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H. E. KAISER ET AL

1,719,431

PROCESS OF REFINING WOOD ROSIN

Filed May 15, 1923

2 Sheets-Sheet 1

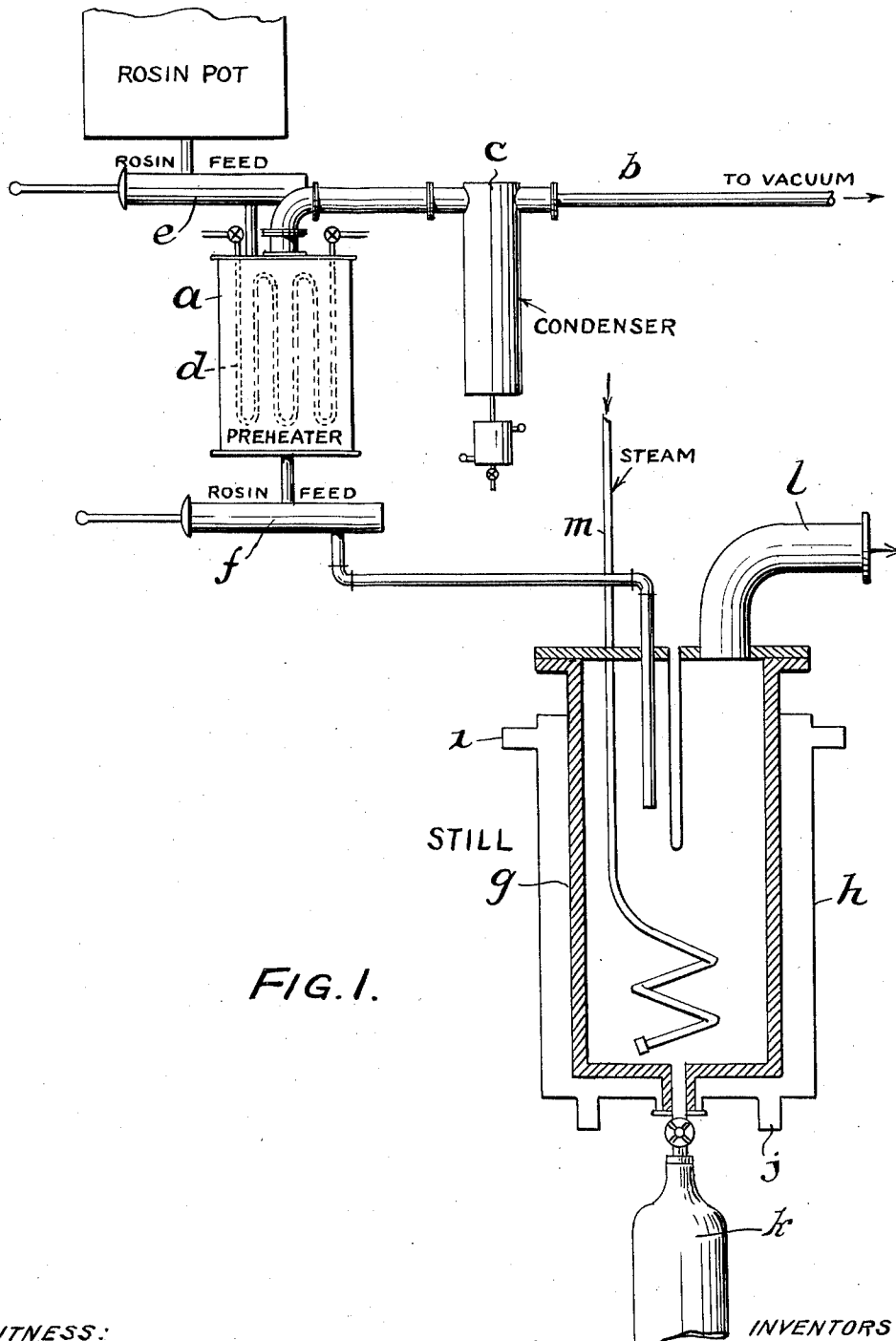


FIG. 1.

