DISTILLATION OF RESINOUS WOOD

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DISTILLATION OF RESINOUS WOOD

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Raw Material

The commercial distillation of resinous wood is carried on at widely scattered plants in the pine regions of the South and Southeast.

The wood generally used is obtained from longleaf and slash pine species. Live trees of these species are also tapped for the oleoresin from which gum turpentine and rosin are obtained. Similar products recovered by distillation or extraction of the wood are referred to as destructively distilled and steam distilled wood turpentines and wood rosin. Some plants process a small proportion of the more resinous wood from shortleaf and loblolly pines. Best results are obtained, however, by limiting the selection of raw material to longleaf and slash pine species. Since the marketable products obtained are chiefly derived from the resin in the wood, the most desirable raw material for the processing is wood of high resin content.

Stem stock or the average sawmill and logging waste from longleaf, slash, shortleaf, loblolly, and other pine species does not contain sufficient resin for satisfactory plant distillation. Only the very resinous or pitchy portions of the tree are commercially suitable. Such pitchy portions may be selected as they occur in wood operations or in the course of lumber production.

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1 The intent of this publication is to provide only the general information regarding the methods and products of the resinous-wood-distillation industry. This is a revision of an earlier Forest Products Laboratory report of the same title, by L. F. Hawley.

2 Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

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The resinous portions of fallen or felled trees like longleaf and slash pines, when allowed to remain upon the ground, resist decay indefinitely. The less resinous areas, particularly the sapwood, decay rapidly and disappear. The remaining resinous material is called "lightwood," "fatwood," or, in the case of the conically shaped pieces of pitchy wood from the base of the limbs, "pine knots." In the same way, sapwood and the less resinous portions of the heartwood of the stumps decay, leaving a core of pitchy heartwood. Waste log and limb lightwood, together with larger proportions of stump wood from old cut-over areas, comprise most of the raw materials for product recovery. Selected pitchy slabs and trimmings from pine-mill operations provide further limited amounts of raw material to the plants.

The resinous wood of other species, such as the red or Norway pine of the North and the Douglas-fir and ponderosa pine of the West and Northwestern States, has attracted attention as raw material for destructive distillation and solvent processes. Potentially large supplies of raw material from these species await possibly further developments in processing and wood-procurement methods. Expanded markets for the products might also lead to more economical production. Previous efforts to use mill and stump waste from these species have not been commercially successful. However, there has recently been renewed interest in the possible utilization of these woods as raw material for extraction and distillation. Fairly extensive pilot-plant extraction studies have been conducted for some time by one large company now operating on southern resinous woods. Other pilot-scale investigations at Oregon State College, have indicated further possibilities in the use of woods of lower resin contents, such as Douglas-fir mill waste, by modified destructive distillation, charcoal briquetting, and tar-recovery procedures.

Destructive Distillation of Southern Pine

Distillation Equipment

Both retort and oven-type distillation equipment is used in this industry. The retorts are cylindrical steel vessels set horizontally in brickwork with a fire box under one or both ends. They vary in capacities from 1 to 2 cords of wood and are charged and discharged through an end door. Retort volatiles are piped to condensers located to the side or to the rear of the retorts.

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The larger oven-type heating chambers are loaded with wood contained in
cars or "buggies," while the smaller retorts are charged and discharged by
hand. Some of the ovens are of concrete construction and are heated intern-
ally by full-length iron flues. Another type of oven is of boiler-plate construc-
tion. Heat is supplied by fireboxes located at one or both ends of the oven. In
some cases, also, with ovens of concrete construction, the bottom is not
heated directly and part of the crude liquid product is withdrawn from the
bottom of the oven. The remainder, in the form of volatiles, passes through
an outlet pipe to the condenser.

The kinds of fuel and the methods of firing vary at different plants. Natural
gas, oil, and coal are among the more common fuels used. Time periods of
24 to 48 hours are required to complete the distillations to an end temperature,
generally of about 800° F.

Products

The crude products obtained at most plants are the water-oil distillate, un-
condensable gas, and charcoal. The vapors and gases pass from the heating
chamber through a condenser for recovery of the distillate and separation of
the gases. The gases are piped for fuel use under the wood charge or to the
boiler plant, or are vented to the atmosphere. Upon standing, the crude total
distillate separates into a watery or pyroligneous acid layer and a lighter oily
layer. The oil layer contains the most important and valuable products. The
crude-oil distillate is not always obtained as a total product, but may be ob-
tained from the retort in various fractions. Plant procedure may include the
collection of the first oily distillate or crude turpentine separately at a com-
paratively low temperature followed by other products at progressively higher
temperatures. Another practice includes heating the bottom of the retort
only moderately high to allow collection of the less volatile products at this
point. Pitch or tar may be drawn off, depending upon the temperature ap-
plied. Whether the crude oils are collected as a whole or in fractions, re-
distillation in tar stills is required for separation of primary products.

