

PROCESS FOR EXTRACTING TURPENTINE AND ROSIN FROM RESINOUS WOODS.

992,325.

D. C. Walter
Leona Kibertz

INVENTOR:
J. Homer J. Gurney,
J. H. Hall, N. Y.

UNITED STATES PATENT OFFICE.

HOMER T. YARYAN, OF TOLEDO, OHIO, ASSIGNOR TO THE YARYAN NAVAL STORES COMPANY, OF TOLEDO, OHIO, A CORPORATION OF OHIO.

PROCESS FOR EXTRACTING TURPENTINE AND ROSIN FROM RESINOUS WOODS.

992,325.

Specification of Letters Patent.

Patented May 16, 1911.

Application filed June 10, 1908. Serial No. 437,662.

To all whom it may concern:

Be it known that I, HOMER T. YARYAN, a citizen of the United States, residing at Toledo, in the county of Lucas and State of Ohio, have invented certain new and useful Improvements in Processes for Extracting Turpentine and Rosin from Resinous Woods; and I do declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same, reference being had to the accompanying drawings, and to the letters and figures of reference marked thereon, which form a part of this specification.

In removing turpentine from the logs, stumps, and various parts of certain pines, firs, larches and other resinous woods the method heretofore employed is to either reduce the wood to small chips which are then subjected to the action of live steam, whereby a comparatively small portion of the turpentine, but not resin, is distilled and saved, or to submit the woods to destructive distillation in retorts, whereby only impure turpentine and tar are recovered.

My invention relates to and its object is to provide a process for overcoming the objections to the methods above indicated, and, more particularly, to furnish methods by means of which all of the turpentine and resin may be quickly and cheaply recovered from resinous woods, and by means of which such woods shall be rendered fit for subsequent use in the manufacture of wood pulp.

A further object of my invention is to provide safeguards against the dangers arising from the explosive character of the vapors generated in my apparatus in practicing my process.

To this end, the resinous wood to be treated is fed to a cutting machine or "chipper" which reduces the wood to small thin chips, cut across the grain of the wood. These chips are placed in a stout iron tank or extractor having a door at top through which material is introduced into the chamber of the vessel, and having also a door near the bottom through which the exhausted chips are removed. The vessel has, near its bottom, a perforated false bottom or diaphragm upon which the chips rest and directly beneath which is a steam coil supplied with high pressure live steam to maintain

the proper temperature in the extractor during the process. Superheated steam is now injected into the mass of chips contained in the closed chamber of the extractor. Substantially all of the lighter turpentines are thus vaporized and pass over with the steam to a condenser leaving in the wood the heavier turpentines and oil of pine. The water and turpentine being separated, either by gravity or distillation, the turpentine is discharged into a tank. Thus far the mass of chips has given up only a portion of its turpentine and none of its resin. The chips are now hot and in an extremely porous state. A partial vacuum is next,—by means of a condenser or vacuum-pump, or both,—produced in the extractor, thus removing any excess of moisture, still further opening the pores of the wood and facilitating extraction in the further progress of the process. The chips are now treated with a liquid hydrocarbon, by spraying the same liberally upon the top of the hot mass and allowing it to percolate through to the bottom of the extractor.

In my process the naphtha and benzin of commerce may be employed, but in my method I am enabled to use and prefer to avail myself of the heavier hydro-carbons having a boiling point of about 240° to 270° F. The advantages of the use of the heavier hydrocarbons are their economy in first cost and comparative freedom from highly explosive vapors, as well as their higher boiling points which facilitate the melting of the rosin and the separation of the turpentine. In the process which I have devised in which the lighter turpentines are first removed by steam, the turpentines remaining in the wood have a boiling point extremely high, say, from 340° to 360° F., and I am therefore enabled to remove these with a heavy hydro-carbon and to afterward separate by distillation, owing to the great difference in the boiling point of the solvent and the turpentine. The bath of hot hydrocarbon, in its course, quickly and completely penetrates all the woody particles, and dissolves and washes out from the hot and highly porous chips and carries with it all the remaining turpentine and resin. From the bottom of the extractor the hydrocarbon solvent with its turpentine and resin is conducted to a still where the separation of these three constituents is effected: A par-

