DISTILLATION OF HARDWOOD

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UNITED STATES DEPARTMENT OF AGRICULTURE
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By

L. F. Hawley,
Principal Chemist

Destructive Distillation of Hardwood[1]

Raw Material

The hardwood distillation industry is confined principally to northern Pennsylvania, southern New York, and Michigan, over 80 percent of all plants being in these states where, except for the Appalachian and southern hardwood belts, the hardwoods are most common and most easily accessible at the present time.

The species used occur in mixed stands and consist principally of beech, birch, and maple, with a small proportion of other hardwoods that naturally grow in these forests. Several new wood distillation plants have recently been constructed in the South where the southern hardwoods, oaks, red gum, ash, elm, and hickory have been used for destructive distillation. It also appears that for some species slabs give yields as large as or larger than bodywood. The importance of this fact in the utilization of mill waste is evident. Small wood, such as thin edgings or small limbs takes up so much room and is so costly to handle in comparison with equal weights of larger material that it is not commonly used while very small sized material, such as sawdust and shavings, has not been used satisfactorily. The small size of such material makes it so poor a conductor of heat that it is impossible to char it completely in the ordinary forms of apparatus and new types of apparatus for stirring the sawdust during distillation have had difficulty on account of the charcoal dust which clogs the condenser tubes. The finely divided charcoal is difficult to cool and the market for it is limited.

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[1] This circular has not been compiled with the intention of giving technical information or the results of original investigations, but to furnish a few facts about hardwood distillation in this country at the present time that it is impossible to give in answer to the many inquiries the Forest Service receives concerning this industry.

There is one process using small sized wood which is in commercial operation on a large scale. This process does not use sawdust or shavings, however, but fairly large chips, and the charge is not stirred. It is a continuous process, the warm, dry chips being delivered continuously to the top of the vertical stationary retort and the charcoal being removed continuously from the bottom. The retort is not heated since the decomposition of the chips furnishes enough heat to distill the fresh chips coming in.

Formerly only the best wood was used for distillation, wood suitable for lumber frequently finding its way to the retorts. This practice is becoming less frequent. Several plants are being run in connection with sawmills so that only the smaller trees not suitable for lumber are used for distillation, together with the limbs down to 4 or 5 inches in diameter and the larger slabs. Operation in connection with sawmills offers the additional advantage of a large supply of waste available as fuel to carry on the distillation and refining processes.

The wood is cut into cordwood lengths and in the best practice is allowed to season for about a year, either in the woods or at the plant. While it is not probable that the yield of products from green wood differs greatly, cord for cord, from the yield from air-seasoned wood, the reason for seasoning lies in the saving of fuel. The higher water content of green wood necessitates more fuel for carbonization and dilutes the distillate, causing further excessive use of fuel in the refining operations. Several plants have installed apparatus for the artificial drying of the wood using the waste heat from the ovens.

Distilling Apparatus

Hardwood is distilled in three forms of apparatus in this country: (a) In brick kilns; (b) in retorts; and (c) in ovens.

From the old types of charcoal kilns, where wood was charred under sod, all the valuable vapors escaped into the air and were wasted, while in the modern charcoal kiln the vapors are passed through condensers and saved. Kilns are mainly used where large quantities of charcoal are required, such as for blast furnaces making pig iron. They are made of brick, with a circular base and the entire structure resembles a beehive in form. These kilns are built to hold 50 to 90 cords each and are charged and discharged by hand. A complete distillation, including charging and discharging, takes from 17 to 25 days. The heat necessary to distill the wood in the kilns is supplied by the combustion of part of the charge within the apparatus. Because of this, and also because of the partial combustion of the vapors of distillation within the kilns, the yields of valuable products are lower by this method than by some others. The kiln type of apparatus is only economical when the chief product desired is charcoal, for which a fair price can be obtained and when cheap fuel for carbonization is not available.

The "retort" is a small cylindrical vessel holding about three-fourths of a cord. Retorts are set horizontally in brickwork, in pairs, each pair forming a "battery," and are heated externally by the hot gases from a fire box placed usually at one end of the apparatus. They are filled and discharged from a single door in front which can be tightly fastened. The top of the battery is often tiled and serves as a drying floor for acetate of lime. A "run" from charging to recharging takes 24 hours. The small round retort has been replaced in many plants by a larger rectangular retort or "oven."

