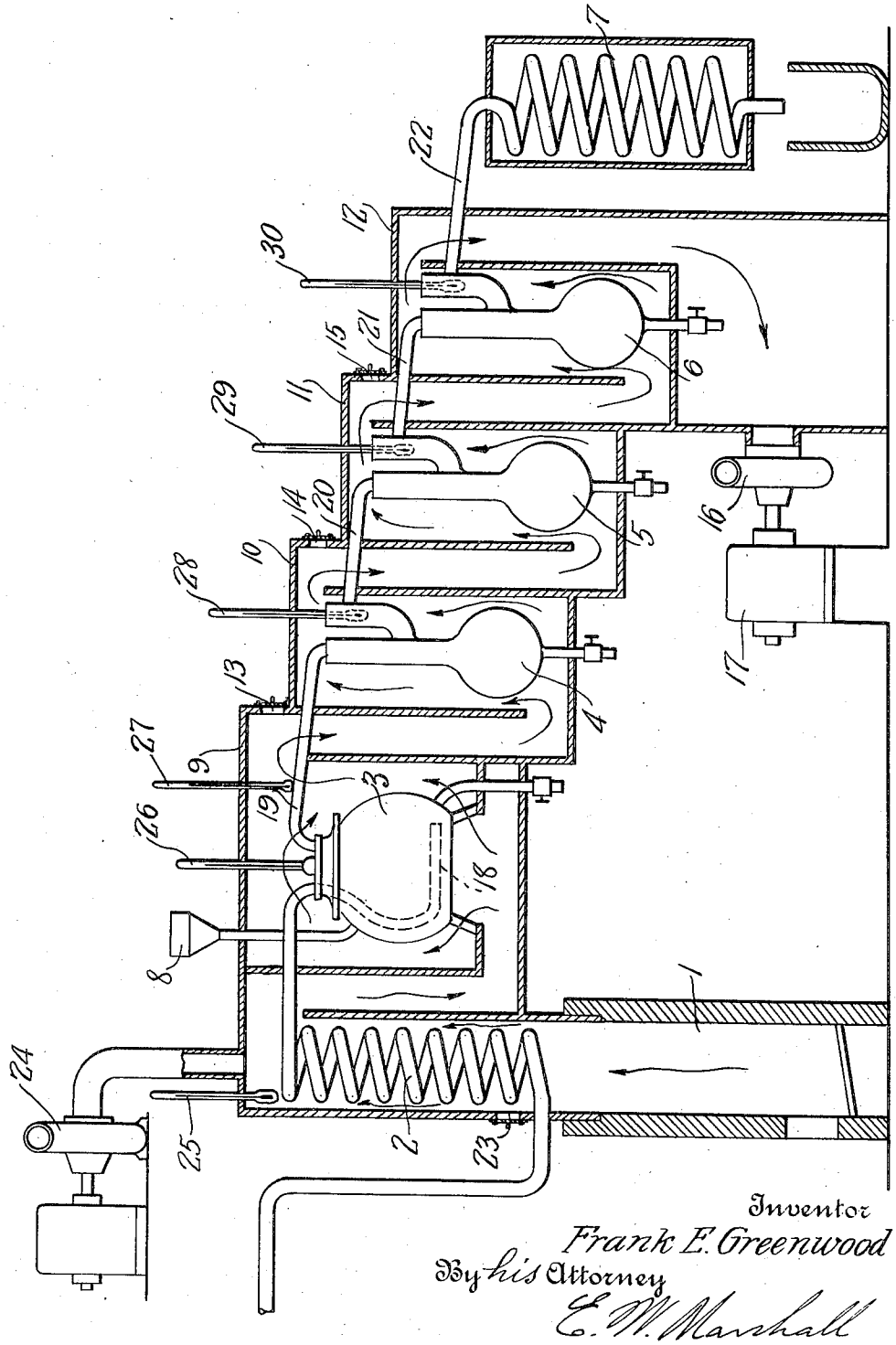
July 27, 1926.

1,593,656

F. E. GREENWOOD

DISTILLATION OF RESIN

Filed August 25, 1920



Inventor

Frank E. Greenwood

By his Attorney

E. W. Marshall

Patented July 27, 1926. 1,593,656

UNITED STATES PATENT OFFICE.

FRANK E. GREENWOOD, OF NEW ROCHELLE, NEW YORK, ASSIGNOR TO PINE WASTE PRODUCTS, INC., OF NEW YORK, N. Y., A CORPORATION OF DELAWARE.

DISTILLATION OF RESIN.

Application filed August 25, 1920. Serial No. 405,923.

The present invention relates to the recovery of rosin and rosin oils from the so-called "rosin soap" derived from the black liquor of pulp mills. This material contains various acid resins, as their sodium salts or resinates, as well as various neutral resins and terpenes emulsified thereby.

Heretofore, this rosin soap, though containing a good proportion of real "rosin", as well as many other resins, has been generally considered and treated as a waste product.

The objects of my invention are to utilize this rosin soap and to provide a commercially feasible and economical method and suitable apparatus for extracting these valuable resinous products.

According to an advantageous method the rosin soap is obtained from the pulp mill black liquor by a separation process, involving usually simply a skimming operation, in which advantage is taken of the fact that the rosin soap being lighter, rises to the surface of the pulp mill black liquor and can then be removed by simply skimming it off the top. After such separation the rosin soap, is first treated with an acid (for instance, sulphuric) to transform the alkali resinates and set free their acids. This product is then heated and subjected to the action of superheated steam. The steam exerts a selective distilling effect, separating the rosin and rosin oils from other matters contained in the rosin soap. The extracted matter is suitably collected as by means of a series of condensers.

