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A. H. WHITE

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PROCESS FOR THE PARTIAL PYROLYSIS OF WOOD AND PRODUCT OF THE SAME

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FIG. 1.

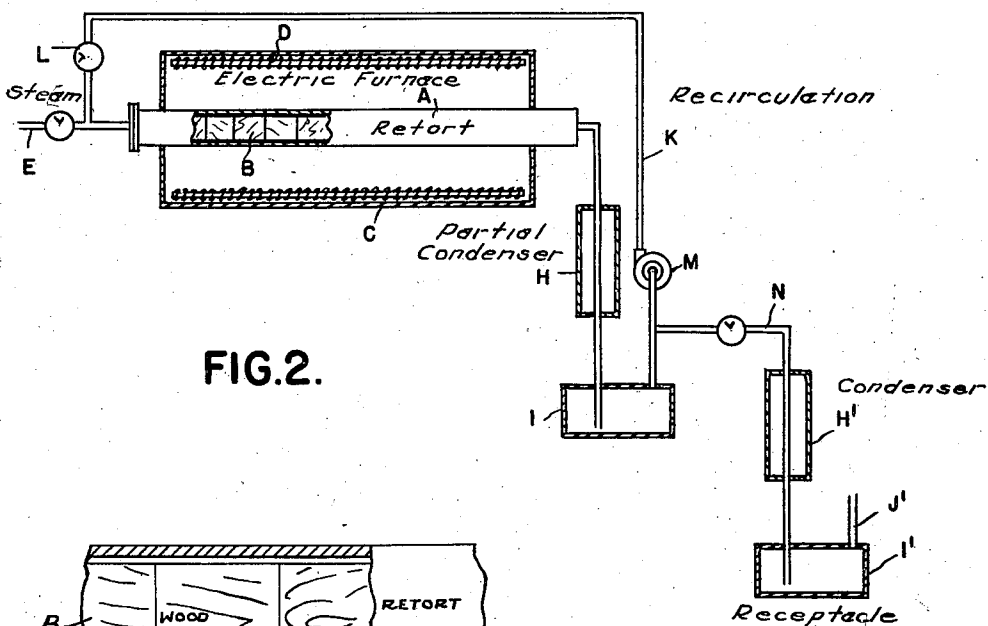
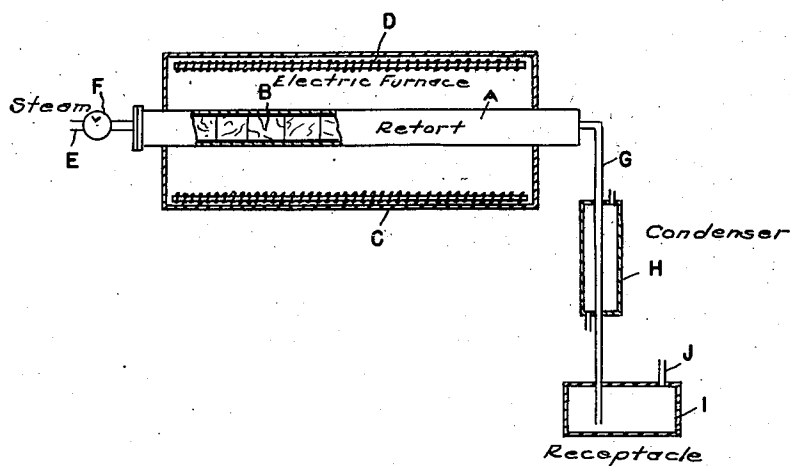


FIG. 2.

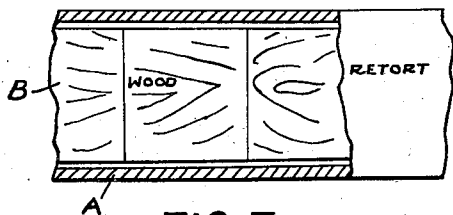


FIG. 3.

INVENTOR.

ALFRED H. WHITE

BY *Whittemore Hulbert & Belknap*
ATTORNEYS

UNITED STATES PATENT OFFICE

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PROCESS FOR THE PARTIAL PYROLYSIS OF
WOOD AND PRODUCT OF THE SAME

Alfred H. White, Ann Arbor, Mich.

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7 Claims. (Cl. 202—16)

The invention relates to the manufacture of products by pyrolysis of wood and has for its object the obtaining of a process in which the wood under treatment is not completely destroyed or reduced to charcoal but retains a fairly high percentage of its original structural strength. It is a further object to obtain a modified wood product having improved characteristics for certain uses. Still further, it is an object to obtain other products by partial pyrolysis of the wood which, while not as great in quantity as would result from complete pyrolysis, are still sufficient to be of value. With these objects in view, the invention consists in the process and products thereof as hereinafter set forth.

