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PROCESS FOR THE DIGESTION OF VEGETABLE GROWTHS TO PRODUCE CELLULOSE

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9 Claims. (Cl. 92—9)

In my copending application, Serial #660,649, of March 14, 1933, I describe processing for the digestion or decomposition of wood, for secural of cellulose fibre by means of nitric acid thoroughly mixed with or disseminated throughout a liquid as carbontetrachloride. Sixty percent fuming nitric acid was employed most advantageously by me since its specific gravity and that of carbontetrachloride vary but little, so that comingling of the two liquids was readily brought about. In using nitric acid with benzol, the requisite conditions were not as easily established. Recently, however, I have brought about comingling or thorough mixing of the latter liquids by what, I believe, has been termed a "turbo mixer", that is apparatus in which rapidly traveling metal blades produce mixing of the liquids within a closed metal cylinder. The liquids thus thoroughly mixed were allowed to flow directly into the digester which had been previously filled with the wood chips. Very satisfactory digestion was then secured when using about twenty percent of nitric acid on weight of dry wood. The benzol used was about three to one on the weight of dry wood. Digestion was carried out either cold or hot in a type of digester described in my Patent #1,820,276 of August 25, 1931.

After digestion, the benzol was first drawn off by suction from the digester and then a hot dilute caustic soda solution was run into the digester. The benzol contained in the digested organic material was thereby volatilized and condensed by cooling coils situated externally to the digester. After treatment with the hot caustic solution, the latter was also drawn off by suction from the digester and the cellulose fibre washed with water, either hot or cold, with possible finally slightly acidified water.

I have also brought about digestion in the type of digester above described when using carbontetrachloride and oxygen, without the use of nitric acid. The digestion is bettered by first admitting steam to the digester to the extent of twenty to forty percent on the weight of dry wood. Of course other percentages of water may be employed. The digestion was carried out under about a hundred pounds pressure of oxygen under the conditions above noted and with hot carbontetrachloride, although even cold can be satisfactorily used. Subsequent drawing off and volatilization of the carbontetrachloride and final treatment with an aqueous hot caustic soda solution were carried out as usual. Only one set of traveling metal bars were used in the digester and as the edges of the moving metal blades were brought nearly to the bottom of the digester, or just above the screen at the outlet of the digester and also situated close to its bottom, these blades tend to keep the screen free of excess pulp or fibre, so that the liquids, as carbontetrachloride, etc., are readily drawn off through such screen. In the latter arrangement, the shaft carrying the heater blades or bars were situated not centrally to the digestion cylinder, but between its centre and circumference.

Similar digestions were made with benzol and other coal tar distillates and with water and alcohols. The higher alcohols, as propyl, butyl and amyl, peculiarly become miscible with water from the resin extracted from the wood, so that such solution of resin can be used directly in the pulp beater. In carrying out this work with the use of benzol, the wood was first steamed to about a twenty to forty percent content of water before digestion with the organic liquid, although in the case of the lower alcohols, such as methyl and ethyl, the water and alcohol may be more readily simply first mixed together. I also used ammonia when employing the alcohols as a further digestion aid, and likewise carried out several digestions with bonzol and the alcohol aqueous mixture in an atmosphere of hydrogen at about one hundred pounds pressure. Subsequently to the digestion with the organic liquids noted, a hot caustic solution was employed for complete freeing of the fibre. The beating of the fibre was also continued during this latter step. A hot caustic solution appears most advantageous before final treatment of the fibre, although, of course, other commonly employed digestion solutions might be used in the diminished quantities or percentages required because of the, what we might term, primary organic digestion. Of course wood digestion is ordinarily carried out at superatmospheric pressure, about one hundred and twenty five pounds, whereas I use in the final digestion only a hot aqueous solution at atmospheric pressure.

