This application is a division of my copending patent application Serial No. 157,254, filed Aug. 4, 1937.

It is well known that cellulose materials such as sawdust, wood-shavings and the like are converted into soluble products by hydrochloric, hydrofluoric &c. acid, resulting under certain conditions in the formation of hexoses (sugar) and lignin. This process which is well known per se cannot be conducted and interrupted at will after the desired effect is obtained, but when carrying out the process varying, and in the most cases unsatisfactory results are obtained, the satisfactory results being rare and merely incidental.

Now applicant has recognized that for obtaining a sure result and high output it is necessary above all to be able to carry out the action of the hydrochloric or the like acid in one and the same operation as uniformly and rapidly as possible throughout the whole material, for only in this case it is possible to interrupt the process at a given moment. The object of the present invention is to provide a number of measurements which as well separately as particularly in connection ensure the desired result.

Above all the conversion produced by the hydrogen halogenide, especially hydrogen chloride, must proceed uniformly in the whole mass of wood, i.e. the moment at which the desired point of a conversion resulting only in a saccharification is attained should be the same in all particles of the wood. This is firstly attained by making the particles of wood swim in the organic liquid distributed as uniformly as possible, their precipitation or agglomeration being prevented. For this purpose as the fluid a mixture of liquids is used, the specific gravity of which may be accommodated to that of the material of wood employed by selecting certain proportions of the components. Moreover the liquid should be adapted to be separated readily and rapidly from the material of wood, i.e. the fluid should be thin-liquid, of low viscosity and relatively easily volatile. After decanting the liquid the parts of water remaining in the solid material must be easy to be removed. As in the present case heating and distilling is contemplated as a means for removing these remnants the liquid should be volatile and low-boliling. On the other hand it should not be so volatile as to produce a considerable pressure of the hydrogen halogenide in the space not filled with liquid. Moreover a high volatility could result in causing greater losses, and, of course, the recovery of the liquid from the expelled gases would be rendered difficult. Care should be taken to have the boiling limits of the components of the mixture as close to one another as possible to prevent a too strong disunion of the components during the distillation.

Now I have found that for the present purpose a mixture of light gasoline with carbon tetra-chloride is excellently adapted, about two parts by weight being employed for three parts by weight of gasoline. Of course, the exact proportions depend upon the specific gravity of the material of wood used in each case. Of course, instead of gasoline other organic solvents and instead of carbon tetrachloride other hydrocarbons, preferably halogenized, especially chlorinated hydrocarbons may be used. For instance, trichlor-ethylene. Of course, the fundamental condition to be imposed upon the solvents in question is the possibility of adjusting the specific gravity by mixing solvents having a different specific gravity, furthermore the mutual mixability of the components and their completely inert behaviour in view of each other and of the reagents to be used, the cellulose material and the hydrogen halogenide.

Furthermore the liquid medium should be easy to be removed from the reaction vessel after the conversion has been completed. The liquid remaining in the residue after the main quantity has been withdrawn is removed in the well known manner by driving off, preferably by means of superheated air with or without reduced pressure or merely by distilling in vacuo.

The above characterized rapid and uniform conversion is further obtained by sorting or sifting the chips or thelike of wood to be used, preferably by mechanical means, according to the size of the chips, so that for a determined charge only chips of approximately equal size are introduced into the reaction vessel; furthermore by using, as far as possible, for one and the same charge a uniform material of wood, for instance, pine wood or mixtures of different kinds of wood, f. i. pine and Scotch pine wood composed in predetermined proportions. Furthermore the saturation of the wood should be completed in the shortest time, with in 4 to 6 hours at most, for when the conversion lasts too long there is a risk in the single particle being decomposed beyond the conversion desired, whereas the interior of the particle of wood is not yet converted to the desired extent. This risk is prevented by limiting the length of the wood particles (chips, for instance) in the direction of the wood fibre to about 4 to 8 m. m., by employing uniform material of...
wood, and finally by previously treating the raw material of wood with saturated steam in order to wet and to loosen it thoroughly. The greatest permissible width and thickness of the particles cannot generally be exactly determined, the structure of the different kinds of wood differing to a considerable extent and opposing a greater or less resistance to the infiltration by hydrochloric acid. This resistance, however, does not only depend upon the chemical composition of a species of wood, but to a greater extent upon the physical properties of the chips (their brittleness) and consequently upon the method of comminuting the wood. It is decisive for the capability of absorbing hydrochloric acid whether the wood is comminuted across or along the fibre. As to the shavings or chips, obtained by paring, it is of great consequence whether they have been prepared by shaving “with the wood” or “against the wood,” that is to say whether the chips are clean like a ribbon or brittle. It was ascertained that the action of the hydrochloric acid goes on the more rapidly and thoroughly the more the fibres were broken up during the comminution of the wood, i.e. the more the natural capillary vessels of the wood substance were opened. For instance, the brittle and fragile chips of oak-wood are easily and rapidly converted, but it is true that the outputs are small. The clean chips of pine-wood require a longer time of conversion and give better outputs because of their higher cellulose contents. However when using pine-wood or other kinds of wood giving clean chips the time of conversion may be considerably reduced, without diminishing the output, by employing hacked or chopped chips instead of shavings. Hacked chips give absolutely the shortest conversions and the best outputs, and the best utilizing of the vessel room.

For instance, when treating a certain kind of wood (fir-wood) in an ordinary apparatus with hydrochloric acid for five hours, hacked chips gave 18%, shavings in a uniform thin layer 15%, hacked chips and shavings mixed (differing in kind) 10% of alcohol.

Therefore it is recommendable to subject the different kinds of wood, those having different size and those produced by different methods of comminution, separately to hydrolysis, whereby the safest result is obtained.

The above-mentioned previous treatment with saturated steam effects in all cases a further loosening of the wood structure whereby the conversion of the wood substance by the hydrogen halogenide is facilitated.

I claim:

1. The process of converting cellulose material into soluble compounds which comprises subjecting the material to the action of hydrogen halogenide whilst suspending the material in an organic liquid having substantially the same specific gravity as the material to be treated.

2. The process of converting cellulose material into soluble compounds which comprises subjecting the material to the action of hydrogen halogenide whilst suspending the material in a mixture of organic liquids having substantially the same specific gravity as the material to be treated.

3. The process of converting cellulose material into soluble compounds which comprises subjecting the material to the action of hydrogen halogenide whilst suspending the material in a mixture of about three parts by weight of gasoline and about two parts by weight of carbon tetrachloride having substantially the same specific gravity as the material to be treated.

4. The process of converting cellulose material into soluble compounds which comprises subjecting the material to the action of hydrogen halogenide whilst suspending the material in an organic liquid having substantially the same specific gravity as the material to be treated, and continuously assimilating the specific gravity of the liquid to that of the material under treatment as the latter varies during the process.

5. The process of converting cellulose material into soluble compounds which comprises subjecting the material to the action of hydrogen halogenide whilst suspending the material in a mixture of about three parts by weight of gasoline and about one part by weight of carbon tetrachloride, and gradually increasing the amount of the latter to three parts by weight.

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