The Brown Coal Distillation Industry of Germany*—I

Methods, Apparatus and Products Resulting

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BROWN coal is a substance intermediate in its nature between peat and ordinary coal. Formed in comparatively recent geological times, Tertiary or Cretaceous, it has not yet developed into a true coal. Peat contains 80 or 90% of water from which it parts with great reluctance; it cannot be separated by pressure. Brown coal or lignite has 20% to 60% of moisture, common coal 2% or 3%.

There is little brown coal in Britain. At Bovey Tracey in Devonshire it has been worked for 200 years but is of only local importance. There are vast fields of it in the United States of America; great fields of unknown extent in Alberta, Saskatchewan, and Manitoba, Canada; and some 1,700 square miles of it in Victoria, Australia. Brown coal generally has no great covering on it, lies in beds of great thickness, and can often be worked opencast. The amount of water in it is a great drawback, and also its want of coherence when air-dried. In Manitoba, Saskatchewan, and Alberta it cannot be used for locomotive fuel on the railways on account of the dangerous sparks it throws out.

Brown coal is widely distributed through middle and north Germany and in Austria. Near Cologne it is 260 to 400 feet thick. Improved methods of mining and of preparing the products have given great importance to the industry in Germany and these methods are worthy of consideration in other parts of the world.

In the German Empire in 1905 the total coal output was 121 million tons and 30% of that was brown coal. The total output of coal in 1910 was 222 million tons. The coal production in the British Empire in 1910 was 312 million tons.

In Germany for the last half century brown coal has been subjected to low temperature distillation, and it may be of interest to give some description of the process for comparison with our Scottish shale oil industry. The products of the distillation are somewhat similar: permanent gas, spirit, lamp oil, gas oil, lubricating oil, and solid paraffin, but in Germany the oils require more chemicals in the refining, and after all the result is not so good. Dr. Scheithauer, Dr. Graefe, and others have described the German methods very fully.

This industry requires a very special brown coal and it is found only in two localities, namely, in the region of Halle on the Saale in Saxon Thuringia, and at Miesel near Darmstadt. The material is quite different in the two localities, and the retorting different. The principal center is at Halle, and works are situated between the towns of Weissenfels and Zeitz, and of Aschersleben and Eisleben. The ordinary brown coals of Germany do not contain sufficient bitumen to make retorting profitable. At first it was thought that any brown coal would do, and starting works on this supposition and without proper scientific expert advice, many ended in ruin. So a distinction arises at once between retort-coal and fuel-coal, but it often requires an expert, or chemical examination, to tell the one from the other.

THE SAXON INDUSTRY

The retort coal is found in layers situated among the fuel coal, there often being several layers located at varying depths. There is twice as much fuel coal as retort coal.

Freshly mined retort coal forms a plastic and sometimes greasy mass, of a brownish black color; when dry it is yellow to light brown. The fuel coal becomes black or light brown when dried. Retort coal melts on ignition and burns with a smoky flame; fuel coal does not melt. Retort coal has sp. gr. 0.9 to 1.1; fuel coal 1.2 to 1.4. The value for retorting depends on the proportion of bitumen present. The coal is of Tertiary age, Lower Oligocene, and contains 50% to

60% of water. In the Tertiary period the brown coal districts were occupied by extensive bogs with a luxuriant sub-tropical flora. Submerged in swamps the plants and trees were converted into peat as in our own peat-bogs. Sometimes large areas were exposed to atmospheric influences for a time by droughts or alteration of water level, so that the cellulose became partly or completely decomposed and only waxy, fatty, or resinous materials remained, and these covered up later by peat and sediments became through time the retort coal. Botanists state that the plants of that period supplies such wax or resin. The purest bitumen, called pyropissite, was at one time found throughout the coal then used, but seems to be now exhausted. It was yellow in color or in the purest form white. The bulk of the bitumen in the coal is soluble in benzene, toluene, ether, acetone, alcohols, carbon disulphide, and carbon tetrachloride; but the amount and character of the material got in this way depends on the solvent used.

Bitumen is extracted by a solvent, or by distillation with superheated steam under vacuum, and from it montan wax is manufactured. It is a pure white or yellowish wax with a very high melting point, the crude wax having m.pt. 80° or 90° C., refined 70° C. It is manufactured by six companies with a combined output of 400 to 500 tons per annum. The coal is generally dried and then extracted with benzol. There is a wet extraction process in which alcohol and benzene are used at once on the fresh coal, the alcohol

in. There were 10 or 12 retorts in a bench, and they were heated by fuel coal of equal volume to the retorted coal. Distillation was completed in 8 hours and the coke raked out. Riebeck, the James Young of Saxony, started building works at Webau in 1858, and built several others afterwards. In 1858 Rolle constructed a vertical retort, and its use became general, and with some minor improvements, is now the only type of retort used in Saxony. It gives a higher yield and a better quality of crude oil, works continuously and with less labor, and puts through five times as much per unit.