Digestions of waste cotton seed hulls for securing the fibre content were carried out very similarly to those already described. A larger percentage of chemicals, however, were required for their digestion, for instance, about thirty percent of nitric acid. As noted in my copending application I have carried out digestion with other vegetable growths than wood or cotton seed hulls and secured analogous results. My complete series of experiments, however, were conducted with wood, a fair percentage of same were likewise applied to cotton seed hulls and a

few digestions made with other fibres. In view of similarity in all of these digestions or experiments I feel that I have a just basis for claiming the basic principles of digestion or of extraction to all vegetable growths.

My apparatus was built either of steel or of an iron-nickel-chromium alloy. Acid corrosion of the former appeared very slight but no doubt for continuous operation the alloy would be preferable.

My three steps of development or of invention cover, first a cheap method of digestion or of destruction of the resin content of wood or of other vegetable growths, with nitric acid, and recovery of the cellulose fibre, secondly a further reduced cost in resin destruction because of the use of oxygen (air), or combinations of the two steps, and finally the disintegration or digestion of the wood with the recovery of the resinous material as well as the cellulose fibre.

I claim:

1. In a process for the digestion of vegetable growths to secure cellulose fibre, the step of subjecting the said growths to the action of nitric acid mixed with organic liquids.

2. In a process for the digestion of vegetable growths to secure cellulose fibre the step of subjecting the said growths to the action of fuming nitric acid mixed with organic liquids.

3. In a process for the digestion of vegetable growths to secure cellulose fibre the step of subjecting the said growths to the action of nitric acid mixed with benzol.

4. In a process for the digestion of vegetable growths to secure cellulose fibre the step of subjecting the said growths to the action of nitric acid and benzol and subsequently treating the residual material after drawing off of the benzol with a hot aqueous solution of sodium hydroxide.

5. In a process for the digestion of vegetable growths to secure cellulose fibre the step of subjecting the said growths to the action of nitric acid mixed with benzol and the step of disintegrating the said vegetable material mechanically by the action of cylindrically rapidly moving metal pieces simultaneously with the process of digestion, and, after withdrawal of the benzol, the step of subsequently treating the residual material from such digestion with a hot aqueous solution of sodium hydroxide and simultaneously subjecting the products of the digestion to the action of cylindrically rapid moving metal pieces.

6. In a process for the digestion of vegetable growths to secure cellulose fibre and for extraction of resinous matter, the step of subjecting the said growths to the action of organic liquids extraneous to the naturally occurring vegetable fibre constituents obtained by aqueous digestion and the step of disintegrating the said vegetable material mechanically by the action of cylindrically rapidly moving metal pieces simultaneously with the process of digestion or of extraction.

7. In a process for the digestion of vegetable growths to secure cellulose fibre and for the extraction of resinous matter, the step of subjecting the said growths to the action of benzol and the step of disintegrating the said vegetable material mechanically by the action of cylindrically rapidly moving metal pieces simultaneously with the process or of extraction.

8. In a process for the digestion of vegetable growths to secure cellulose fibre and for the extraction of resinous matter the step of subjecting the said growths to the action of water and benzol above its boiling point and at super-atmospheric pressure and the step of disintegrating the said vegetable material mechanically by the action of cylindrically rapidly moving metal pieces simultaneously with the process of digestion or of extraction and, after withdrawal of the benzol, the step of subsequently treating the residual material from the said digestion or extraction with a hot aqueous solution of sodium hydroxide and simultaneously disintegrating such residual material mechanically by the action of cylindrically rapidly moving metal pieces and finally the suitable washing of the cellulose fibre thus produced.

9. In a process for the digestion of wood to secure cellulose fibre and for the extraction of resinous matter the step of subjecting the wood cut into chips to the action of water and of benzol above its boiling point and at super-atmospheric pressure and the step of disintegrating the said pieces of wood mechanically by the action of cylindrically rapidly moving metal pieces simultaneously with the process of digestion or of extraction and after withdrawal of the benzol the step of subsequently treating the residual material from the said digestion or extraction with a hot aqueous solution of sodium hydroxide and simultaneously disintegrating such residual material mechanically by the action of cylindrically rapidly moving metal pieces and finally suitably washing of the cellulose fibre thus produced.

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