The invention of the "oven" form of carbonizing vessel was a decided advance in wood distillation processes, especially from the standpoint of economical handling of the wood and charcoal. The ovens are rectangular in cross section and are made to hold from two to four cars, which are run in on tracks, each loaded with 2 to 2-1/2 cords of wood. They are fired in a manner similar to the round retorts by means of fire boxes at one or both ends, and the tops are also tiled to serve as a drying floor for the acetate. The vapors pass out through one and sometimes two openings at the side instead of at the end, as with the retorts. Except for the means of loading and unloading and the size, the ovens resemble the retorts in many respects. A distillation, from charging to discharging, takes from 24 to 30 hours. The standard distilling apparatus at present is the 10-cord oven.

The capacities of the plants vary greatly with the type. The minimum size for the most economical operation varies from 10 to 12 cords per day for retorts to 40 cords per day for ovens and over 100 cords per day for the kiln type.

Crude Products

There are four crude products obtained from each of the three forms of carbonizing vessels: (1) A noncondensable gas, which is carried off by suitable pipes at the end of the condenser; (2) an aqueous liquor known as "pyroligneous acid;" (3) wood tar which is condensed with the pyroligneous acid; and (4) charcoal, which remains in the distilling vessel.

With kilns the charcoal is allowed to cool from 3 to 5 days before being removed; with the round retorts the charcoal is shoveled into air-tight drums or cans immediately after the distillation is completed, and with the ovens the loaded cars are run out at the end of the distillation and placed in large coolers, which are similar in form to the ovens. There are usually two of these coolers for each oven, the charcoal going from the first cooler into the second at the end of the next distillation. For both retorts and ovens the charcoal is cooled for at least 48 hours in closed vessels, and in the best practice is then cooled 48 hours longer in the air before shipping. Wood charcoal has a strong tendency to spontaneously ignite, due to its remarkable

gas absorption properties, and for this reason must be carefully and thoroughly cooled.

The noncondensable gas still contains appreciable quantities of pyroligneous acid, and in a few plants is passed through scrubbers to recover the valuable products. In nearly all distillation plants, whether or not it is passed through scrubbers, the gas is either piped under the carbonizing vessel, as with retorts or ovens, and burned to assist in the distillation, or is burned under the boilers which furnish the steam for the refining operations.

The pyroligneous acid and tar run off together from the condensers into wooden vats where the tar is allowed to settle. The pyroligneous acid is reddish brown in color, has a strong, characteristic, burnt-wood odor, and contains from 3 to 4 percent of wood alcohol, 10 to 12 percent acetic acid, 5 to 6 percent of tarry matter, and the rest water.

The tar when in thin layers is dark brown in color and has a bad odor. Dissolved in the tar are some acetic acid and wood alcohol, which are recovered ordinarily by simple distillation with steam. In most cases, after separating out the acid and alcohol by distillation, the tar is used for fuel at the plant by burning under the boilers or retorts, being mixed with steam and sprayed with a suitable nozzle. Up to the present time the commercial uses for hardwood tar have been limited, and since the fuel value is comparatively high the most frequent uses is as fuel at the plant. A few plants have distilled the tar for the production of a hard pitch and various tar oils which have been used largely for flotation oils.

Refining

The refining method having the most general use at present is essentially as follows:

(1) Distilling the pyroligneous acid in a copper still to free it from the tarry matter dissolved in it; recovering the acid and alcohol from the settled tar, and adding it to the tar-free pyroligneous acid.

(2) Neutralizing the combined tar-free distillate with lime or milk of lime.

(3) Distilling the neutralized liquor in iron stills called the "lime lees" until all the alcohol is distilled off.

(4) Evaporating the residue from the "lime lees" first to a "mud" in shallow pans and finally to dryness on the tiled floor over the ovens or in specially constructed drying apparatus, this residue being "gray acetate of lime."