The retort is circular, of firebrick and heated by firebrick flues around the outside. Inside the retort a hollow cylinder is made of a series of flat iron rings. The rings are bevelled and arranged above each other louvre fashion, and the coal passes down the narrow space 3 to 4 inches wide between the rings and the brick wall. The products of distillation pass through the louvres into the hollow center of the retort and are carried off to the condensers by a pipe from near the top and another from near the bottom. The top pipe may either pass down outside the bench to join the pipe coming from the bottom, or it may pass downwards inside the retort to join the bottom pipe. A Koerting injector sucks the vapors from the retort through these pipes and passes them on to the condensers. At first all the firing was done by coal. Sometimes gas-firing is now used alone, but generally it is assisted by a fuel coal furnace at the side. Gas-

firing, besides being a great economy, has given 20 to 30% increase of distillate. The temperature in the heating flues around the retorts increases from the top downwards from about 400° C. (752° F.) to 600° C. (1,112° F.). The retort was at first 3 feet in diameter and 12 feet high, but is now 5 or 6 feet in diameter and 20 to 25 feet high.

The vapors issuing from the retort should have a temperature of 120° to 150° C. (250° to 300° F.).

In Scotland large amounts of steam are introduced into the shale retorts. No steam is introduced into the Saxon retorts. The raw coal has over 50% of moisture and this supplies the steam. The coal must be crushed or broken by hand, and lumps must not be larger than 1½ to 2½ inches in diameter. If too damp it is partially dried on the charging platforms of the retorts. The moisture content must not

A simplified diagram of a schedule for distillation

removing the water and the benzene dissolving the wax and the solution separating into two layers.

The water in the materials as they came from the pit was about 50%. The sulphur is seldom above 1%. The basic substances of the ash are oxide of iron, alumina, and lime, with smaller quantities of magnesia, potash, soda, and sometimes manganese and traces of strontia. These are combined with silica, sulphuric acid, sulphurous acid, hydrogen sulphide, carbon dioxide, traces of hydrochloric acid and occasionally phosphoric acid. The ash is practically of no use as a fertilizer, but it improves the physical state of the soil.

Mining.—The coal is first proved by boring. If the cover is not thicker than the coal it is worked opencast. If, however, the coal is thick, sometimes even up to two of cover to one of coal is worked opencast, and then the cover is removed by steam navy. With small cover it may be removed by hand. Opencast workings have haulage inclines operated by chain or rope. Most of the retort coal is worked by shafts. The depth is seldom more than 33 fathoms, although a few shafts are over 40 fathoms.

Retorting in Saxony.—At first, as in this country, the retorts used were of the old iron gas-retort type. Various forms were tried. At the last they were made of oval section, 8 to 10 feet long, about 30 inches wide, and 14½ inches deep. Thickness of metal 1 to 1½ inches. A layer of coal 4 inches thick was shoveled

be more than 60%, otherwise the coal tends to stick in the retort; nor less than 30% for then the oil vapors are decomposed by the heat, steam being necessary to protect them from decomposition. The top of the retort is closed by a heap of the coal ready to be passed down.

The residue of the distillation is a coke of some value for fuel purposes. It falls into a box below, which is shut off from the retort while the box is being emptied. This box is emptied 30 or 40 times in 20 hours. Automatic arrangements prevent the box being opened before it is shut off from the retort. The coke is dropped into hutches and drenched with water to quench it; or immersed in and hauled through pools of water, as used to be the practice in Scotland.

The retort rings and walls get encrusted in time; after cooling down the retort, the rings are lifted by a tackle and cleaned, and the retort walls are also cleaned. This requires to be done at intervals of 5 to 12 months according to circumstances. The coal ash is basic and the firebricks used must be basic also. If acid bricks are used the ash gets encrusted as a double silicate on the brick, and when cleaning this off, part of the brick breaks off also. The bricks are segments of a circle, keyed into one another, and when building they are ground into each other for tightness. To stand the heat the bricks are best made of pure aluminum silicate. There are various recipes for mortar, of clay, finely ground sand, kaolin, and ground firebrick, mixed with molasses.

* From a paper read before the Edinburgh Section of the Society of Chemical Industry, and published in the Journal of the Society.