tial vacuum is now, by any suitable means, produced in the still to expedite the operation, to lower the boiling point of the mixture and to prevent undue heating of the product. The solvent, although it may be of nearly the same specific gravity as the turpentine, yields readily and, passing over as vapor to a condenser, is first recovered and conveyed to its tank to be again used as required. The turpentine and rosin, however, cling stubbornly together notwithstanding these are at the boiling point within the still. Next a stream of live steam is injected into the heated mixture. This has the effect of rapidly and completely separating the turpentine from the rosin. The turpentine vapor and steam, passing over to a condenser, are condensed and separated, the turpentine being discharged into its tank. The melted rosin, which is now freed from the solvent and turpentine, is next withdrawn from the still into barrels. The chips are next again steamed to recover the residue of the solvent and are again subjected to a vacuum to remove condensed steam. The chips are now nearly dry and are ready for use in the manufacture of wood pulp. Because of their entire freedom from resin, these chips yield readily to either the caustic or sulfite process.

The process above described and the apparatus herein illustrated and explained, assumes for convenience, that but one extractor is to be employed. In practice, however, I use a series of several tanks or extractors connected together in such fashion that they may be used in groups of two or three or more at once, as may be desired, the arrangement of the apparatus being such that the groups may be caused to progress, step by step, through the whole series. Some of the advantages of this arrangement are: 1st., economy of fuel, since the superheated steam after passing through the wood chips in the first extractor in a group may pass through a second or third member of the group without waste of its heat, the necessary temperature of the steam being maintained by the live steam coils in the bottom of the extractor; 2d., economy in the quantity of hydrocarbon to be recovered by distillation as well as the number of operations necessary for the separation of the hydrocarbon the turpentine and the resin, for, manifestly, the same hydrocarbon may be used for a group of extractors, the liquid becoming richer as it proceeds through the series, so that one distillation answers for all the members of the group, and 3d., economy of time and labor, because when the material in the first extractor in the group is exhausted this extractor is cut out from the group, to be now emptied and refilled, while at the same time a freshly filled waiting extractor is added to and becomes last

in the group. Thus the active group of extractors progresses constantly through the series, the extractors being successively disconnected from the group, emptied and filled as fast as their contents become exhausted, while a freshly filled extractor is added to the group, so that the process becomes practically continuous.

In practicing my process with the lighter hydrocarbons certain parts of the apparatus when partly emptied contain air saturated with hot vapor of the solvent, which mixture is highly explosive. To guard against the dangers of explosion, in such case, I connect the tanks and other parts of the apparatus in which this explosive mixture may be present with a gasometer or gas-holder in which the air is displaced by some gas having no free oxygen, such for instance, as CO or CO₂ or nitrogen. The parts referred to being charged with this inert gas, the air being excluded and the gas being retained and used over and over, the danger of explosion is reduced to a minimum. In case of fire, no explosion would occur within the tanks, as would be the case if an inert gas were not substituted for the air. An important additional advantage in the use of a gasometer or gas-holder is that it accommodates itself to the variations of the volume of gases and vapors in the apparatus, and preserves a uniform pressure in the various parts of the apparatus with which it is connected.

While ordinarily I prefer to practice my process by the use of the successive steps above enumerated, I do not limit my invention to a process which includes all these steps, as it will be understood that the bathing of the wood with a hot liquid hydrocarbon solvent in connection with the preliminary distilling off the lighter turpentines by steam, which is entirely novel, if continued for a sufficient time with an adequate amount of the solvent will extract the turpentine and the resin. Therefore the vacuum steps above referred to, while highly useful and important, are not indispensable, and it should be understood that my invention is broad enough to comprise the steps of treating woods with steam to remove the lighter turpentines and then with a hot hydrocarbon solvent for the extracting of their turpentines and oils of pine and rosin. Neither do I limit my invention to the use of a hydrocarbon solvent as it will be understood that, although I prefer to employ this solvent, other neutral volatile solvents may be used in lieu of a hydrocarbon solvent. By the use here and in the claims of the term "neutral" it is understood to define substances having neither an acid nor an alkaline reaction.

