(b) *Lessing Process for the Separation of Oils and Pitch from Tar.*

For the purpose of recovering the highest possible yield of liquid oils from coal tar and to avoid thermal decomposition upon distillation, Lessing investigated the separation of tars of various character into oil and pitch by means of solvents. The fact that the asphaltic constituents of coal tar are precipitated by petroleum ether was made known as early as 1869 by Jacobsen.[213] He found, however, that the pitch is precipitated together

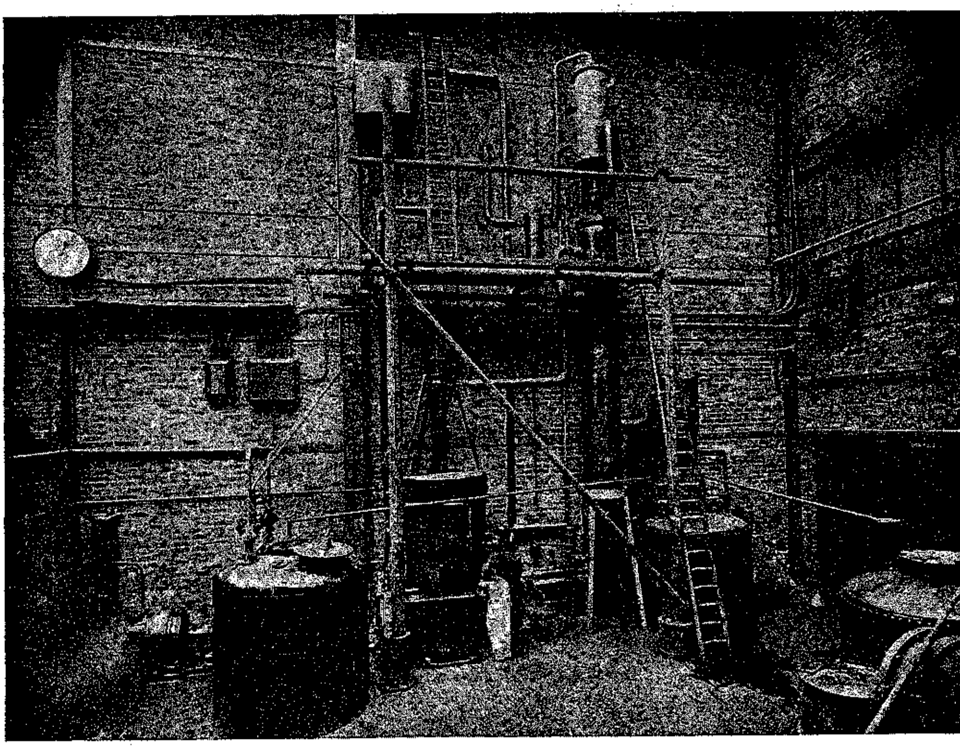

FIG. 75.

with part of the oil, forming a tough mass, which is difficult to handle. Other attempts to remove pitch from tar by means of solvents also failed for lack of sharpness of separation.

Lessing found [214] that a perfect separation could be obtained if coal tar is treated with a non-aromatic hydrocarbon fraction of suitable boiling range at a temperature above the melting point of the pitch to be made and below the initial boiling point of the solvent. He found that coal tars of any description, whether obtained at low or high temperature, in retorts, coke ovens, gas producers or blast furnaces, could be separated at a temperature in the neighbourhood of 100° into hard pitch and oil. The oil from gas works or coke-oven tar has a characteristic brilliant red colour, similar to that of bromine, and is of low viscosity. Primary tar and other low-temperature tars are of less vivid red or brown colour and, if containing paraffin wax, as in the case of producer or Scottish blast-furnace tars, solidify at room temperature.

One of the main practical difficulties in the substitution of coal tar for natural oil is due to the formation of solid deposits upon mixing or blending them with petroleum or petroleum products. This process provides a means of overcoming this difficulty by the complete removal from the oil of all those compounds which are liable to such precipitation.