In the drawing:

Figure 1 is a diagrammatic illustration of an apparatus suitable for carrying out my improved process;

Figure 2 is a similar view showing a modified construction;

Figure 3 is an enlarged section of a portion of the retort shown in Figure 2.

The pyrolysis of wood in retorts to form charcoal with recovery of methanol, acetic acid and other by-products has been practiced for almost a hundred years. Theoretical studies of the pyrolysis of wood were made by Violette in 1851, Klason in 1909 and by many other investigators, notably Hawley and his colleagues at the Forest Products Laboratories.

There is general agreement that when wood is heated in a closed retort to about 280° C. an exothermic reaction occurs which causes the temperature to rise rapidly, with formation of charcoal and evolution of methanol, acetic acid and other by-products. All of the commercial methods for the pyrolysis of wood have permitted the temperatures to rise to a point where this exothermic reaction took place with formation of charcoal.

Hawley in his book on Wood Distillation summarizes the experiments of earlier investigators who had studied the pyrolysis of wood and states, page 54:

"These results show plainly that there is very little decomposition of wood by heat at temperatures below about 250° C., but on account of other more indefinite data this conclusion must be limited to the conditions of the experiment in which the heat is applied rapidly. . . . So far as actual destructive distillation is concerned, then, the reaction begins about 250° C., soon becomes exothermic and is finished at about 350° C."

It has been recognized that moisture in the wood produces steam which may influence the reaction, but there is no agreement as to whether the steam exerts an important effect on the products formed. Hawley states on page 58 of the reference already cited:

"It is now possible to speculate on the possible effect of varying moisture content of the wood on the yields. . . . Palmer has made a study of this question in small scale apparatus and found that the effect varied with the species and with the speed of distillation so that no general conclusions can be drawn."

The process which I have invented deals with the partial pyrolysis of wood in an atmosphere which contains a large amount of steam, and in which the temperature is controlled so that there is no pronounced exothermic reaction and the final solid product is modified wood instead of charcoal.

The results obtained by my process will be illustrated by the following data from a single run. The charge consisted of nine blocks of oak wood each 1" x 1" x 4" which had been dried at 105° C. prior to the test. These were placed in a steel retort which was surrounded by an electric heating element. As soon as the temperature in the retort reached 150° C. which required about 2 hours, steam at atmospheric pressure was allowed to flow slowly through the retort. The temperature rose to 240° C. in about 2 hours more and was maintained at that level for 10 hours. The temperature was measured by two thermocouples, one embedded in one of the wooden blocks and the other in the vapor space. These two couples differed very little from each other throughout the whole run. Atmospheric pressure was maintained throughout the test. The steam and gases evolved were passed through a condensing system and the liquid distillate was collected. In this test the weight of steam introduced was 2.4 times the weight of the wood. At the end of the 10 hours heating period the retort was allowed to cool to 200° C. when it was opened and the blocks of wood were removed.

The heat-treated oak wood was a dark brown in color but otherwise looked like the original wood and showed no signs of charring. Some of the changes in physical properties and chemical composition are given in Table I. It will be noted that the treated wood has a compressive strength (when the pressure is applied to the end of the grain) of 7090 pounds per square inch

which is 88 per cent of the original strength. The fracture indicated that the treatment had made the wood considerably more brittle. It was also found that the treated wood could readily be pounded to small pieces, and then ground to a fine powder. The treated wood was also more porous, but absorbed a smaller amount of water and expanded less when kept in a damp box at 25° C. until equilibrium was reached, as shown in Table I.

Chemical analysis showed that almost 94 per cent of the pentosans and 35 per cent of the cellulose had been decomposed by the heat treatment, while the lignin showed an apparent increase. The acetic acid obtained by hydrolysis of the wood dropped from 4.0 in the untreated wood to 0.2 per cent in the treated sample. The methoxyl content was decreased almost 30 per cent by the heat treatment, but no appreciable amount of methanol was produced.

Some of the products formed in this partial pyrolysis are listed in the first column of Table II. The second column gives, for comparison, data from another test in the same apparatus in which the temperature was allowed to rise slowly to 400° C. with production of charcoal. The results of this second column correspond more closely to those obtained in commercial operation, although steam was introduced in a similar manner in both tests. The partial pyrolysis at 240° C. caused the evolution of 78 per cent as much acid, calculated as acetic, and of 62 per cent as much furfural, but no measurable amount of methanol.

