

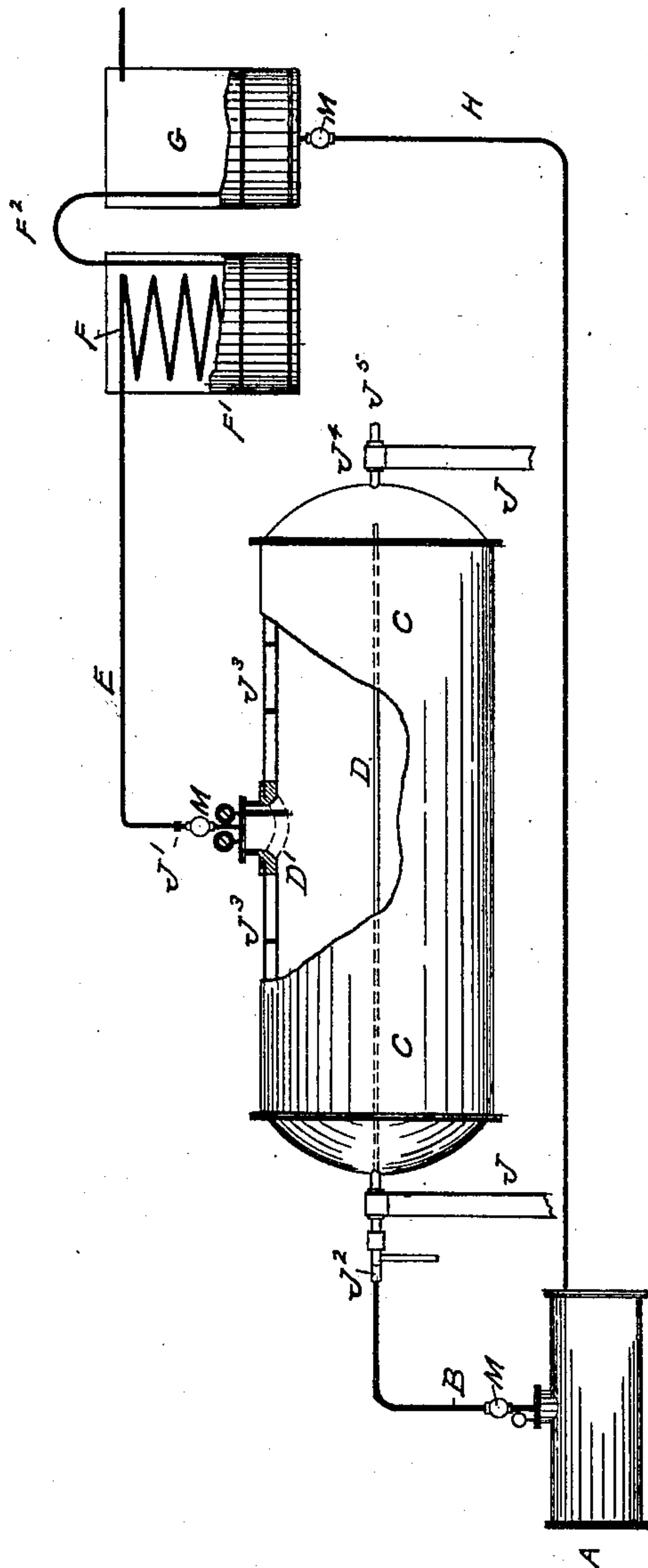
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PROCESS OF CONVERTING WOOD CELLULOSE.

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NO MODEL.



Witnesses.

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# UNITED STATES PATENT OFFICE.

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## PROCESS OF CONVERTING WOOD CELLULOSE.

SPECIFICATION forming part of Letters Patent No. 763,472, dated June 28, 1904.

Application filed March 26, 1904. Serial No. 200,117. (No specimens.)

*To all whom it may concern:*

Be it known that we, MALCOLM F. EWEN and GEORGE H. TOMLINSON, citizens of the United States, residing at Chicago, in the county of Cook and State of Illinois, have invented a certain new and useful Improvement in Processes of Converting Wood Cellulose and the Like, of which the following is a specification.