The coke after quenching contains moisture 20%, ash 15 to 25%, and the remainder is carbon. It is sold in this state. Its calorific value is 6,000 to 7,000 calories. Up till the middle of the "seventies" the coke was tipped on spoil heaps. Now it is burned in special but very simple stoves for cooking, for heating workshops, in metallurgy as a reducing agent for zinc, and for other purposes.

The retorts are grouped in benches of 10 to 12 in a single row, with a safety wall dividing the benches. The tops are protected by walls with felt roofing. Retorts are estimated to last a dozen years. The through-put of retort coal seems to be about 3 tons per retort per 24 hours, and less than this if the coal is rich.

The condensing is all done by air-cooling. The vapors are passed through thin-walled malleable iron pipes beginning 36 inches diameter and decreasing further on to 31, 28, 20, 16 and 12 inches, finally finishing up in 1 inch pipes. They are all horizontal until near the end when they are vertical. For a retort 5 or 6 feet in diameter a condensing surface of 900 to over 1,000 square feet is allowed. Slow cooling is held to be necessary. Water cooling has been tried and rejected, although lately tried again. The suction from retort to condensers used to be by Koerting steam injectors; but now fans are much more in favor. There is no oil-scrubbing of the gas for spirit in Saxony, as there is at Messel and in Scotland.

In Saxony the water condensed along with the crude oil amounts to 40 to 50% of the retort coal, and more if Koerting injectors are used for propelling the vapors. The amount of ammonia in the water is only 0.03 to 0.07% and it does not pay to recover it, although efforts have been made to do so. The liquor is supplied to neighboring farmers gratis; it does not pay to convey to any distance. The great bulk of it has to be sent into the streams after some purification by aeration and filtration, or by filtering through spoil heaps. It contains besides ammonia, aldehydes, ketones, methyl alcohol and acetonitrile, with acetic, propionic, butyric and valeric acids and catechol, and is faintly alkaline in reaction.

The crude oil contains hydrocarbons of various aliphatic series, and also acid and basic substances, with some aromatic compounds. The cellulose, humic acids and bitumens of the coal produce their own products of decomposition. There is about 0.3% of nitrogen in the coal, and in the crude oil there is $\frac{1}{4}$ to $\frac{1}{2}$ % of nitrogenous bodies; 10% of the nitrogen of the coal is found in the crude oil, 12% in the condensed water, 66% in the coke, and 12% in the gas. These vary with the kind of coal and the method of distillation. The Rolle type of retort is adhered to on account of the good coke it produces.

In the Saxon field there are 12 works putting through together about one million tons of brown coal per annum, producing 60,000 tons of crude oil and yielding over 7,000 tons of solid paraffin. The crude oil yield now is about 18 gallons to the ton of coal. (Scottish production of crude oil is 273,500 tons per annum.)

THE MESSEL INDUSTRY

Retorting methods.—The industry began in 1885. The retort coal is quite different from that of Saxony, and is distilled differently. It seems unique in its nature, being a mixture of bituminous clay and coal, and having the organic matter in combination with the mineral matter. The bitumen cannot be extracted by solvents. It has a high percentage of moisture, like brown coal, and a high percentage of ash, like shale. It is more lumpy than the Saxon brown coal, which allows steam to pass through it in the retort. It lies under not more than 13 feet of cover, and is about 480 feet thick. It fills an ancient hemispherical depression, and is in layers like an onion. There are 240 acres of surface. It is worked down 80 feet or so and will last a long time. The fossils indicate a joint animal and vegetable origin. It has the consistency and cut of Dutch cheese, is blackish green in color, with conchoidal fracture when dry. Alternate frost and thaw make it separate into thin sheets like paper. The moisture is 45%, the ash 30%. The calorific value is low and would not pay cost of carriage for heating purposes. Dry distillation in the laboratory yields water 44%, crude oil 7.8%, coke-residue 36%, gas 6.2%. The condensed water contains volatile and fixed ammonium salts together with catechol and its homologues. The ammonia is recovered. In the fixed ammonium salts are quite a series of homologous fatty acids. The ash is rich in iron oxide.

