XXIV. BLECHHAMMER.

Introduction:

Practically the entire Blechhammer staff left the plant at the end of January, one or two days before the Russians entered the area. Following a pre-arranged plan, they congregated at Naumberg about 20 miles from Leuna where an office was set up to arrange for their distribution to other German hydrogenation plants. By the end of February, Naumberg was so full of evacuees that the authorities insisted on the removal of the Blechhammer people and a large proportion of them went to Vorhum, near Peine (Brunswick). The movement of these people was surrounded with a good deal of secrecy due, it was disclosed, to their fear of being handed over to the Russians for restart of the Blechhammer factory. The managing director of Blechhammer, Dr.Josenhans, was interviewed in Naumberg and Dr.Kronig, the chief chemist, was interrogated at Vorhum.

700 ats. pressure hydrogenation of selected Upper Silesian coal provided the main source of liquid products at Blechhammer. Hydrogen production was based on gasification of medium temperature coke made by carbonisation of bituminous coal briquettes. The tar produced as a by-product to coke manufacture was obtained in two fractions by controlled condensation. The lighter fraction was sold as Navy fuel oil after topping off light petrol and the heavy tar was used as a pasting oil component in the coal hydrogenation process.

The original plan was to hydrogenate coal in the ordinary way, i.e. recycling to the liquid phase stage all products heavier than middle oil. Before the erection of the plant was completed, however, it was decided to increase the production of Navy fuel oil by operating the liquid phase hydrogenation stalls in such a way that the production of distillate heavy oil exceeded pasting oil requirements. Blechhammer data on this method of operation are of particular interest because they provide a basis for assessment of a three-stage coal hydrogenation process, i.e. 700 ats liquid phase hydrogenation of coal to give heavy oil, middle oil and petrol, liquid phase hydrogenation of distillate heavy oil to give middle oil and petrol, and vapour phase hydrogenation of middle oils to petrol products.

Another change which was made to the original Blechhammer plan was that as the shortage of aviation fuel became more acute, it was decided to make aviation fuel instead of motor petrol. D.H.D.,butane dehydrogenation and alkylation units were therefore erected.

Ten medium temperature carbonisation units were installed, each with a design capacity of 275 tons/day of coal. These units behaved very satisfactorily and a throughput of up to 325 tons/day was achieved. The coal was briquetted using sodium sulphite lye from paper manufacture as a binding agent and the finished briquettes contained 2% of moisture and 12% of ash. The coal had a carbon content on the ash-and-moisture-free basis of 83.5%. The tar yield on carbonisation was 9% by weight on the coal, the heavier tar fraction amounted to 35% of the total tar.

The carbonisation units were started up in October 1943. Before the hydrogenation plant was ready, the tar middle oil, topped to 120°C in order to reduce the flash point below 85°C, was sold as fuel oil and the heavy tar was disposed of to other companies for use as road tar, etc.

The hydrogenation plant actually installed at Blechhammer included four liquid phase stalls for 700 ats operation. Each stall had four converters similar to those used at Gelsenberg. The internal diameter was 1000 mm. and height 18 M. The internal diameter of the reaction basket was 850 mm. and the reaction volume provided by four converters was roughly 37 M^3.

Two vapour phase pre-saturation stalls were installed. Each had three converters and was designed for 300-350 ats operation. The plant was equipped with two vapour phase splitting stalls fitted with two converters each and designed to operate at 300-350 ats.

The coal used for hydrogenation was specially selected Upper Silesian coal with an ash content of 5% and a carbon content on the ash-and-moisture-free basis of 80-82%.

It was originally planned to add two further sections to the hydrogenation plant, each comprising two 700 ats liquid phase units, one pre-saturation and one vapour phase splitting stall. With these additions, the total output of Blechhammer was to have been 450,000 tons/year of motor spirit and 500,000 tons/year of fuel oil.