In the accompanying drawing I have illustrated more or less diagrammatically a form of apparatus designed for the practice of the invention, but as this illustration is primarily in the nature of a flow sheet intended for the purposes of disclosure, I would have it understood that the invention is not limited to this particular form of apparatus.

In the illustration, 1 designates a furnace or heater, 2 a superheater coil, 3 a retort in the nature of a still, 4, 5 and 6 a series of air condensers, and 7 a water cooled condenser.

The rosin soap after acidification and separation of the aqueous solution of salts, or of some of this solution, is charged into the retort 3, as by means of a supply hopper 8 which may be steam or otherwise heated to cause the crude resinous matter to feed into the retort.

The retort is externally heated as by being encased within a chamber 9 receiving the waste gases from the furnace, the arrows in the illustration indicating the direction of flow to and about the retort.

In similar fashion, the air cooled condensers are shown as encased in chambers 10, 11 and 12 forming continuations of the waste gas conduit. The heating of these condensers is preferably controlled to prevent condensation of the steam, while permitting condensation of the products which it is desired to collect. The selective control of the heat in the several chambers is obtained in the illustration by means of cold air inlet dampers 13, 14 and 15, and by means of a suction fan 16 applied to the outlet of the last condenser chamber. This fan is shown as driven by a variable speed motor 17, and therefore as capable of being regulated to vary the flow through the retort and condenser chambers. By regulating this flow and the several cold air inlets, the temperatures in the several chambers may be accurately governed.

The steam from the superheater coil is admitted into the bottom portion of the retort through a perforated pipe 18. The retort and the condensers are connected in series by the successive connections indicated at 19, 20, 21 and 22.

The temperature of the retort chamber is controlled by governing the heat of the furnace and is further shown as controlled by means of a cold air inlet damper 23 and an exhaust fan 24 which may be used to bypass the heat away from the chamber.

Thermometers are shown at 25, 26 and 27 for indicating the temperature of the superheated steam, the temperature within the retort and the temperature of the retort chamber. I have also shown thermometers at 28, 29 and 30 for indicating the temperatures in the respective air condensers.

At the proper temperature, distillation takes place and the vapors of rosin and rosin oil are carried from the retort over to the first condenser, this flow being made possible by the flow of superheated steam. In tests which I have made the retort was heated to approximately 300 degrees C., and the

steam, which was at slightly above atmospheric pressure, was heated to 340° C.

The temperature of the first condenser is controlled to cause the rosin which has the highest boiling point to deposit there by condensation and to cause the oils having lower boiling points to be carried on to the next condenser. This heat also is sufficient to prevent condensation of the steam at this point.

By the proper control of the temperature in the successive condenser chambers, rosin oils of various boiling points can be selectively condensed and collected, the steam and low boiling oils being finally condensed in the water cooled condenser at the end of the apparatus.

With this improved process and by means of the apparatus disclosed, I have obtained from the first condenser a good grade commercial light-colored rosin, and from the other condensers valuable rosin oils of different boiling points.

The superheated steam operates selectively in that it carries off the more volatile resin acids, which may be here called abietic acid, of the resin soap, leaving the less volatile and dark colored contaminating products as a residue. The more volatile abietic acid is thus in effect volatilized by the steam away from the contaminating products and is condensed in a purified condition. The residue may be drained from the retort, as required, by a suitable outlet drain, as indicated at 31.

In a companion application of mine filed of even date herewith, Serial No. 405,921, I have disclosed what is known as the plural-solvent method of extracting the valuable resinous constituents from rosin soap. In that method the rosin soap is first mixed with a solvent, such as gasoline, which is immiscible with water and which does not dissolve the soap but forms an emulsion therewith. To this emulsion there is added a mineral acid, such as sulphuric, which liberates abietic acid etc. and enables absorption by the solvent. This solvent carrying the abietic acid as well as certain other bodies which it is desired to separate from the abietic acid, is then mixed with a second solvent, such as alcohol, which is immiscible with the first and in which the abietic acid is readily soluble. This second solvent operates selectively to remove the abietic acid content from the first solvent and the refining process is usually continued by mixing fresh portions of the second solvent with the first solvent until a point is reached where fresh additions of the second solvent remove little or none of the abietic acid from the first solvent. The gasoline solution is then distilled to separate the gasoline from the residue, and, the alcoholic solution is similarly distilled to recover alcohol and a residue of purified resin acids.

My present invention contemplates the treatment of the residue of either or both of the solvents of my other application with the superheated steam for the purpose of directly extracting the abietic acid, or in other words, the rosin and rosin oils therefrom.

This modification simply involves the substitution in the retort of the residue obtained from the solvents for the crude resins of the rosin soap, as heretofore described, the operations and effects being generally similar.

I am aware that it has been proposed to refine certain entirely different materials, the various grades of commercial rosin by a steam treatment. This is disclosed in the United States patent to Hunt & Pochin, No. 22,558, January 1, 1859, but the present invention relates to the steam treatment of something else, the resinous matters recovered from black liquor.

What I claim is:

1. The process of recovering resin and the products thereof from pulp mill black liquor which comprises separating rosin soap from said liquor, treating said soap with a solvent and an acid, treating the resultant solution with a second solvent immiscible with the first, separating the solutions, evaporating the solvent from the second solution and subjecting the residue from the second solution to distillation in the presence of superheated steam.

2. The process of recovering resin and the products thereof from pulp mill black liquor which comprises separating rosin soap from said liquor, treating said soap with a solvent and an acid, treating the resultant solution with a second solvent immiscible with the first, separating the solutions, evaporating the solvent from the second solution, subjecting the residue from the second solution to distillation in the presence of superheated steam, and condensing the distillate in successive stages.

In witness whereof, I have hereunto set my hand this 23rd day of August, 1920.

FRANK E. GREENWOOD.