There is no standard practice for preparing final plant products from the
crude-oil distillate, since special products may also be produced at differ-
ent plants to fit specific use and market requirements. The total crude oil
after it is separated from the pyroligneous acid by means of settling tanks
is distilled in a pine-tar still. The products of this distillation are usually
pitch, light and heavy pine tars, and a composite fraction of several light
solvent oils. Further chemical treatment and fractional distillation of the

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solvent oils yield turpentine, dipentene, and pine oil, together with small amounts of various hydrocarbons.

The water portion of the total distillate or pyroligneous acid contains methanol and acetic acid like the crude distillate from hardwood distillation, but in amounts too small to be recovered profitably.

The charcoal remaining from the primary distillation is cooled in the retorts or, in some cases, may be raked out hot into suitable metal containers and sealed airtight. When ovens with cars are used, the cars with the charcoal are run into closed sheet-iron coolers of about the same size and shape as the ovens, for periods of 24 to 48 hours.

Yields

Because few plants operate under the same conditions and various end products are recovered from the crude oil, it is difficult to indicate the average yields of products obtainable from the longleaf and slash pine raw material. Moreover, the raw material may also vary widely in resin content and provide further yield variations. The following tabulation shows roughly the ordinary yields obtained in destructive-distillation practice per ton of average southern pine stump and "lightwood."

<table>
<thead>
<tr>
<th>Product</th>
<th>Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total oils</td>
<td>35 to 50 gallons</td>
</tr>
<tr>
<td>Destructively distilled wood turpentine</td>
<td>4 to 6 gallons</td>
</tr>
<tr>
<td>Tar</td>
<td>20 to 30 gallons</td>
</tr>
<tr>
<td>Charcoal</td>
<td>350 to 400 pounds</td>
</tr>
</tbody>
</table>

Uses of Products

Turpentine. --Incompletely refined wood turpentine obtained from destructive-distillation processing is occasionally sold as a material inferior to gum turpentine, largely on account of its off odor. There appear to be numerous uses, however, in which this odor is no disadvantage. The refined destructively distilled wood turpentine is used for many of the same purposes as gum or steam-distilled turpentine. Chemically, the wood turpentines differ considerably from gum turpentine, although the properties are not markedly different. The main difference in composition is essentially the presence of a rather large beta-pinene fraction in the gum product and its virtual absence in the wood turpentine.

Some of the more common uses of turpentine are in the manufacture of paints, varnishes, enamels, and shoe, furniture, and floor waxes. It is also used...
in polishes, disinfectants, soaps, pharmaceuticals, sealing wax, inks, wood stains, insecticides, greases, crayons, and synthetic camphor.

Pine oil. --Destructively distilled pine oil is made to conform closely to the properties of the steam-distilled oil. Its uses and markets are similar to those shown under "Extraction Process."

Tar and tar oils. --The tar has many miscellaneous uses, the most important of which are in the manufacture of cordage, rubber, oakum, fish nets, tarpaulins, paper, soaps, insecticides, roofing cements, and paints.

The tar oils are good solvents for a variety of materials. They are used in some cases as a reagent in ore flotation and extensively in the manufacture of paints, stains, roofing compounds, disinfectants, insecticides, and soaps.

Pyroligneous acid. --This watery distillate, as such, has rather limited use in meat smoking, leather tanning, and as a weed killer.

Charcoal. --The chief uses for the charcoal are similar to those for kiln and recovery-plant hardwood charcoal. The more common outlets are in the recreational fuel, metallurgical, and chemical fields. Further specific uses include those in domestic, dining car, and incinerator heating, and in meat, fish, and tobacco curing. This charcoal is also used in the manufacture and treatment of steel, nickel, armor plate, aluminum, and copper, and in the preparation of activated carbon, black powder, carbon disulfide, nursery mulch, sodium cyanide, pharmaceuticals, and poultry and stock feeds. No accurate figures on plant cost are available at the present time. Based on somewhat earlier cost figures, it is estimated that the investment for a resinous-wood-distillation plant would now be $9,000 to $10,000 per cord per day capacity. Plant capacities in this industry are comparatively small, with a rated average capacity of about 60 cords of wood per day. In order to produce profitably at the present time, a minimum capacity for new construction is understood to be about 100 cords per day.