Our invention relates particularly to a process for converting wood cellulose into sugar.

We have illustrated diagrammatically a device by means of which our process can be carried out. It is shown in the accompanying drawing.

A is a tank for an acid solution; B, a pipe leading thence to a digester C and terminating within the digester in a perforated pipe D.

D' is a manhole with removable cover, whence leads the blow-off pipe E, terminating in a coil F in the cooler F'. From the coil F leads the pipe F<sup>2</sup> into the chamber G. From the chamber G leads pipe H back to the tank A.

J J are supports on which the digester C is mounted. The pipe E is detachable at J' from the manhole, and the pipe D is detachable at J<sup>2</sup>. It is formed so as to have the steam-jacket J<sup>3</sup> between its outer and inner walls, and into this steam-jacket J<sup>3</sup> may be led steam through the tubular support J<sup>4</sup>, whence leads the pipe J<sup>5</sup> to the steam-boiler. Various control-valves M M are shown in different parts of the pipe-circuits.

It will be understood that this device is merely to be taken as diagrammatic and that we intend to use it here simply as a means of illustrating or describing the process.

Through the manhole D' we insert a suitable amount of wood cellulose. We may introduce a quantity of sawdust. The manhole D' is then closed. The tank A is supplied with a solution of sulfurous acid, and we have found that with the charge of sawdust we ordinarily use we may use a solution containing an amount of SO<sub>2</sub>, say, equal to about three per cent., by weight, of the sawdust, such per cent. being based on the dry weight of the sawdust. Heat is now applied to the tank A, and the SO<sub>2</sub> is first driven off through the pipe B into the digester as a gas. A continu-

ation of the application of heat will produce steam in the tank, and the steam will follow the SO<sub>2</sub> into the digester. It is of course evident that steam from any other source may be introduced into the digester. This process should continue until, first, a sufficient amount of SO<sub>2</sub> and steam has been introduced to effect the desired result or, second, until the pressure in the digester is such that the steam will no longer enter. Under ordinary conditions it is possible to raise the temperature sufficiently high to effect the conversion by thus introducing steam on account of its latent heat. When treating sawdust containing from forty per cent. to fifty per cent. of moisture, (this being the amount of moisture usually found in ordinary sawdust,) the amount of steam necessary to raise the temperature of the material to that for the reaction is sufficient to properly moisten the material and give the necessary amount of water to insure the proper reaction. When working with very dry sawdust, it might be found necessary to moisten the sawdust before treating it. The amount of sulfurous-acid gas used will of course depend upon the conditions presented—such as the amount of sawdust, the proportion of the vessel occupied by the sawdust, &c.; but we have found that the best results are secured when the quantity of sulfurous-acid gas is sufficient to raise the pressure of the gas when heated to the temperature of the reaction above that of three atmospheres, the amount of steam being such as not to raise the temperature above 160° centigrade. The sulfurous acid and the steam may of course be introduced in any desired manner. Ordinarily it is desirable to have the temperature in the digester from 120° to 160° centigrade. When the desired temperature and pressure are obtained, the steam-supply to the digester is shut off and these conditions maintained by any desired means—as, for example, by admitting steam into the steam-jacket surrounding the digester—until the conversion is completed. The length of time necessary will of course depend upon the conditions presented. Under ordinary conditions, for example, this time may be from fifteen minutes to one hour. The action is facilitated by the presence of air in the sawdust or digester and may be fur-



ther facilitated, if desired, by the introduction of air, oxygen, or ozone. The introduction of  $\text{SO}_2$  under the circumstances results in the production within the digester of  $\text{SO}_3$ ,  
 5 and thus the conversion is carried on. At a proper time and when the operation has continued long enough the connection through the manhole with the pipe E is opened and the gas is blown off, cooled in the cooler F',  
 10 and is then discharged into the tank G. In this tank any loss of  $\text{SO}_2$  which may have occurred during the operation is supplied in the desired manner to the solution in the tank G, and such solution may then be run back into  
 15 the tank A.