WITNESS:

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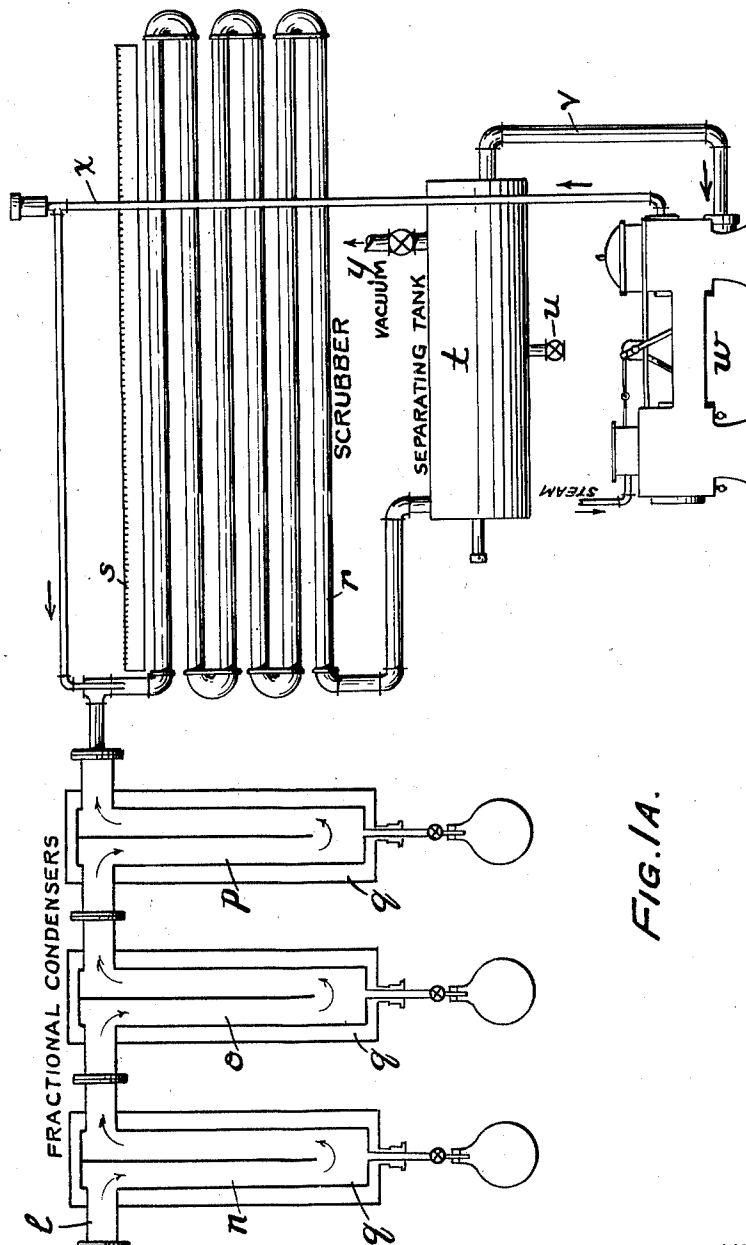


FIG. 1A.

WITNESS:

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UNITED STATES PATENT OFFICE.

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PROCESS OF REFINING WOOD ROSIN.

Application filed May 15, 1923. Serial No. 639,054.

The object of our invention is to so treat wood rosin as to provide a rosin so highly refined that it is adapted for use in sizing the higher grades of paper, in the manufacture of better grades of varnish, and for other purposes for which a high grade rosin is adapted.

A known process of producing wood rosin of a lower grade, and the process that we preferably employ as a preliminary process in the manufacture of our more highly refined rosin, consists in grinding wood and stumps from pine trees, placing the wood stock in large digesters, passing steam through the stock and thus driving off a considerable quantity of turpentine, flooding the stock with gasoline or other solvent, thereby extracting certain materials, chiefly rosin, pine oil and various color bodies, passing the gasoline solution, commonly known as "drop liquor", through suitable evaporators, and fractionally removing the gasoline and pine oil. The pine oil produced by the distillation is refined and sold. The recovered gasoline is again used in the process.

The recovered rosin is a marketable product, but is satisfactory for sizing only the lower grades of paper and in making the inferior grades of varnish, and therefore must be marketed at a relatively low price.