Extraction Process

The extraction process, sometimes referred to as the "solvent process," is the other principal method of obtaining resinous products from longleaf and slash pine wood. The products removed remain essentially unchanged. In the destructive-distillation process new products are formed by heat decomposition of the wood fiber and associated resinous material. The turpentine and pine oil from the latter kind of processing, therefore, become somewhat contaminated with other distillation products.
In general, the process of extraction— is a two-stage operation in which (1) the resinous material is removed by the solvent and (2) the retained solvent is recovered from the extracted chips by steaming. The highly resinous longleaf and slash pine stump wood is shredded into small chips and loaded into vertical, cylindrical extractors. Extraction is accomplished generally with a petroleum or coal-tar solvent. Multiple- rather than single-step extraction is carried out in a series of extractors in which each charge of new chips is extracted with several portions of solvent in succession. Each portion of solvent is used on several different charges of chips. The extraction is carried out counter-currently in a manner whereby fresh solvent is used for the final extraction of a charge and then is used successively on the charges that are to receive one, two, and three more extractions. After a charge of chips has received a final solvent washing, the solvent is drained off and the chips are pressure-steamed to recover residual solvent.

This extraction procedure requires a number of extractors or digestors. One group of these contains chips in various stages of extraction, and another contains extracted chips being pressure-steamed for solvent recovery. An extractor is also being charged with new chips or discharging spent chips. These operations usually progress from one extractor to another until a cycle is completed.

The solvent from the total terpene oil-rosin solution leaving the extractors is recovered by vacuum-distillation separation and is reused in further chip extraction. The terpene oils are separated by fractional distillation and recovered mainly as refined turpentine, dipentene, and pine oil. The nonvolatile residue from the extract is wood rosin of rather dark color. Upgrading of the rosin is carried out by clarification methods that generally may include bed-filtering or furfural treatment of rosin-solvent solution.

Processing for the recovery of primary products is fairly well standardized among the solvent plants. Yields of the products will vary, however, with individual plant operation and with the nature of the available raw material. Wood maintaining an average of about 22 percent resinous content by weight provides generally suitable material for satisfactory plant operation. The average yields of products obtained by extraction processing per ton of good-quality stump and lightwood material are shown in the following tabulation:


Turpentine ............... 6 gallons
Dipentene ................ 1-1/4 gallons
Pine oil ................... 4 gallons
Rosin ..................... 385 pounds

Steam-distilled Turpentine

Some uses for turpentine and pine oil obtained from destructive distillation processing have already been listed. Corresponding and reportedly more varied uses for similar products obtained in extraction processing provide the chief market outlets.

Dipentene

This material is a monocyclic terpene hydrocarbon present in commercially important amounts in the higher-boiling fractions of wood turpentines. While the commercial product is mainly dipentene and sold as such, it usually contains also varying amounts of other terpenes. Some of its uses are in the manufacture of paints and varnishes, and as a penetrating and softening solvent in rubber reclamation.

Steam-distilled Pine Oil

The principal uses for pine oil are in the manufacture of soaps, disinfectants, polishes, insecticides, deodorants, and protective coatings. It is also used as a solvent and wetting agent in the rubber and dye industries and as a flotation agent in metallurgical operations. Within recent years a synthetic pine oil has been produced from turpentine to meet the extensive demands for the natural product.

Wood Rosin

Wood rosin may be produced in color grades comparable to those of gum rosin, and its uses are in general similar to those of the gum product. As an industrial commodity, rosin has numerous and varied uses. Large amounts are consumed directly in the manufacture of soaps, varnishes, paints, paper sizing, greases, sealing wax, and roofing cement. As a raw material; it is used extensively in the production of metallic resinates and rosin oil, and gloss oil, and pitch derivatives obtained by dry-heat distillation methods. Additional compounds and derivatives in commercial amounts are obtained by polymerization and hydrogenation processes.
It is not possible to provide direct data on the cost of construction of extraction plants. Extension of such information as was available several years ago would show an estimated cost range at this time to be a minimum of $10,000 per ton per day capacity.

The majority of the older plants now operating have wood capacities within a range of 150 to 500 tons per day. Two of the three larger plants of the industry have a rated capacity of 700 tons per day, while the largest processes about 1,500 tons of wood per day. New plants operating on upwards of 500 tons of wood per day probably would be required to meet present-day operating conditions.

Current production of naval stores by the extraction and destructive-distillation processes consumes an estimated amount of 1,500,000 to 2,000,000 tons of stump wood a year. Of this amount, an estimated 1,500,000 tons of this old wood are processed in the former type of operation. Plants operating by these processes are now confined to locations in the states of Louisiana, Mississippi, Alabama, Florida, and Georgia. Future plant locations in other resinous wood regions would depend to a considerable extent upon equally good supplies of suitable raw material.