The above process may be practiced by means of the apparatus illustrated dia-

grammatically in the accompanying drawings in which the single figure is a side elevation of my apparatus with some of the parts, (such as the gas holder, which will be
5 clearly understood without illustration,) being omitted for the sake of clearness.

In the drawings, 1 is a cylindrical upright iron tank or extractor, closed at top and at bottom, and provided at top with a
10 door 2, through which the extractor is charged, and near the bottom with a door 3 through which the extractor is emptied. These doors when closed are steam tight. The extractor is provided near its bottom
15 and just below the door 3 with a foraminous false bottom 4. In the space between this false bottom and the bottom proper is a steam coil 5 connected by a pipe 6 to boiler 7.

20 8 is a suitably valved steam pipe leading from super-heater 9 to the chamber of the extractor 1, by means of which steam may be blown through the contents of the chamber.

25 10 is a hydrocarbon tank from which leads pipe 11 and which is connected with a spraying device 12 in the chamber of the extractor near its top.

Leading from the bottom of the extractor
30 is a pipe 13 connected with a tank 14 which is, in turn, connected with a pump 15. This pump is arranged to withdraw the contents of the tank 14 and to discharge the same through pipe 16 into still 17. From the top
35 of the chamber of the extractor 1 leads a pipe 18 to a condenser 19 connected with a turpentine tank 20. A pipe 21 connects the top of the chamber of the extractor with a vacuum condenser 22. A pipe 23 connects
40 the top of the chamber of the extractor with another condenser 24, this condenser being also connected by pipe 25 with the still 17. A vacuum pump 26 is connected with the still 17 and with the vacuum condenser 22
45 through pipes 27 and 28. A pump 29 is connected with the condensers 22 and 24 through a suitably valved pipe 30 and is arranged to pump the condensations from these condensers back to the solvent tank
50 10 through the pipe 31, or elsewhere, as may be desired.

The chamber of the extractor being charged with the comminuted wood to be treated and the doors being tightly shut,
55 steam is admitted to the coil 5 so that the mass becomes heated. Steam is now blown through pipe 8 through the mass, thus vaporizing the lighter turpentine. The vapors pass over through pipe 18 to the condenser 19 where the water and turpentine are separated by gravity or otherwise, the turpentine being drawn finally into the
60 turpentine tank 20. The jet of steam is now discontinued. Next the chamber of the
65 extractor is connected with the vacuum con-

denser and pump 22—26 and the contents of the extractor are by these means largely freed from moisture and the pores of the wood are still further opened. The hydrocarbon solvent is next admitted through pipe
70 11 to the spraying device in the top of the chamber of the extractor. The sprayed solvent percolates down through the chips, which become thoroughly bathed and saturated, and flows out through pipe 13 into
75 tank 14. The hot hydrocarbon dissolves and carries with it the remaining turpentine and the rosin, the flow of the solvent continuing until the chips are thoroughly exhausted. From the tank 14 the pump 15 passes the
80 solution through the still 17 from which the hydrocarbon vapor first passes over through pipe 25 to the condenser 24 from whence it is taken by pump 29 and returned to the tank
85 10 to be again used. The turpentine vapor is next liberated and is conducted through pipe 18^a to condenser 19 and thence to the turpentine tank 20. There now remains in the still rosin which is drawn off from the
90 still into barrels, as at 32. The vacuum pump 26 is connected by pipe 27 to the coil of the still 17. The partial vacuum produced in the still by this pump hastens the action of the still, lowers the boiling point
95 of the contents of the still and prevents their undue heating. A steam pipe 33 is also connected with the coil of the still 17 through which pipe may be furnished a steam jet to promote the rapid separation
100 of the turpentine from the rosin after the recovery of the solvent. The chips are next again steamed to recover the residue of the solution and are again subjected to a vacuum through the pipes 21, vacuum
105 condenser 22 and pump 26 to remove the condensed steam. The chips are now clean and nearly dry and are ready for use in the manufacture of wood pulp.