In our process we subject this lower grade rosin to a treatment which greatly improves the color of the rosin and imparts to it generally such high quality, both chemical and physical, as to adapt it for sizing the higher grades of paper and for use in the better grades of varnish.

To secure the best results we prefer to preliminarily distill the rosin to remove some of the objectionable coloring matter. This preliminary distillation is not essential in order to secure valuable results, but such preliminary distillation insures a perhaps higher grade ultimate product and a conversion of a larger proportion of the rosin into such higher grade ultimate product.

After such preliminary distillation, the rosin is subjected to the direct action of steam in a still under a vacuum of within about two inches of the normal barometric pressure of mercury column. The rosin is at the same time heated by means of oil maintained at a temperature of from 500° to 650° F.—preferably within the higher part of this range.

The distillation may be in batch or continuous. If in batch, the still may be surrounded by a jacket in which a circulation of the hot oil is maintained. If the continuous process is used, the rosin may flow over coils of pipe through which the hot oil is circulated.

The still vapors are then passed through fractional condensers. Proper temperature control of the condensers has an important bearing on the quality of the recovered rosin. We have found that when the condensers are operated at a temperature of about 350° F. or over, a rosin is condensed that is comparatively free from rosin oils and pine oils. At this temperature a water-free product results and there is little danger of the condensed rosin crystallizing. Thus, if the rosin is passed through three condensers in which temperatures of about 400° F., 350° F. and 300° F. are maintained, the rosin that condenses in the first condenser is of a very high quality and suitable for sizing the highest grade paper and for use in the very best varnish. The product of the second condenser is a good rosin, while the product of the third condenser is of lower grade.

It is impossible, in these condensers, to condense all the rosin and rosin oil and we have therefore designed a scrubber for use at the end of the condensing system. This scrubber comprises a number of tubes, arranged in series, which receive the uncondensed rosin vapor or mist and through which circulates a stream of gasoline, kerosene or other solvent. During the flow through the tubes, they are subjected to the action of cold water. In these tubes the steam, rosin and rosin oil vapor are condensed. The tubes are connected with a tank in which the water and gasoline solution separate. The water is drawn off and the gasoline pumped back to the solvent line. After the gasoline solution approaches the saturation point, it is separated from the rosin by distillation.

The above is a general description of the entire process, but a more detailed description may facilitate the practice of the process by those skilled in the art.

In the drawings, which illustrate apparatus for carrying out the major part of our process in its preferable form,—

Fig. 1 is a diagrammatic view of that part of the complete rosin refining apparatus comprising the preheater and still.

Fig. 1A is a similar view of the remainder of the apparatus, comprising the fractional condensers and scrubber.

The rosin is first heated in a preheater *a* having a vacuum connection *b* by way of a small condenser *c*. The preheater may be effectively heated by means of a coil *d* through which heated oil is circulated. There are two automatic feeds, one *e* into the preheater and the other *f* from the preheater to the still. The rosin should be heated in the preheater to a temperature of about 480° F. By preheating the rosin, any low boiling coloring matter that may be in the rosin as it is introduced into the system is taken off and condensed. As different rosins have varying proportions of this low boiling coloring matter, the preliminary distillation will insure substantial uniformity in the material that passes to the main still.

The still *g* is preferably of iron lined with aluminum and may be heated in any convenient manner, such as, preferably, by heated oil circulated through coils or, as shown in the drawing, through a jacket *h*. The oil should be at such temperature as to heat the rosin to a temperature well over 500° F. and preferably about 600° or 650° F. *i* is the oil inlet and *j* the oil outlet. The bottom of the still is connected with a vessel *k* to receive residue. It is preferred to inject superheated steam into the body of the rosin in the still, although this improvement in the process is no part of our invention, having been conceived by George E. Jenks, who has filed an application for patent therefor, Serial No. 639,062, dated May 15, 1923. For this purpose, a steam pipe *m* extends into the still. Through this pipe superheated steam is discharged into the body of the rosin in the still.

The still *g* is adapted to distill the rosin in batch, but it is equally practicable, and perhaps preferable, to distill the rosin continuously. In view, however, of the fact that the invention is not dependent upon the use of any particular apparatus, illustrations of the different constructions of stills that may be practicable to use is not required in order to fully explain the process and enable those skilled in the art to satisfactorily practice it.