The crude oil is distilled from the coal in the presence of steam, and the coke is dropped into a gas-producer below and the gas used to heat the retorts. There is no marketable coke. At one time a special stove was used to reduce the moisture from 44% to 6%, the dried material was distilled in the presence of

steam, and the coke dropped into the gas-producer. Thus coal was consumed in drying the retort coal, and also in raising steam from water for the coal distillation. Now the moisture of the coal is used as a steam for the retort. Vertical retorts are used of a type somewhat similar to that used in Scotland (see Ger. Pat. 200,602, May 23, 1906). The working is continuous. There is a hopper on top from which the coal is let into the retort by a valve as required. More than two-thirds of the retort is of iron; the lower part is of firebrick, and this is the gas-producer, and from it the coal ash is dropped into a hutch below. In the top part of the retort the coal is dried and the steam resulting is drawn from the top by an electrically driven fan; a part is returned into the retort further down, and part caught by another fan is passed into the bottom of the gas-producer where superheated steam acts on the carbon and nitrogen to produce water-gas and ammonia. The oil is distilled at the middle of the retort and the products of distillation led off from here to the condensers. The flues around the retorts are much more spacious than the flues of the Scotch retorts. The flues are divided by partitions into three compartments, and from one compartment to the one above there is communication by a comparatively small passage. The result is that there is a surging circulation of the products of combustion in each compartment, keeping each at the required temperature, and only a quantity passes off towards the chimney in proportion to the suction. The utilization of heat is good. The products of combustion pass to the chimney at 200° C. The vapors passing from the retort get partially cooled which causes the separation of ammoniacal liquor containing fixed ammonium salts, and some soluble organic compounds. The still hot vapors are passed on up a kind of Glover tower where all the ammonia is absorbed by dilute sulphuric acid. The ammonium sulphate solution so obtained is concentrated by the heat of the vapors, and after being centrifuged and dried the product is ready for the market. The oil vapors pass from the Glover tower and are cooled by water condensers. The water now condensed along with the oil is free from ammonia, and to reduce the quantity of effluent it is used in tubular condensers and towers and so is much evaporated. The gas is finally led up heavy-oil towers to catch the light spirit still left in it. From 70 to 80% of the permanent gas (some 10,000 cubic feet per ton) is used for heating the retorts; the rest goes to the power station. It may be used to raise steam, or after purification from hydrogen sulphide with iron oxide, it may go to gas engines. There is above 20% of carbon dioxide in the gas, and when this is absorbed the gas is well adapted for incandescence lighting. The ammoniacal liquor is passed down a tower-still with alkali, and the ammonia absorbed in sulphuric acid. From the spent water catechol and its homologues are recovered, and also fatty acids. The Germans extract every practicable substance from their by-products.

At one time the retort coal at Messel was graded, and yielded 4 to 14% of crude oil; now all goes together and averages about 7½% (=19 gallons per ton).

Would a retort of this type not be suitable for distilling a rich peat? Air-dried peat might serve quite well if moist peat is too wet. Peats no doubt vary in the proportion of bitumen they contain and they ought to be examined from this point of view. The brown coals of our Colonies should also be investigated.

Refining in Saxony. Distillation.—The crude oil is of a yellow-brown or dark brown color, sp.gr. 0.850—0.890, usually determined at 44° C. When richer coal was obtainable the sp.gr. was sometimes as low as 0.820 to 0.850 and seldom above 0.880. The present-day crude oil is generally 0.880 to 0.900. It sets between 20° and 30° C. The constituents boil at 80°—400° C., the greatest volume between 250° and 350° C.

The refining of the crude oil is in outline the same as in Scotland. The operations consist of several distillations with fractionation, treatments with sulphuric acid and with caustic soda between the distillations, and separation of the solid paraffin from the heavier oils by cooling and pressing. The first distillation is always partly a destructive distillation, and gives a deposit of carbon and production of permanent gas.

The distillations in Scotland are done at atmospheric pressure, and in the presence of steam more or less superheated. In Saxony they are carried on sometimes at ordinary pressure, sometimes under extra pressure, sometimes under partial vacuum; sometimes with steam, often without; but under pressure it is not for "cracking." The stills are vertical cylinders with hemispherical bottoms for a charge of 440 to 550 gallons, not more; this is only one-fourth of the size of the

smallest of our Scotch stills. The height of the still is 5 feet and the width at top 5½ feet; the cover is domed and is made in one piece with an exit-pipe about 20 inches long. There is a manhole in the center of the top, 20 in. × 24 in. The stills are bricked in in various ways so as to protect them from direct impact of the flame and send the products of combustion around the flues. The material is cast-iron, rarely wrought iron. Cast steel has been tried and rejected. Till comparatively recent years it was the invariable custom to work each still independently. The condensers are circular coils of piping in a vertical cylindrical water-tank. The coil is about 27 yards long, made of lead or iron, that is of gas-piping, or of cast iron semi-circular segments with flanges. The cooling surface per still is 85 to 95 sq. ft. Between the condenser and receiver there is a "U" bend and from between the condensers and this there rises a pipe to lead the permanent gas upwards into the atmosphere or onward to a gas-holder. There are two receivers, one at each side, each of 33 gallons capacity. When partial vacuum is used it is produced by Koerting steam injector or a vacuum pump.

[TO BE CONTINUED]

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