H. W. DOUGHTY.

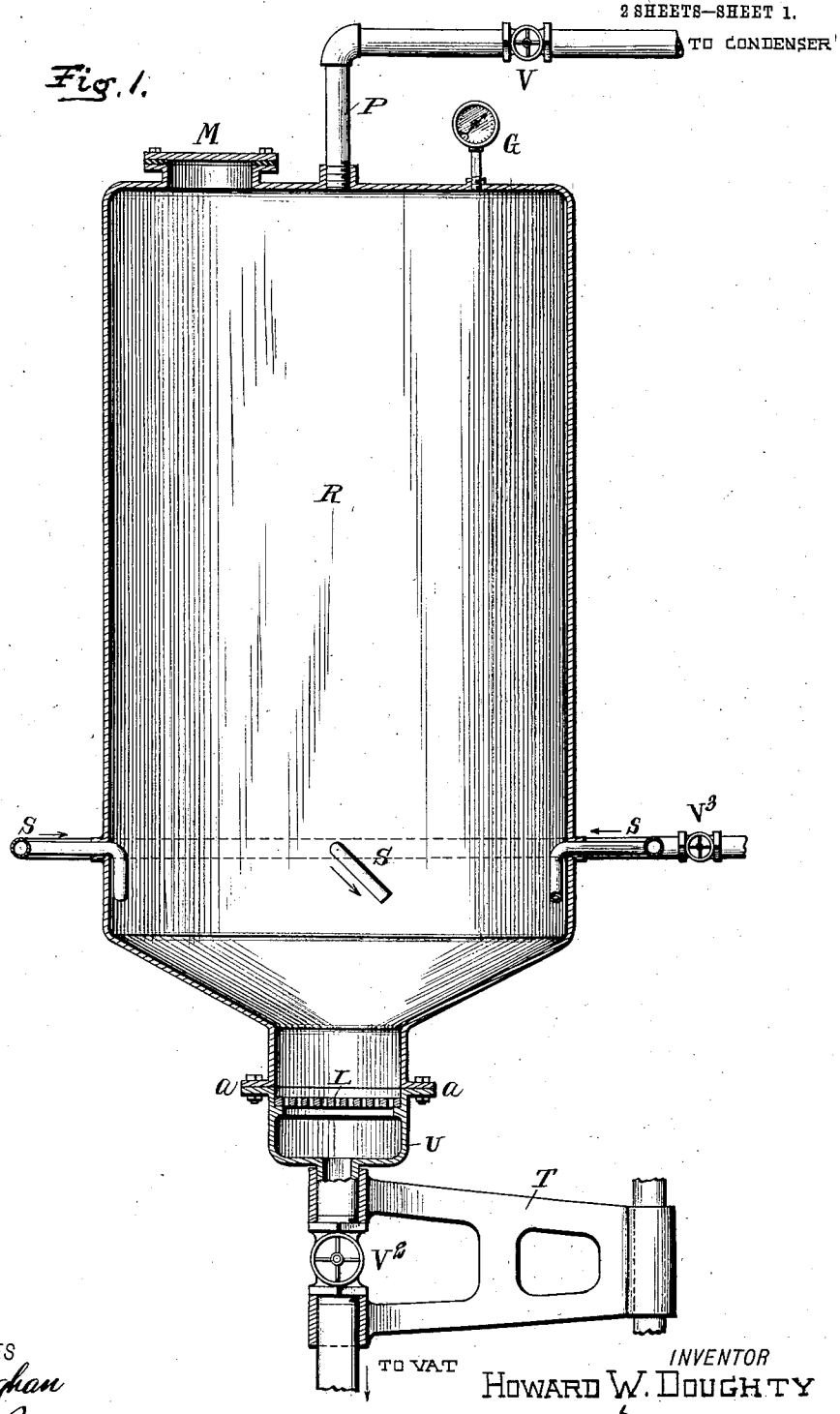
METHOD OF TREATING WOOD DURING DISTILLATION.

APPLICATION FILED MAR. 30, 1908.

930,274. Patented Aug. 3, 1909.

2 SHEETS—SHEET 1.

Fig. 1.