(5) Distilling the weak alcohol from (3) in column stills. In this step some plants obtain only a crude wood alcohol of 82 percent strength, which is sent to a refinery for further treatment, while others give the crude 82 percent additional distillations in fractionating columns and obtain 95 to 99 percent wood alcohol without color or unpleasant odor. The bad odor often noticed in wood alcohol is due to impurities present because of incomplete refining.

In this refining process the distillate from each step is completely condensed in water-cooled condensers. There are several opportunities for combining these steps to effect large economies in the steam and water necessary for these operations. Plants producing large quantities of distillate and those situated where cheap fuel is not available offer the best opportunity for such economies. The use of the more modern triple effect vacuum evaporators in place of simple stills for obtaining the tar-free pyroligneous acid, and for the concentration of acetate liquor, will affect a saving in steam. The separation of alcohol from the neutralized pyroligneous acid by means of column stills provides another method of steaming economy. A heat economy introduced lately is drying the acetate mud in chambers heated by waste heat from the retort stacks or blast furnace stoves. These more modern methods are now being used at several of the larger plants.

Yields

Kiln plants in which the combustion of part of the valuable products serves as heat to distill the rest yield on an average per cord of hardwood:

Charcoal	45 to 52 bushels.
Gray acetate of lime	80 to 120 pounds.
Wood alcohol (82 percent)	4 to 6 gallons.

Oven and retort plants secure about the following average yields per cord of wood:

Charcoal	45 to 52 bushels.
Gray acetate of lime	160 to 200 pounds.
Wood alcohol (82 percent)	8 to 11 gallons.

The lack of chemical supervision in a number of plants makes statements of yields a little confusing, since wood alcohol and acetate of lime are variable in quality and the number of gallons and pounds may, therefore, actually represent products of different composition, and also since the proportion of the various species used is quite different in different localities.

Uses of Products[2]

Charcoal has its largest use in blast furnaces for the production of pig iron. Large quantities are used as a domestic fuel in some localities, particularly in the eastern states. Charcoal is also used in the manufacture of gunpowder, by tin and copper smelters, as an insulating material, in poultry and stock feeds, and for various chemical uses, such as casehardening compounds and clarifiers in sugar refineries.

Refined wood alcohol is made and sold in various grades, each grade having a particular use or uses. Methanol is the name now commonly used instead of methyl or wood alcohol on account of confusion with ethyl alcohol. The former large use for wood alcohol as a solvent in the production of shellac varnishes, in hat making, etc., has diminished greatly in the last few years but its chemical uses as in the coal-tar dye industry, in manufacture of formaldehyde, photographic films and celluloid have increased. Certain fractions obtained in the refining process also have an important use as a denaturant for grain alcohol to produce "industrial" or "denatured" grain alcohol.

Acetate of lime is a gray, finely crystalline substance which is used principally in the manufacture of acetic acid and acetone, but is also used for the production of many commercial acetates, acetic ether, and other products. Acetic acid is used extensively in numerous chemical manufactures, such as dyeing of cotton cloth, production of pigments, cellulose acetates, etc. From acetone may be produced chloroform and iodoform. Acetone is also used in smokeless powder manufacture, as a solvent for acetylene in connection with acetylene welding processes, and in numerous chemical industries.

The use of acetone in the manufacture of smokeless powder (especially in England) and the use of acetic acid and acetone in the preparation of the "dope" or coating for airplane wing fabrics placed acetate of lime in the class of war munitions. The wood distillation industry, therefore, expanded greatly during this period.

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[2] The market prices of these products vary considerably from time to time. They are, however, regularly quoted articles in the various chemical trade journals.

Commercial Operation

The establishment of hardwood distillation plants for successful operation is subject to the usual requirements of any other highly technical industry such as (1) an adequate supply of raw material to operate the plant for a long period, (2) a market for all the products, and especially a nearby market for charcoal. The plants require expensive equipment, the present cost being in the vicinity of $5500 per cord per day capacity for a complete plant to manufacture acetate of lime and crude wood alcohol.

May 7, 1924.

For the last few years some of the products of wood distillation have met with keen competition because of the development of new sources of these products. Acetone, acetic acid, and methanol are now either being produced by fermentation or are being manufactured synthetically. With the appearance of these synthetic products the industry is being forced to depend more and more on the tars, oils, and charcoal for its profits.

November 15, 1932.