The rosin vapors are carried off by a pipe *l* through a succession of fractional condensers, which may be of any number, preferably three or four. Three are shown and marked *n*, *o* and *p*. Each condenser is maintained at a predetermined temperature by means of a surrounding jacket *q* in which heated oil is circulated. It is preferred to maintain the three condensers *n*, *o* and *p* (where three are used) at temperatures of 400° F., 350° F. and 300° F. respectively.

A high degree of vacuum seems essential to successful rosin distillation. A vacuum of twenty-eight inches of mercury column (if the normal atmospheric pressure is about

thirty inches) has been used successfully. It is obvious that with a still higher degree of vacuum the minimum temperature of distillation may be lowered (say 25° F.) When the vacuum drops below twenty-seven inches at the atmospheric pressure specified, the distillation is sluggish. With lower atmospheric pressure, the degree of vacuum would be correspondingly higher. Leakage of air (or oxygen) into the still or condensing system should be avoided, as its effect is to discolor the rosin. The temperature of the superheated steam admitted through pipe *m* into the still should be quite close to that maintained in the still.

Rosin condensed in the first condenser *n* is of excellent color and of such high quality, both physically and chemically, that it can be used for sizing the highest grade paper, for making the best varnish, and for other purposes. The rosin condensed in the second condenser *o* is of good quality and commands a substantially higher price than the rosin introduced into the system. The rosin condensed in the last condenser *p* is inferior in quality to that condensed in the other condenser. To produce really high grade rosin, the temperature of the condenser should not be less than 350° F. and should be nearer 400° F. At this temperature is produced a water-free product comparatively free from rosin oil and pine oil, and there is little danger of the condensed rosin crystallizing. By increasing the number of condensers, or by heating the condensers to temperatures different from the temperatures herein specified, or by both means, a smaller proportion of the very highest grade rosin, or a larger proportion of rosin of somewhat lower, but still high, grade may be secured. The number of condensers and the temperature regulation will depend upon the grade of rosin that it is desired to get and particularly the purpose for which the rosin is intended to be used.

The rosin and rosin oil vapors that escape from the last condenser pass to the solvent scrubber shown. These vapors, which are essentially a mixture of rosin and rosin oil, probably do not amount to over two or three per cent of the rosin distilled. The vapors pass through the return bend pipe condenser *r*, which is water-cooled by means of a distributor *s*. A recirculating stream of solvent (gasoline or kerosene) is maintained inside of the condenser pipes. Without this hydrocarbon solvent, the rosin that would condense in the pipes *r* would not be in sufficient quantity or sufficiently fluid to flow freely. During any single traverse of the condenser pipes, the solvent dissolves much less rosin and rosin vapor than it is capable of dissolving, and it is therefore recirculated until it becomes saturated. The solution of hydrocarbon oil and rosin and rosin oil, mixed with water of condensation, passes to a solvent settling or sep-

arating tank *t*, wherein the water settles to the bottom and is withdrawn through the valved pipe *u*. The hydrocarbon oil, with rosin and rosin oil dissolved therein, over-
5 flows into a pipe *v* and, by means of a pump *w* is returned through a pipe *x* to the pipe condenser *r*. As the solvent becomes saturated it is replaced with fresh solvent.

The advantage of the scrubbing operation
10 is not primarily to save the small quantity of rosin or rosin oil recoverable thereby, but to prevent the condensation of rosin mist in the vacuum lines, which would result in clogging up the vacuum lines and prevent regular op-
15 eration.

The vacuum in the apparatus may be maintained by connecting the settling tank *t*, through pipe *v*, with vacuum.

The main advantages of our process arise
20 from the preheating and preliminary distillation (preferential) of the rosin, the fractional condensation, the condensation at a temperature above 350° F., and the avoidance of condensation of rosin and rosin oil
25 vapor or mist in the vacuum lines.

Having now fully described our invention, what we claim and desire to protect by Letters Patent is:

1. The process of refining wood rosin which
30 comprises heating the rosin to a temperature within about 500–650° F. while under a vacuum corresponding to an absolute pressure not higher than about three inches mercury and condensing a portion of the rosin at a
35 temperature not lower than 350° F.