Other experiments conducted in a similar manner showed that wood was decomposed to a lesser extent at as low a temperature as 180° C. A test at 180° C. showed that the dried wood had lost 6.5 per cent of its weight and that acid, titrated as acetic, had been formed to the extent of 2.2 per cent of the weight of the wood. Experiments at temperatures of 260° C. showed distinct charring and a compressive strength of the wood residue which was only about half that of the wood before treatment.

The tests reported above have been on oak but tests on birch and on other woods have shown similar results, and there is reason to believe that all types of woods and vegetable tissues will respond to this treatment in a similar manner although the upper and lower permissible temperatures will vary somewhat with the different species.

In the example which has been given dried wood was heated in an atmosphere of steam. It was not necessary to use dried wood and some of the steam might equally well have been generated from the moisture in the wood. It is recognized that steam is always present in the commercial pyrolysis of wood, but the amount becomes low in the later stages of the commercial process as the wood becomes dried and as gas is evolved through the pyrolysis of the wood. In my process, the steam has its greatest value at temperatures above 220° C. when there is danger that an exothermic reaction will start and cause the temperature to rise in an uncontrolled manner. There is always some gas in the retort in my process but I aim to keep the volume of steam at least nine times that of the fixed gas throughout the active stages of the process.

The process which has been described is a partial pyrolysis under controlled conditions which prevent an exothermic reaction from gaining headway and at temperatures below that at which noticeable charring occurs. The steam acts

not only as an inert gas but also is active in causing hydrolysis of the organic compounds.

TABLE I

5 Properties of oak wood before and after partial pyrolysis

	Dried at 105° C.	After treatment in steam at 240° C.
10		
Relative weight.....	100	74.7
Relative length (tangential).....	100	92.4
Relative apparent density.....	100	85.0
Crushing strength (pounds per sq. in.).....	8,250	7,090
15		
After exposure to damp air at 25° C. until equilibrium was reached:		
Per cent moisture absorbed.....	20.3	9.9
Per cent increase in length.....	6.4	2.3
Chemical composition in per cent of dried wood:		
Total pentosans.....	20.6	1.2
Cellulose (Cross and Bevan).....	55.4	36.4
20		
Lignin.....	25.7	34.7
Methoxyl content.....	5.9	4.2
Acetic acid by hydrolysis.....	4.0	0.2

TABLE II

25 Products of partial pyrolysis of oak wood

[Expressed in per cent by weight of dried wood]

	After treatment in steam—	
	At 240° C. maximum temp.	At 400° C. maximum temp.
30		
Total acids (as acetic).....	5.30	6.82
35		
Methanol.....	None	2.25
Furfural.....	2.45	3.95

Various forms of apparatus may be used for carrying out my improved process but, as diagrammatically illustrated in Figure 1, A is a retort of any suitable construction for containing the wood B to be treated. This retort is placed within a furnace C shown as an electrical furnace having a heating resistor D. E is a conduit connected to one end of the retort and controlled by a valve F which regulates the admission of a constant stream of steam. G is a conduit leading from the opposite end of the retort passing through a condenser H, the condensate being collected in a receptacle I while the uncondensed gases escape through a vent J.

In Figure 2, a modified construction is shown which, in addition to the elements above described, has a return conduit K leading from the receptacle I to the conduit E and controlled by a valve L. A blower or other propelling means M in the conduit K forces the recirculation at a predetermined rate. There is also a conduit N leading from the conduit K through a second condenser H' and into a second receptacle I' provided with a vent conduit J'. With this construction, the condenser H is operated at a temperature only slightly below the boiling point of water so that only a portion of the products formed by pyrolysis and of the steam condense and are collected in the receptacle I. A portion of the steam and of the gases and vapors formed in the retort are returned by the blower M with a resultant saving in fuel, and a higher concentration of valuable products in the receiver than would be the case if no steam were recirculated.

Although the modified wood has become more brittle through the heat treatment, it may still be shaped by ordinary wood-working methods.

Pieces of wood may be given approximate final shape before placing in the retort, since the shrinkage within the retort is uniform and no subsequent drying or curing operation is necessary. The lower density of the modified wood and the smaller volume changes which it undergoes when subjected to changes in atmospheric humidity make the product valuable for cabinet making. Its greater porosity increases its value as a heat insulator. When thin slabs of wood or veneers are given this treatment, the resultant modified wood lends itself to a stable laminated product because of the ready absorption of the adhesive and the ability of the wood to withstand short exposures to 250 C. in the curing press without injury.