WITNESSES

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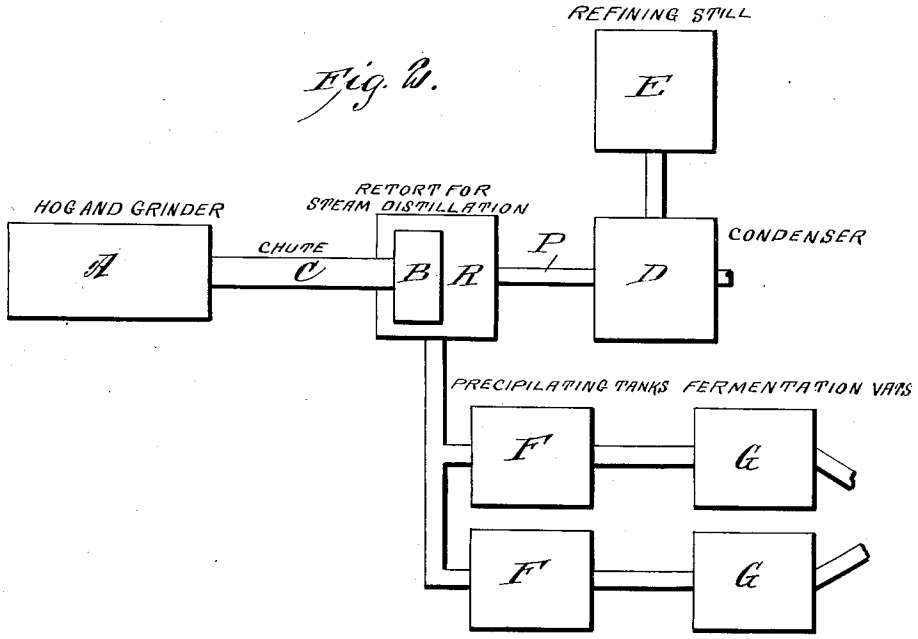
2 SHEETS—SHEET 2.

Fig. 2.



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UNITED STATES PATENT OFFICE.

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METHOD OF TREATING WOOD DURING DISTILLATION.

No. 930,274. Specification of Letters Patent. Patented Aug. 3, 1909.

Application filed March 30, 1908. Serial No. 424,060.

To all whom it may concern:

Be it known that I, HOWARD W. DOUGHTY, a citizen of the United States, residing at Amherst, in the county of Hampshire and State of Massachusetts, have invented a new and useful Improvement in Methods of Treating Wood During Distillation, of which the following is a specification.

The object of my invention is to provide a new and economical method of utilizing the waste from resinous pine wood, in the form of saw-dust, mill-chips &c., to recover, in commercially valuable forms, the various constituents of such waste.

The method comprises, first the distillation, from such waste, of the oils that are volatile with steam, such as turpentine; second, the simultaneous hydrolyzing of the remaining cellulose with a non-volatile acid, such as sulfuric acid; third, separating the resinous acids; fourth, fermenting the filtrate and distilling therefrom the ethyl alcohol, and fifth the separation of the unchanged cellulose, which may be utilized for paper pulp or be dried and mixed with a binder to form fuel briquets or be molded into various useful articles.

The novelty of my method resides chiefly in the use of the non-volatile sulfuric acid during the distillation with steam, so that the hydrolysis of the cellulose may take place simultaneously with the removal of the volatile oils and by the same heat of distillation.

My invention is illustrated in the accompanying drawings in which—

Figure 1 is a vertical section of a retort for carrying out my invention, and Fig. 2 is a diagrammatic view showing the arrangement of the various pieces of apparatus used in the process.

In Fig. 1, R is the retort that I propose to use. M is a manhole through which the soupy mass of ground wood and dilute acid is introduced. S, S, S, are pipes which deliver steam into the retort. The delivery ends of these pipes are set at an angle of about 45° to the vertical, so that the force of the steam may impart a whirling motion to the contents of the retort. The main pipe from the boiler, which connects with these pipes, is provided with a throttle valve V^3 by which the pressure of steam in the retort can be regulated when this valve is used in conjunction with a similar valve V, in the condenser pipe P. The pressure is registered on the gage G. L is a perforated plate of lead, copper, or earthenware, which serves as a filter to retain the pulp when the acid and alkali solutions are run out and during the washing of the pulp. The lower part of the retort is removable by disconnecting the flanged joint at *a*, *a* and swinging the part U aside by means of a swinging arm T. The pulp then drops into a can or other receptacle, and the retort is then closed and is ready for another charge. V^2 is another valve by opening which the liquid contents of the retort can be run off. The retort is to be made of copper or of steel lined with some material resistant to acid, such as lead and the piping is of copper.

In carrying out my invention I proceed as follows:

1. The waste resinous material is first reduced to a pulpy mass by being "hogged" in the apparatus shown at A in Fig. 2, followed by such treatment with edge runners or similar devices as may be necessary.

2. This pulpy mass is then mixed with dilute sulfuric acid to a thick creamy consistency, the concentration of the acid being 1 to 2 per cent., reckoned on the total water present. This material is introduced into the retort R by means of the chute C and hopper B and heated; steam is passed through, pressure being gradually increased as distillation proceeds up to about 100 pounds per square inch, and the steam distillate is condensed in suitable apparatus D. Turpentine, "pine oil", and in general oils volatile with steam, are thus removed from the pulp and are collected and refined in the usual manner in the still E. The pulpy mass is kept in constant agitation by the steam and the cellulose during the distillation process is hydrolized by the sulfuric acid to fermentable sugar.

3. After the volatile oils have been removed, the eduction pipe P of the retort is closed, and the heating is continued under a pressure of about 100 pounds per square inch, thus completing the hydrolysis. The liquid portion of the contents of the retort is then by opening valve V^2, run off into vats F, where it awaits the product of the next stage of the process. The residual pulp is washed with water and the washings added to the liquid already obtained. This

liquid contains sulfuric acid and fermentable sugars, with other less important products.