Fig. 75 shows a small working unit erected at H.M. Fuel Research Station at Greenwich. The plant is designed on the continuous principle. The tar, from which benzol and naphtha have been removed by " topping," is fed in a steady stream into the extractor, where it is brought into intimate contact with the solvent. Pitch is precipitated instantaneously and is drawn off either continuously or intermittently from the bottom of the extractor. The oil solution passes into a fractionating column charged with Lessing Contact Rings.[215] The oil is completely freed from solvent so as to comply with the British Admiralty specification for flash point, and runs through the bottom outlet into the oil receiver. The solvent vapours are condensed in a reflux condenser and the condensate is returned to the extractor without being cooled below the temperature of condensation, so as to effect the greatest possible heat economy. The temperature differences between any points of the plant are thus reduced to a minimum.

It has been found in practice that the plant can be run with a negligible loss of solvent. In the case of low-temperature tars and any tars practically free from aromatic compounds in their lower fractions, a full yield of spirit boiling below the " cutting " point selected is obtained by passing the excess vapours through a final condenser.

The extraction of tar acids and bases is effected while the oil is still in solution, so that a more rapid and cleaner separation is obtained than in the direct treatment of tar oils.

Table LXXXVII shows the composition of different tars as obtained by Lessing's process.

The principal technical advantage of the process is that it affords a means of obtaining the whole of the oil actually present in the crude tar without loss.

The tar oil can be used directly as fuel oil, Diesel oil or wood preservative. It fulfils the condition of the Admiralty that home-produced oils must be capable of being added to the " common stock," mostly consisting of petroleum oils, without forming any deposits.

On distilling the oil obtained by this process the ordinary range of tar products is obtained. The degree of thermal decomposition can be controlled much easier than in the distillation of crude tar; the process can therefore be applied with advantage as a preliminary stage in tar distillation, whereby the throughput of a given still capacity is considerably increased, the fire risk is reduced and the burning out of still bottoms is avoided.

TABLE LXXXVII

Type of plant.	Coke oven.	Low-temperature.	Low-temperature.	Mond producer.	Tully producer.	Blast furnace.
Coal used.	Yorkshire.	Lancashire.	Yorkshire.	Staffs.	Scottish.	Scottish.
Neutral oil	54·01	55·98	66·0	36·89	52·07	46·44%
Tar acids	8·18	24·41	17·4	14·36	16·03	29·53 ,,
Tar bases	—	0·17	—	0·58	1·43	—
Total oil	62·19	80·56	83·4	51·83	69·53	75·97 ,,
Pitch	37·81	16·96	14·8	48·17	29·20	23·65 ,,
	100·00	97·52	98·2	100·00	98·73	99·62 ,,

In addition, the method of separating the tar without subjecting it to the risk of thermal decomposition is of importance for the study of its composition inasmuch as the tar can be subdivided into groups of compounds without secondary change. The difference from the customary examination of tar by distillation becomes manifest from Table LXXXVIII, upon comparing the figures given for free carbon in the pitch from distillation and that obtained on precipitation.

TABLE LXXXVIII

Type of plant.	Horizontal gas retorts.		Vertical gas retorts.	
Coal used.	Yorkshire.	Durham.	Yorkshire.	Durham.
Free carbon in:	%	%	%	%
Pitch from distillation	50·15	46·81	52·96	42·24
,, ,, Lessing process	33·61	34·08	18·38	24·67
Free carbon calculated for dehydrated tar:				
Pitch from distillation	30·31	33·17	18·29	17·31
,, ,, Lessing process	19·78	20·41	5·15	7·16
Found in dehydrated tar	19·27	18·78	6·46	6·07