The modified wood is less liable to decay and attack by insects because of the low water absorption in damp air and because the heat treatment has destroyed most of the pentosans and similar products which furnish the most available food for many varieties of molds, bacteria and insects.

The color of the modified wood has been darkened uniformly throughout the cross section of the piece by the heat treatment, so that a piece, after heat treatment, may be turned or planed without the development of inequalities in color.

The wood may be introduced into the retort in the form of veneers, chips or sawdust. These pieces of small cross section require only a relatively short time in the retort, and may be heated to a somewhat higher final temperature than the larger pieces. Sawdust and chips are especially suitable forms of raw material when wood flour is to be the final product, since the heat-treated product pulverizes readily. The resultant wood flour is especially adapted for incorporation in phenolic resins and other plastics, because of the porosity, rigidity and small volume changes of the particles with variations in atmospheric humidity, and their stability at curing temperatures somewhat above 250° C.

What I claim as my invention is:

1. The process for producing a partial pyrolysis of wood which comprises externally applying heat to the wood in a retort thereby raising the temperature of the wood to above 180° C., continuously flowing through said retort, when the temperature of the wood has reached 150 to 180° C., an atmosphere containing at least 90% of steam at a pressure not substantially above atmospheric thereby continuously removing the volatile products liberated from the wood and also thereby preventing a temperature rise above 275° C. due to exothermic reaction, and maintaining the temperature of said wood between 180-275° C. for a time sufficient to produce at least 2% of acids calculated as acetic acid on weight of dry wood with no appreciable amount of methanol and to reduce the pentosans by at least 50% and cellulose at least 20% of the dry weight of said wood while retaining the lignins and at least 50% of the compressive strength of the wood.

2. The process according to claim 1 in which the continuously flowing atmosphere is superheated steam.

3. The process for producing a partial pyrolysis of wood which comprises externally applying heat to the wood in a retort thereby raising the temperature of the wood to approximately 240° C., continuously flowing through said retort, when

the temperature of the wood has reached not more than 220° C., an atmosphere containing at least 90% of steam at a pressure not substantially above atmospheric thereby continuously removing the volatile products liberated from the wood and also thereby preventing a temperature rise above 275° C. due to exothermic reaction, and maintaining the temperature of said wood at approximately 240° C. for a time sufficient to produce at least 2% of acids calculated as acetic acid on weight of dry wood with no appreciable amount of methanol and to reduce pentosans by over 50% and cellulose at least 20% of the dry weight of said wood while retaining the lignins and at least 25% of the compressive strength of the wood.

4. The process according to claim 3 in which the continuously flowing atmosphere is superheated steam.

5. The process for producing a partial pyrolysis of wood which comprises externally applying heat to the wood in a retort thereby raising the temperature of the wood to approximately 240° C., continuously flowing through said retort, when the temperature of the wood has reached not more than 220° C., superheated steam at substantially atmospheric pressure thereby continuously removing the volatile products liberated from the wood and also thereby preventing a temperature rise above 275° C. due to exothermic reaction, and maintaining the temperature of said wood at approximately 240° C. for a time on the order of ten hours.

6. The process for producing a partial pyrolysis of wood which comprises externally applying heat to the wood in a retort thereby raising the temperature of the wood to above 180° C., continuously flowing through said retort, when the temperature of the wood has reached 150 to 180° C., an atmosphere containing at least 90% of steam at a pressure not substantially above atmospheric thereby continuously removing the volatile products liberated from the wood and also thereby preventing a temperature rise above 275° C., due to exothermic reaction, maintaining the temperature of said wood between 180-275° C. for a time sufficient to produce at least 2% of acids calculated as acetic acid on weight of dry wood with no appreciable amount of methanol and to reduce the pentosans by at least 50% and cellulose at least 20% of the dry weight of said wood while retaining the lignins and at least 50% of the compressive strength of the wood, condensing out certain of said volatile products from said atmosphere at a temperature not substantially below the boiling point of water, recirculating a portion of the atmosphere containing uncondensed steam through said retort and condensing out from the remaining portion of said atmosphere the condensable volatilized products from said wood.

7. A modified form of wood produced by the partial pyrolysis of natural wood, free from the volatile products of decomposition from said partial pyrolysis, having 75% less pentosans and 20% less cellulose than the original wood, containing as much lignin as the original wood, having at least 50% of the original compressive strength, having a higher resistance to decay and characterized by substantially smaller volume changes when exposed to humid conditions than the original wood.

ALFRED H. WHITE.