4. The pulp remaining in the retort is then treated with a dilute solution of sodium hydroxid, varying from one to five per cent. according to the conditions as hereinafter explained, and steam is passed in through pipes S, S, S, to agitate the mass and mix it thoroughly. The retort is then emptied and the residual solid filtered from the soda solution and washed thoroughly. The solution contains the sodium salts of the resinous acids, particularly sodium abietate.

5. The soda solution (4) is run into the vat containing the sulfuric acid solution (3) in such proportions that a neutral solution of sodium sulfate results, and abietic acid, together with other insoluble resin acids, is precipitated. This precipitate on heating gives rosin and various oils.

6. The solution containing sodium sulfate also contains the fermentable sugars from the hydrolysis of cellulose (3). It is subjected to fermentation in the vats G and the alcohol is obtained in the usual manner.

7. The mother liquor after removal of alcohol contains sodium sulfate. This liquor can be evaporated and the sodium sulfate recovered and sold as "salt cake", or the liquors can be "causticized" by treatment with lime, obtaining calcium sulfate as the end product.

8. The pulpy residue (4) consists of more or less impure cellulose, which may be worked up into paper, paste board, artificial wood, fuel, or otherwise.

9. Should the rosin in the waste be small in amount, or should the pulp resulting be intended for fuel, the treatment with sodium hydroxid may be omitted and the sulfuric acid solution treated directly with lime, and, after filtering, be subjected to fermentation. The pulpy residue will then contain all the resinous material, and may be pressed into briquets and used for fuel, the resins in the mass serving as the "binder".

The time required for the various stages of the process is difficult to state, for it depends very largely on the size of the retort and the amount of turpentine and rosin in the wood. So far as the retort is concerned, the operations in it require about four hours. The distillation with steam begins at about atmospheric pressure, and the turpentine goes over very rapidly at first, and is perfectly colorless. After most of the turpentine has gone over, the valve V is partially closed, so that the pressure in the retort is increased. The valve V^3 in the main pipe leading to S, S, S, is opened more widely at the same time. The pressure in the retort is thus gradually increased until it reaches about 100 pounds per square inch, and the entire treatment with acid and steam requires about three hours. During the earlier stages very little glucose is formed, but the percentage increases rapidly with the higher pressure. The boiling with alkali takes about half an hour, and the washing about half an hour longer. The strength of the alkali depends on conditions. If the alkali is used under pressure a one per cent. solution of sodium hydroxid would suffice, but on account of the large volume of the solution, better results are obtained by using a stronger solution, up to five per cent.

The advantages of my process are as follows:

1. The volatile oils, as turpentine are separated in salable condition.

2. The hydrolysis of the cellulose proceeds simultaneously without transfer of material and by the same heat of distillation used in separating the volatile oils.

3. The rosin is extracted from the wood, if desired, and recovered in salable condition.

4. The acid and alkali extracts mutually neutralize each other, and the rosin obtained helps to defray the cost of the acid and alkali used in the process.

5. Ethyl alcohol is obtained.

6. The residual pulp is cellulose free from rosin and turpentine and in general from all substances soluble in acid or in alkali.

7. The soda may be recovered.

8. By omitting the use of sodium hydroxid and neutralizing the acid solution with lime, the resins may be retained in the pulp, increasing its fuel value.

I claim—

1. The herein described process of treating wood during distillation, which consists in contemporaneously hydrolyzing the cellulose with sulfuric acid and steam under pressure and driving off the volatile oils by the same heat, treating the solid residue with sodium hydroxid and after filtering bringing the acid and alkali solutions together in equivalent proportions, whereby the sulfuric acid is neutralized and the resinous acids are precipitated, then filtering, fermenting the filtrate and distilling off the alcohol.

2. In the distillation of resinous woods, the method of contemporaneously hydrolyzing the cellulose with a non-volatile acid, distilling off the volatile oils by the same heat, separating the liquid contents from the residual pulp, neutralizing the acid liquor and extracting the resinous acids with sodium hydroxid, fermenting the liquor, and then distilling therefrom the ethyl alcohol.

3. In the distillation of resinous woods, the method of contemporaneously hydrolyzing the cellulose with a non-volatile acid by the same heat employed for the distillation of the volatile oils, separating the volatile oils by distillation, drawing off the acid

liquor, washing the remaining pulp with water and adding the washings to the acid liquor, treating the washed pulp in the retort with a dilute solution of sodium hydroxid and agitating it by steam, separating this sodium hydroxid solution from the pulp and adding said sodium hydroxid solution to the acid liquor of the first distillation to neutralize it and precipitate the resinous acids, then fermenting the sweetened solution and distilling off the alcohol.

4. In the distillation of resinous woods, the method of contemporaneously hydrolyzing the cellulose and extracting the volatile compounds which consists in reducing the wood to a pulpy mass, mixing said pulp with dilute sulfuric acid in a closed vessel, passing through the mass steam under pressure, and conducting off the volatile oils freed by the distillation.

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