. After being contacted for 30 minutes the wood mass has absorbed 2.5 times its weight of acid solution. After drainage, it is dumped into the cooking vessel $C_1$ having a capacity of 100 hecto-

litres wherein it is cooked during 45 minutes under a steam pressure of 5 kilos per sq. cm., corresponding to a temperature of about 165° C.

The material is then forwarded to diffusion battery $D_1$ which includes 6 diffusors having a capacity of 100 hectolitres, wherein it is countercurrently contacted in usual manner with an aqueous solution containing 4 grams of acid per litre which is supplied from tank $A_2$ at a temperature of 90° C through pipe 2.

A solution having a large content of pentoses and further including a little hexoses flows through pipe 3 and is gathered in collector S. Excess washing liquid flowing out of each diffusion element is recovered in pipe 4 and returned to tank $A_2$.

As to starting material thoroughly freed from sugar and impregnated with acid liquor having an acid content of 4 grams per litre, it is elevated after draining by raising means 5 of any kind to the cooking vessel $C_2$ of the second unit wherein it is subjected for 15 minutes to a cooking treatment under steam pressure of 8 kg per square centimeter corresponding to a temperature of about 175° C.

From cooking vessel $C_2$, the material is dumped into diffusion battery $D_2$ wherein it is washed with water having an acid content of 3 grams per litre coming from tank $A_3$. A solution containing an amount of sugar corresponding to thorough exhaustion of the starting material is withdrawn from the discharge part of the battery.

A like process takes place in cooking vessel $C_3$ and in battery $D_3$, the acid solution used in the latter having an acid content of 2.5 grams per litre and the pressure in cooking vessel $C_3$ being 12 kg per square centimeter corresponding to a temperature of 185° C. Cooking is performed during 15 minutes. Again the withdrawal of sugar liquid from $D_3$ is so controlled as to secure thorough exhaustion of the material.

In the last cooking vessel $C_4$ the steam pressure is about 20 atmospheres corresponding to a temperature of 195° C and the duration of the cooking process can be as short as 7 minutes. The washing liquid used in diffusion battery $D_4$ is water preferably heated to 90° C. From the discharge end of battery $D_4$, there is obtained on the one hand a sugar solution which is removed in the same way as the preceding ones, for instance through collector S, on the other hand pure water which is discarded through pipe 6 and finally exhausted deacidified lignin which may be employed for any purpose, particularly for manufacture of charcoal for gas producers (conduit 7).

The above described plant can be modified in various respects without departing from the spirit of this invention. Particularly it may be advantageous to provide cooking vessels of decreasing sizes from the first one to the last one in order to make allowance for the volume reduction suffered by the starting material as the treatment proceeds. The following sizes may be adopted as an example:

| | |
|---|---|
| First unit: | $C_1$ 100 hectolitres |
| | $D_1$ 6 diffusors of 100 hectolitres |
| Second unit: | $C_2$ 80 hectolitres |
| | $D_2$ 6 diffusors of 80 hectolitres |
| Third unit: | $C_3$ 62 hectolitres |
| | $D_3$ 6 diffusors of 62 hectolitres |
| Fourth unit: | $C_4$ 50 hectolitres |
| | $D_4$ 6 diffusors of 50 hectolitres. |

Again the sugar solutions obtained from each diffusion step can be mixed together or separately reeovered.

Still again only the liquid from the first diffusion step may be gathered separately from the remainder of sugar solutions, as it has a high pentose content and the pentoses may be converted into valuable products such as furfurol.

Nor will the spirit of this invention be departed from if diffusions are effected by means of pure water, the moist mass thus obtained being then admixed for the next hydrolysis with such a mass of acid that suitable acidity will be secured without the volume of liquid retained by the material being increased.

The method according to this invention is valuable for treating not only wood chips or sawdust but also all cellulosic materials of any type whatever.

The sugar solutions manufactured in accordance with this invention may be treated for recovering the sugars contained therein or subjected to any fermentation for converting them into valuable products for instance alcohol. In the latter case it is advisable to employ the yeast reuse method described and claimed in U. S. Patent 2,054,736.

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