Having described my invention, what I claim and desire to secure by Letters Patent is,—

1. The process of recovering turpentine and rosin from resinous woods which comprises distilling the wood with steam, then
115 interrupting such distillation when the lighter turpentine have been recovered, and then extracting the remaining turpentine and the rosin with a neutral volatile solvent.

2. The process of removing turpentine and rosin from resinous woods, which consists in confining small fragments of the wood within a suitable chamber; then subjecting the wood to the action of steam to remove a portion of the turpentine; then
120 producing a partial vacuum in the chamber to remove excess of moisture, and then treating the wood with a hydrocarbon solvent to remove the rosin and the remaining turpentine.

3. The process of removing turpentine
130

and rosin from resinous woods, which consists in confining the wood in comminuted form within a suitable chamber; then treating the wood with steam to vaporize
5 and remove a portion of the turpentine; then treating the mass with a hot hydrocarbon solvent, and then distilling the mixture thus obtained to separate the solvent the turpentine and the rosin.

10 4. The process of removing turpentine and rosin from resinous woods, which consists in confining the wood in comminuted form within a suitable chamber; then treating the wood with steam under a partial
15 vacuum to remove the lighter turpentines; then treating the mass with a hot hydrocarbon solvent; then recovering the solvent from the solution thus obtained, and then separating the turpentine from the residue.

20 5. The process of removing turpentine and rosin from resinous woods, which consists in confining the comminuted wood within a suitable chamber; then steaming the wood to remove the more volatile tur-
25 pentines; then treating the mass with a hot hydrocarbon solvent; then distilling off and recovering the solvent from the solution thus obtained; then by further distillation separating the remaining turpentine from
30 the rosin, and then further treating the wood with steam to vaporize and recover from the mass the residue of the solvent.

35 6. The process of removing turpentine and rosin from resinous woods, which consists in confining the wood in finely subdivided form within a suitable chamber; then subjecting the wood to the action of steam to remove a portion of the turpentine; then
40 treating the wood with a hot hydrocarbon solvent to remove the rosin and the remainder of the turpentine, and then treating the wood with steam to recover the residue of the solvent, and, finally, removing the condensed steam by means of a vacuum.

45 7. The process of removing turpentine and rosin from resinous woods, which consists in confining the wood in comminuted form within a suitable chamber; then passing through the mass a volume of steam to
50 vaporize a portion of the turpentine; then condensing and separating the steam and turpentine vapors; then treating the mass with a hydrocarbon solvent to remove the rosin and the remaining turpentine, and then

distilling the mixture thus obtained to separate the solvent, the turpentine and the rosin. 55

8. The process of removing turpentine and rosin from resinous woods, which consists in confining the wood in comminuted
60 form within a suitable chamber; then treating the wood with steam to remove a portion of the turpentine; then causing a partial vacuum within the chamber to remove excessive moisture; then subjecting the wood
65 to the action of a hot hydrocarbon solvent to remove the rosin and the remaining turpentine, and then again treating the wood with steam to recover the residue of the solvent. 70

9. The process of removing turpentine and rosin from resinous woods, which consists in first treating the wood in comminuted form with steam to recover the lighter
75 turpentines; then treating the wood with a hydrocarbon solvent, to dissolve the turpentine and rosin, and then by fractional distillation under a partial vacuum recovering the solvent from the solution and separating the
80 turpentine from the rosin. 85

10. The process of removing turpentine and rosin from resinous woods, which consists in treating the wood with steam to remove a portion of the turpentine; then
85 treating the wood with a hydrocarbon solvent to dissolve the remaining turpentine and rosin; then by distillation recovering the solvent; then by further distillation separating the turpentine from the rosin, and, during the latter step, blowing a jet
90 of steam into the material under treatment. 95

11. The process of removing turpentine and rosin from resinous woods, which consists in treating the wood with steam; then
95 treating the wood with a hydrocarbon solvent to dissolve the turpentine and rosin; then by distillation recovering the solvent; then by further distillation under partial vacuum separating the turpentine from the rosin, and, during the latter step, blowing
100 a jet of steam into the material under treatment.

In testimony whereof I affix my signature in presence of two witnesses.

HOMER T. YARYAN.

Witnesses:

C. S. ROWLEY,

ADA E. CAMERON.