The process here described is one which may be carried out without regard to the degree of moisture in the sawdust itself and without regard to the source from which such  
 20 steam is derived and the source from which such gaseous sulfurous acid is derived. As explained above, the steam and the acid may be derived in whole or in part from heating a solution. It may also be carried on with a  
 25 solution of any degree of density, as it is only necessary to have the requisite amount of  $\text{SO}_2$  in the tank A, and the density of the solution does not affect the process except as to the amount of heat necessary to apply to the tank  
 30 A. Thus, also, the  $\text{SO}_2$  lost during the process may be easily restored to the solution. The period of operation of the digester is greatly shortened by this process, and thus a  
 35 much smaller number of digesters than would otherwise be required may be employed, or much larger digesters may be used in the case where we use the steam for the heating, since  
 40 its heating effect much more easily penetrates the mass of material than if we should heat by exterior application. This is important, because it enables large masses of material to be  
 45 treated at one operation, as the heating is not entirely accomplished by contact or conduction. Sawdust being an extremely bad conductor, when the heating is done by contact  
 50 alone with a hot surface it necessarily requires a long period of heating and a large heating-surface in relation to the mass of material to obtain the conditions for the reaction.  
 By using weak solutions a saving in the cost of acid is also effected. Strong solutions cannot be obtained directly from technical gases.  
 55 Consequently a complicated apparatus is required for purifying these gases, and special provisions are needed for absorbing the same. Thus an acid plant of complicated design requiring skilled manipulation is rendered unnecessary by our process. There is also a  
 60 substantial saving in the amount of heat required to carry out this process.

The materials in the tank, closed vessel, or digester may be mixed or stirred, if found desirable, to facilitate the action.

We claim—

1. The process of converting cellulose into fermentable sugar, which consists in placing a quantity of the cellulose in a closed vessel, introducing therein a suitable quantity of the  
 70 gaseous product derived from heating a solution of sulfurous acid, and heating the mixture to a temperature from  $120^\circ$  to  $160^\circ$  centigrade until the conversion is effected.

2. The process of converting cellulose into fermentable sugar, which consists in placing  
 75 a quantity of the cellulose in a closed vessel, introducing therein a suitable quantity of the gaseous product derived from heating a solution of sulfurous acid until the vessel contains  
 80 about three per cent. of  $\text{SO}_2$  and about sixty to seventy per cent. of  $\text{H}_2\text{O}$ , by weight, of sawdust, and heating the mixture to a temperature from  $120^\circ$  to  $160^\circ$  centigrade until the conversion is effected.

3. The process of converting cellulose into  
 85 fermentable sugar, which consists in placing a quantity of the cellulose in a closed vessel, introducing therein a suitable quantity of the gaseous product derived from heating a solution of sulfurous acid until the vessel contains  
 90 about three per cent. of  $\text{SO}_2$  and about sixty to seventy per cent. of  $\text{H}_2\text{O}$ , by weight, of sawdust, and then heating the mixture by separate application of heat to the vessel until it shall have been kept at a temperature of  
 95 from  $120^\circ$  to  $160^\circ$  until the conversion is effected.

4. The process of converting cellulose into fermentable sugar, which consists in placing  
 100 a quantity of the cellulose in a closed vessel, introducing therein a suitable quantity of sulfurous-acid gas, introducing steam until the material in the closed vessel assumes a temperature of from  $120^\circ$  to  $160^\circ$  centigrade, and permitting the material so to remain until the  
 105 conversion is effected.

5. The process of converting cellulose into fermentable sugar, which consists in placing  
 110 a quantity of cellulose in a closed vessel, bringing sulfurous-acid gas into contact therewith, and introducing steam into the closed vessel to supply the heat and water required to bring about the conversion.

6. The process of converting cellulose into fermentable sugar, which consists in placing  
 115 a quantity of cellulose in a closed vessel, then introducing sulfurous-acid gas and steam into the closed vessel in such relative proportions as to raise the pressure of the sulfurous-acid gas to a pressure at least greater than three  
 120 atmospheres and without increasing the temperature above  $160^\circ$  centigrade.

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