2. The process of refining rosin which comprises heating the rosin under a vacuum corresponding to an absolute pressure not higher than about three inches mercury and to a tem-
40 perature within about 500–650° F. and fractionally condensing the rosin at a plurality of temperatures adapted to yield rosin of different qualities.

3. The process of refining rosin which com-
45 prises heating the rosin under a vacuum corresponding to an absolute pressure not higher than about three inches mercury and to a temperature within about 500–650° F. and condensing a portion of the rosin at a tempera-
50 ture approximating 400° F.

4. The process of refining rosin which comprises heating the rosin under a vacuum corresponding to an absolute pressure not higher than about three inches mercury and to a tem-
55 perature of within about 500–650° F. and fractionally condensing the rosin at temperatures varying from about 400° F. to about 300° F.

5. The process of refining rosin which com-
60 prises heating the rosin under a vacuum corresponding to an absolute pressure not higher than about three inches mercury and to a temperature of 550–600° F. and fractionally condensing the rosin at temperatures of
65 about 400° F., 350° F. and 300° F.

6. The process of refining rosin which comprises preheating the rosin to a temperature substantially above its melting point and sufficiently high to drive off lower boiling coloring matter and condensing such coloring mat-
70 ter, heating the rosin from which such coloring matter has been removed to a still higher temperature within about 500–650° F. and under a vacuum corresponding to an absolute pressure not above about three inches
75 mercury, and condensing a portion of the rosin at a temperature substantially above 350° F.

7. The process of refining rosin which includes preliminarily heating it to a tempera-
80 ture substantially above its melting point and sufficiently high to drive off lower boiling point coloring matter and condensing such coloring matter, vaporizing the rosin from which coloring matter has been removed by
85 heating it under high vacuum to a substantially higher temperature than that of the preliminary distillation and fractionally condensing the vaporized rosin.

8. In the process of refining rosin the steps
90 which include establishing an endless circulation of a hydrocarbon a solvent of rosin and during such circulation vaporizing rosin by heat under high vacuum, condensing part of the vapors for the recovery of high grade
95 rosin and passing the uncondensed vapors into the circulating solvent until it approaches saturation.

9. The process of refining rosin which comprises heating the rosin under a high vacuum
100 to a temperature within about 500–650° F., condensing at least a portion of the rosin at a temperature not less than 350° F., and absorbing at least some of the rosin that does not condense at that temperature into a
105 liquid hydrocarbon solvent.

10. The process of refining rosin which comprises heating the rosin under a high vacuum to a temperature within about
110 500–650° F., fractionally condensing the rosin at a plurality of temperatures the higher of which is substantially above 350° F., and eliminating the relatively small fraction of rosin and rosin oil mist or vapor from the
115 condensing system by absorption thereof in a liquid hydrocarbon solvent therefor, thereby avoiding the plugging of the vacuum line by the condensation of the relatively small fraction of said rosin and rosin oil that is not
120 fractionally condensed.

11. The process of refining rosin which comprises preliminarily heating the rosin
125 under a vacuum to a temperature substantially above its melting point and sufficiently high to drive off lower boiling point coloring matter, vaporizing rosin from which such coloring matter has been removed by heating
130 it under a high vacuum to a substantially higher temperature than that of the preliminary distillation heat, fractionally con-

densing the vaporized rosin, and eliminating the relatively small volume of vapors that is not fractionally condensed by absorption thereof in a liquid hydrocarbon solvent therefor to avoid the plugging of the vacuum line.

5 12. The process of refining rosin which comprises preliminarily heating the rosin to a temperature below 500° F. and sufficiently above its melting point to drive off lower boiling point coloring matter and condensing
10 such coloring matter, vaporizing rosin from which such coloring matter has been removed by heating it to a temperature substantially

above the temperature of the preliminary heat but not above about 650° F. and fractionally condensing the vaporized rosin, the lowest temperature of condensation being at least 350° F.; said operations being conducted under a vacuum corresponding to an absolute pressure not higher than about three inches
20 mercury.

In testimony of which invention, we have hereunto set our hands, at Kenvil, N. J., on this 11th day of April, 1923.

HARRY E. KAISER.

ARTHUR LANGMEIER.