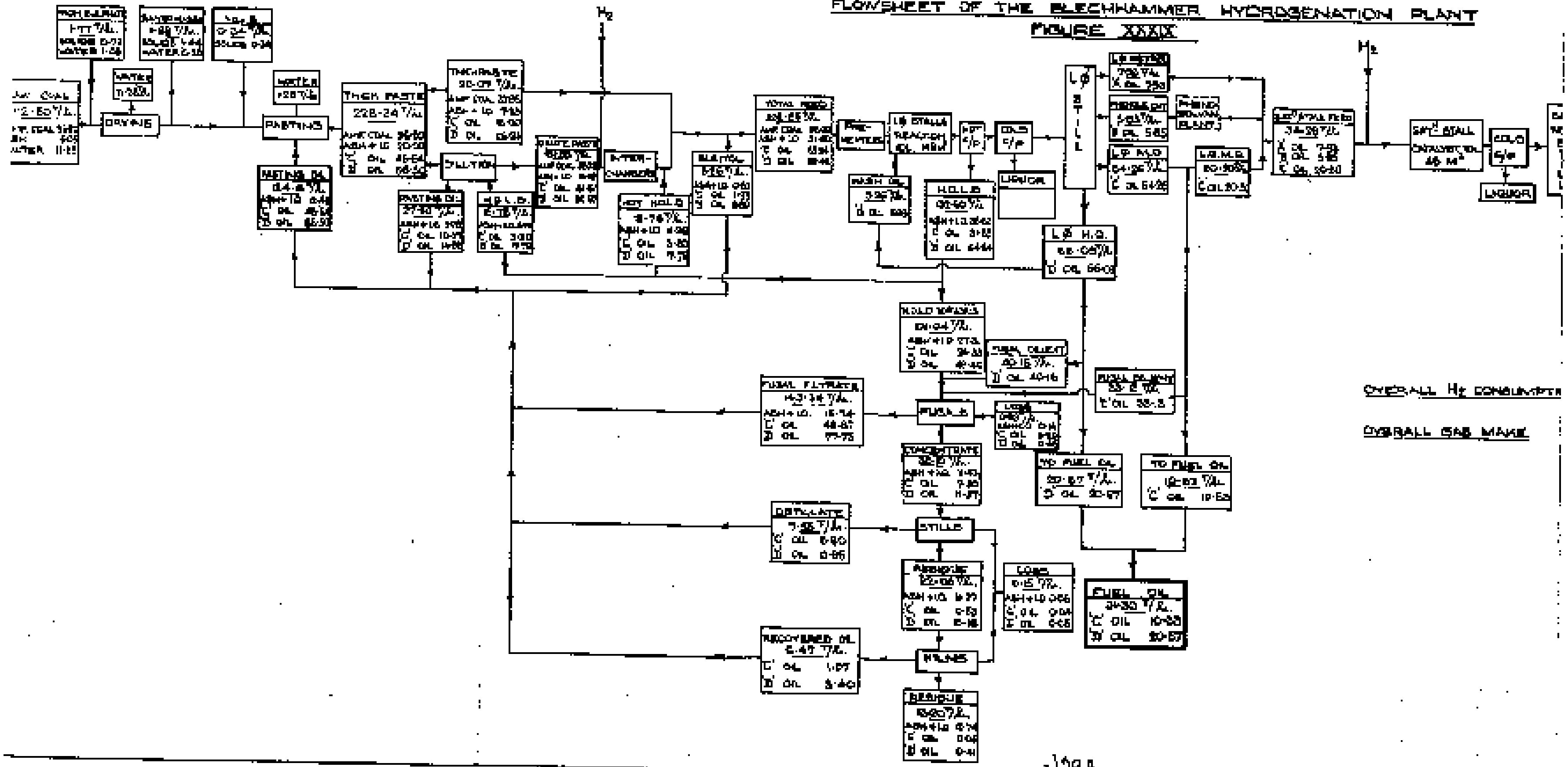
It was considered likely that the demand for fuel oil would decrease and accordingly, consideration had been given to the erection of one or more additional 700 ats stalls designed for the catalytic hydrogenation of distillate coal heavy oil to middle oil. The idea was to re-distil distillate coal heavy oil leaving a 10% heavy residue which would be returned as pasting oil to coal hydrogenation. The distillate was to be hydrogenated over 8376 catalyst at 700 ats and at a temperature of about 450°C.

The hydrogenation plant started up in April 1944 but by July, bomb damage to other hydrogenation plants had resulted in such a large surplus availability of brown coal tar that it was decided to import this material for hydrogenation at Blechhammer. During the three months during which the coal hydrogenation plant operated, it was stated that operation was extremely satisfactory.

PRODUCTION OF AVIATION PETROL & FUEL OIL FROM UPPER SILESIAN COAL

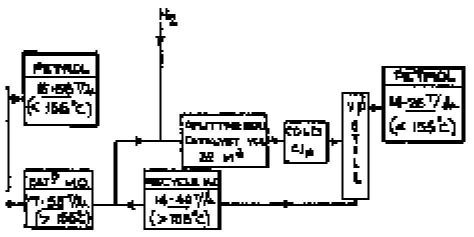
FLOWSHEET OF THE BLECHHAMMER HYDROGENATION PLANT

FIGURE XXXIX



OVERALL H_2 CONSUMPTI

OVERALL GAS MAKE



H 128,000 m^3/hr (87%)

CH_4	5·50
C_2	4·38
C_3	6·50
C_4	6·76
	23·18 T/hr

KEY TO OIL CODE

'A' OIL 0-160°C
'B' OIL 160-210°C
'C' OIL 210-325°C
'D' OIL > 325°C

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It was found that for the hydrogenation of brown coal tar it was necessary to increase the number of converters in each of the vapour phase stalls to four. It was found impossible to operate liquid phase hydrogenation of brown coal tar at 700 ats because the rate of hydrogenation of asphalts was so high that catalysts could not be kept in suspension in the reaction vessels. Pressure was accordingly reduced to 400 ats.