(c) *Hydrogenation of Coal in the Absence of Oil*

During the last two or three years a considerable amount of work has been done on the hydrogenation of coal by the Bergius process, as distinct from the hydrogenation or Berginisation of oil, which until then claimed the almost undivided attention of those interested in Bergius' proposals. Of importance are the papers [216] by Waterman and Kortlandt, Waterman and Perquin, Kling, Bruylants, Erculisse, Shatwell and Graham. Bergius found it necessary to add an oil to the coal, partly in order to treat the coal in a dispersed state and partly in order to simplify the technical procedure. Bergius used for this purpose middle oil, or even coal tar itself. Waterman and his collaborators carried out the Bergius reaction in a medium of paraffin wax, after studying the behaviour of this substance in great detail; Shatwell and Graham employed phenols, which they found to remain unaltered, as was to be expected from Sabatier's researches.

It is not within the scope of this note to examine critically or even summarise the results of these researches; they are merely recorded for ready reference, as additions to the literature since the completion of the German edition.

The most recent publication on the subject is a paper by Franz Fischer and Frey [217]; the interest of their work lies in the fact that they treated various bituminous coals, brown coal and brown coal semi-coke without the addition of an oil of any kind. Bergius had made his preliminary laboratory experiments on coal alone,[218] and Fischer and Frey's results definitely confirmed the possibility of the liquefaction of coal under these conditions.

The coals used were Lohberg gas-flame coal, Osterfeld fat coal, English (Shipley) and Silesian (Lipine) sand (non-caking) coals, Rhenish brown coal and brown coal semi-coke. They found that hydrogenation begins at the same temperature as the formation of primary tar; they concluded therefore that the coal forms primarily gas, water and tar and absorbs hydrogen simultaneously or subsequently. This indicated the way to separate the two phenomena into carbonisation and subsequent hydrogenation of the semi-coke, and also made it desirable, by the use of non-caking coal yielding pulverulent semi-coke, to offer a larger surface to the action of the hydrogen.

In all cases hydrogenation took place, the yield of liquid products varying with the type of coal, the temperature and hydrogen concentration.

The yield of oils is larger than is obtainable by low-temperature carbonisation. The oils obtained are not entirely hydrocarbons, but contain up to 20 per cent. of phenols. The phenols are not those belonging to or derived from the primary tar, for they are also obtained in the hydrogenation of tar-free semi-coke. The following were the yields from brown-coal semi-coke in one of the experiments, showing that it was converted almost entirely into gaseous and oily products, leaving only the mineral matter behind:—

	Water	40 grams	= 8·5%	
	Oil distilled off	143 ,,	= 30·0 ,,	45% oily products.
Left in autoclave	Soluble in benzene	72 ,,	= 15·2 ,,	
	Solid combustible residue	29 ,,	= 6·1 ,,	16% solid residue.
	Ash	48 ,,	= 10·1 ,,	
	Gas and loss	143 ,,	= 30·1 ,,	
		475 ,,	100·0 ,,	

The combined products from three experiments with semi-coke gave a steam distillate which after extracting 13 per cent. of phenols showed the following boiling range :—

75°	110°	120°	130°	140°	150°	160°	170°	180°	190°	200°	210°	225°	230°	240°
Drop	2	4	8	16	24	32	40	44	49	58	68	78	83	80

Non-caking coals (*i.e.*, those containing a large proportion of durain) and semi-coke are more suitable than caking coals. In principle, one may expect corresponding results from the semi-coke of bituminous coals.

The hydrogen balance showing the distribution of the hydrogen used in each experiment, over the water, gas and oil formed gives a telling picture of the hydrogenation process. The tendency is for the hydrogen to form saturated gaseous products, but by judiciously increasing the pressure and reducing the temperature, it is possible to influence the reaction towards the formation of liquid products.

The significance of the sulphur compounds in coal, to which Kling had directed attention, was confirmed and the addition of metallic oxides such as CaO, MgO, Fe_2O_3, recommended by Bergius seems well justified in this respect.

Fischer and Frey conclude their summary by advising the study of the physical factors controlling the hydrogenation process in addition to its chemistry and expect from this a considerable advance in our knowledge of its mechanism.

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