Details of Coal Hydrogenation to give Excess Heavy Oil.

The flowsheet shewn in Fig.XXXIX was supplied by Dr.Kronig. For simplicity, it refers to operation with coal as the only raw material. Actually, the amount of heavy tar which it was proposed to introduce into the coal paste at Blechhammer was comparatively small and would not greatly influence the flowsheet figures. All figures are tons/hour and refer to the operation of the total plant installed at Blechhammer, i.e. 4 liquid phase and 4 vapour phase stalls.

The coal was crushed and the first catalyst, iron sulphate, was added before drying. Dr.Kronig considered that better results would be obtained if the iron sulphate was added as a solution in order to obtain more even distribution. The mixture was then dried and it was stated that, during this process, neutralisation of alkaline constituents of the coal ash was effected. The dry coal, with 2% of moisture, was next mixed with approximately 1.5% of its weight of Bayermasse, a residue from aluminium preparation. This Bayermasse was previously dried to a moisture content of 15%. A third catalyst, 0.3% on the original coal of sodium sulphide, was next added, immediately prior to the pasting stage.

In order to produce heavy oil as one of the new-formed products of coal hydrogenation, it is necessary to reduce to a minimum the amount of recycle heavy oil in the coal paste. This necessitates the use of paste with a maximum coal content and the use of middle oil as a pasting oil component. Special difficulties are thereby introduced. For example, heat exchange between ingoing paste and exit products is not satisfactory with very thick pastes. Again, pastes made up of light oils and containing a low concentration of asphalts tend to settle when hot. These difficulties were overcome at Blechhammer in the following way: All the coal was first made up into a thick paste containing approximately 51% of total solids. About 40% of this thick paste was injected directly to the preheater, bypassing the paste interchanger. The remaining 60% was diluted down to approximately 43% total solids, i.e. maximum solid content for satisfactory paste interchange, and injected to the hydrogenation stall via the paste interchanger. In order to guard against settling of solids in the preheater, as much as possible of the diluent oil was supplied as high asphalt content heavy oil let down from the hot catchpot. Settling in the preheater was further guarded against by circulating a quantity of heavy oil let down from the catchpot to the preheater, without cooling or letting down to atmospheric pressure. This recycle was carried out with the same type of remote control hot recycle pump as used in the tar hydrogenation stalls at Leuna.

A small amount of pasting oil (5% on the coal treated) was continuously injected direct to the preheater. The object of this was, firstly, to prevent plugging of the preheater in the event of a breakdown of the paste injectors and secondly, to provide a lubricant film between the paste and the inner wall of the preheater tubes.

The total liquid injection to the stall was equivalent to a rate of 1.9 tons per M^3 of reaction volume per hour. This corresponds to the treatment of 0.65 tons of ash & moisture free coal per M^3 of reaction volume per hour. The figure supplied at Ludwigshafen for complete 700 ats hydrogenation of bituminous coal to middle oil plus petrol was 0.45 tons per M^3 of reaction volume per hour.

The paste temperature at the exit of the interchanger was not allowed to exceed 310°C. The exit temperature of the preheater was usually controlled to 415°C and was never allowed to exceed 425°C. The reaction temperature in the converters was kept as uniform as possible at about 485°C.

The circulating gas rate was equivalent to 3.5 M^3 per kg. of ash-and-moisture-free coal. The hydrogen partial pressure of the inlet of the converter system was 585 ats.

The whole of the products from the hydrogenation reactors passed to a hot catchpot which separated heavy liquid and solid products to the extent of 45-50% of the ingoing paste. Some 12% of this product was recycled hot to the preheaters as already described. The remaining 88% was cooled and let down to atmospheric pressure, after which a further 12% was returned direct to pasting as diluent for "thin" paste. The residual 76% was mixed with roughly 70% of its weight of diluent heavy oil obtained by topping off petrol and middle oil from the cold catchpot product and the mixture was fugalled. The fugalled filtrate was used as pasting oil and the residue was carbonised for recovery of oil.

The feed to the carbonisation ovens was given a preliminary flash distillation treatment to remove part of the oil. Recovered oil from the flash distillation and carbonisation ovens was used as a pasting oil constituent. The coke from the carbonisation ovens contained all the ash constituents of the original coal plus the catalyst plus the coke equivalent to the coal which had resisted liquefaction by the hydrogenation treatment. The latter amounted to roughly 4% on the a.m.f. coal fed.

The efficiency of recovery of oil in the two-stage sludge carbonisation plant was of the order of 75% and the oil lost at this stage amounted to 4.5 - 5% on the a.m.f. coal treated.

The vapours from the hot catchpot were cooled and the condensed liquid separated from recycle hydrogen in the cold catchpot. The liquid product was let down, separated from aqueous liquor and distilled to give 0-160°C, 160°-210°C, 210°-325°C and > 325°C fractions. Roughly half the 210°-325°C and two-thirds of the > 325°C cuts were recycled as pasting oil components. The yields of new-formed oils, expressed as weight % on the a.m.f. coal treated, were approximately -

0° - 160°C	8.2%
160° - 210°C	6.1%
210° - 325°C	32.4%
> 325°C	21.5%
Total	68.2%

210°-325°C middle oil and > 325°C distillate heavy oil were blended in the ratio of 1 : 2 to give fuel oil, the yield of which amounted to 32.5% by weight on the a.m.f. coal treated.

The remaining middle oil blended with the lighter boiling fractions was subjected to vapour phase hydrogenation in two stages. Pre-saturation was carried out over 8376 and 5058 catalyst, followed by splitting hydrogenation over 6434.

The pre-saturation was carried out in two stalls, each with three converters of 8 M^3 catalyst volume. The first two converters were packed with 8376 and the third with 5058. The pressure was 300 ats., mean reaction temperature 410°C and the maximum throughput 0.8 kg. per litre of catalyst per hour. The yield of saturated product was 97% by weight on the feed. This product was distilled to give a petrol fraction which was blended in a final petrol product and middle oil was fed to splitting hydrogenation. The cutting temperature depended on whether aviation or motor fuel was required. In the former case a 0 - 155°C fraction amounting to roughly 45% of the saturated product, was blended direct into the final petrol.

The residual middle oil was hydrogenated in two stalls, each with two converters of 8 M^3 catalyst volume, packed with 6434 catalyst. Pressure was 300 ats, average mean reaction temperature 390°C and maximum throughput - 1 kg per litre per hour. The conversion per pass was 50-55% when making aviation fuel and 65-70% when making motor petrol. Yields were 81-82% by weight of the feed of 0 - 155°C aviation fuel or, alternatively, 91-92% of 0 - 185°C motor gasoline. The former had a volatility of 55% at 100°C and a clear octane number of 74 which could be increased to 88 by addition of 0.09% by weight of TEL. The volatility of motor petrol was 35% at 100°C and the clear octane number was 70. The final blend of splitting stage motor petrol with the light distillate from the saturated product had a clear octane number of only 64. It was intended that, when making aviation fuel, the light distillate from the saturation product should be treated by the D.H.D. Process. A D.H.D. plant with a capacity of 26 tons/hour of feed had been installed. An alkylate plant for the production of 6.3 tons/hour of final product had also been built.

Phenol Extraction.

Phenols in the liquors from both coal carbonisation and coal hydrogenation were extracted with phenosolvan (isobutyl acetate). This was a development made jointly by Lurgi and the I.G. The two liquors were treated separately and there were three plants; two operating and one spare.

It was intended to increase the output of phenolic products by washing the 160-210°C petrol fraction with recycle hydrogenation plant liquor from the phenosolvan extraction plant. This washing was to be carried out counter-current at a temperature of 75°C.

The spent liquor from the phenosolvan plant contained rather less than 200 mgs. of tar acid per litre. There were serious restrictions on effluent disposal at Blechhammer and the spent liquor had to be treated in the following way:

It was first diluted with recycle cooling water to reduce tar acid content to 20 mgs/litre and to raise the temperature to 25°C. It was then treated with diplococcus-type bacteria which reduced the tar acid content to 2 mgs/litre. Effluent from this treatment was finally pumped over waste ashes from the power station, when phenols were oxidised down to less than 0.5 mgs/litre.

Treatment of Hydrocarbon Gases.

A Linde plant had been erected at Blechhammer in order to provide methane, ethane, propane and butanes from the mixed hydrocarbons produced in the hydrogenation process. These separated gases, apart from those required at Blechhammer for alkylate production, were to be sent to Heidebreck for use as chemical raw materials. A pipeline, 8 km. long, had been installed for this purpose.

Costs.

Very little data on costs were available. The delivered cost of coal was 10-11 marks/ton, skilled workers were paid 80 pfgs - 1 mark/hour and unskilled workers 55-70 pfgs/hour. The middle German brown coal tar which was supplied subsequent to July 1944 cost 80 marks/ton.

According to Dr. Kronig, the estimated costs of production of heavy oil and aviation petrol at Blechhammer were 250 marks/ton and 450 marks/ton respectively. 65% of these costs were capital charges which included interest on capital at the rate of 4½% and amortisation of the plant over a period of 11 years.