

## B HYDROGENATION PROCESSES.

### I. COAL MILL UNIT.

#### (a) Descriptions and Operation. (Fig. 10)

(1) The dry coal and the catalyst are hauled in. in closed cars. These cars are then connected to the conveying system, which brings the coal and catalyst to the bunkers, by means of section completely dust-proof and equipped with valves operated by compressed air. To eliminate large foreign matter, the coal and catalyst, upon entering, must pass through a grate with bars fifty (50) millimeters apart. The conveying system consists of horizontal and vertical

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Redler conveyors, two for the end and one for the catalyst. For vertical lifting of the catalyst it was necessary to replace the Redler by a bucket conveyor, to avoid caking and jamming. The long way that the coal and catalyst must travel required a great multiplicity of conveyors. Altogether there are six (6) Redlers operating in succession, to avoid jamming and breakage caused by faulty sequence to loading and unloading. Each conveyor is equipped with an automatic slip clutch connection to its motor.

(2) All the equipment of the grinding plant is protected by an atmosphere of carbon dioxide checked carefully by analysis. The coal is thereafter passed over a vibrating screen where it is divided into two (2) almost even portions. The coal that passes through the screen goes directly to the bunkers which feed the coal paste mill, while the coal that is retained above the screen is first broken up in two (2) roller breakers. To avoid spilling of the coal, there is installed at the bunker outlet a Redler conveyor which can circulate up to eighty (80) percent of its capacity. The only important part of the installation that follows is the rotary grinding mill, with feeding apparatus for coal, catalyst and diluent oil. The mill parts are: a screen conveyor inlet for coal and catalyst, a preliminary chamber and main chambers are separated by a perforated wall. The preliminary chamber contains as grinding elements steel balls of fifty (50) to eighty (80) millimeters diameter; the main chamber steel bars about twenty (20) millimeters diameter and twenty-five (25) millimeters long. The speed of the mill is twenty-one (21) RPM.

(3) The feeding mechanism consists of an automatic scale conveyor for the coal, and for the catalyst, Redler driven by a variable speed drive.

(4) The feeding and measuring of the diluent oil is done through a slide gate with a volumetric feeding control.

(5) The sulfur is weighed and added manually. In the mill coal, catalyst, sulfur, and diluent oil are mixed in

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a coal paste. This paste goes to a tank, from which it is taken and brought to high pressure by reciprocating pumps, so as to pass under pressure over the vibrating screen and into the seven hundred (700) atmosphere pumps of the finished coal reservoirs.

(b) Development Difficulties and Remedies.

(1) Coal Paste. For smooth operations of the sump phase it is imperative that the coal paste be perfect, and for that purpose any sudden change in its production process must be avoided.

(2) The paste prepared with Rhine lignite, has an abnormally high viscosity. This characteristic is due to a high swelling property of the coal; it is tied with the high water content of the coal and also with the viscosity of the heavy effluent oil from the coal stall having an initial boiling point of three hundred fifty (350) degrees centigrade. It is not advisable to exceed a concentration of solids in the paste, of forty-two (42) percent. Above that figure the paste loses its pumpability and leaks at the screens under high pressure. In order to obtain a well-prepared coal paste, it is desirable to watch:

- ((a)) The proper feeding of its component parts, especially the catalyst.
- ((b)) A low water content of coal and catalyst.
- ((c)) A constant composition of the diluent oil.
- ((d)) A low proportion of solids.

The temperature of the diluent oil must be kept as high as possible (one hundred twenty-five (125) to one hundred fifty (150) degrees centigrade) in order to assure the vaporizing of the water brought in by the coal and the catalyst, and to check the viscosity of the paste.

(3) The analysis of the coal paste in 1943 averaged:

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Sp. gravity at 110°	1.175
H <sub>2</sub> O	1.9 %
Solids	41.0 %
Coal	35.6 %
Ash in solids	13.3 %
Fe in solids	3.0 %
Sulfur (figured on solids)	1.6 %
Retained on a .3 mm. screen	10.6 %

(4) The coal was generally stripped from the mine in good, constant condition. Mechanical difficulties set in when the screens in the drying plant were operated. The water content was around 7.5 percent and ran up to twelve (12) percent. The average is 3.5 percent over the estimate. Although water is not desired in the coal, because it tends to disturb the viscosity of the paste and requires additional heat in the high pressure phase, a high water content was accepted to reduce fire and explosion hazards. The dry coal has a tendency to "run" with the result that a greater quantity of fines is suddenly discharged out of the bin. Effective counter measures consist in avoiding all possible friction of the coal in the Redler, which would tend to increase the proportion of fines, and to empty the bunkers once or twice every week to eliminate the accumulation of fine coal.

(5) The cellular Redler installed under the intermediate bunker is of value here. It restricts the "running" of coal to the chutes between Redler and Hilburg scale. Eventually these chutes were to be reduced in size.

(6) Dry coal dust is unpleasant and dangerous. A serious explosion took place in the drying plant of the mine, and in one grinding plant a number of fires and "puff-backs". The use of CO<sub>2</sub> scavenging of the inside of the apparatus proved satisfactory. After a few preliminary difficulties, there were no more accidents. The dust escaping to the outside caused further difficulties . . . . . (words missing in microfilm . . . . . led to the installation of a funnel shaped restriction in the chute from the scale to the mill, which was a great improvement. Attempts to wet the coal with diluent oil, as they had done at Leuna,

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to reduce the dust production, produced good results. The difficulty against building a spraying plant, however, was one of available space.

(7) The catalyst used was an extraction residue from Bauxite decomposition, called "Bayer Schlamm" dried and in large pieces up to thirty (30) millimeters supplied by Gerdemann in Quadrat, Ichendorf and by Chemical Fabrik Hösch, Düren. The  $Fe_2O_3$  content of the Gerdemann product was always above fifty (50) percent, in the Hösch product it was about five (5) percent lower. Besides, this Red Earth contains  $CaCO_3$  alkalies, aluminum oxide and silicate. The  $H_2O$  content of the catalyst was averaging six (6) to eight (8) percent and occasionally went up to twelve (12) percent. The high water content, found especially in the Gerdemann product were reduced by installation of a steam line for drying of the "Red Earth".

(8) This Earth tends to form crusts in the high bunker which should consequently be emptied and cleaned every three (3) months.

(9) Sulfur. Heretofore, weighing and addition of sulfur was done manually. The installation of an automatic measuring apparatus was stopped on account of war restrictions. To reduce the cost and also to make the measuring easier through handling of larger volumes (inasmuch as only one hundred (100) kilograms of pure sulfur per ton is needed) thoughts are given to the use of sulfur-containing residue of gas cleaning products. All particular incentive to do this comes from the fact that the Chemical Company at Wesseling could supply such products. In order to study the mechanical possibility of the use of these gas cleaning products, especially in the Redler conveyors, the one of the mill's units was operated for two (2) days with such products and no difficulties of operation were encountered.

(10) The diluent oil. This oil is a mixture of coal-stall effluent heavy oil, centrifuged oil, low temperature oil, and outside oil. The mixing is done in the heavy oil tanks by means of Eckard measuring recorders.

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I. COAL MILL UNIT. (b) (Cont'd).

An average composition for 1943 would be:

Coal stall effluent heavy oil	34.2 %
Centrifuged oil and low temperature oil	56.7 %
Outside oil	9.1 %

An analysis of the important data in 1943 is as follows:

Sp. gravity @ 110°C	1.062
Solids	4.0 %
Ash in solids	50.7 %
Asphalt in oil	8.8 %
Fraction boiling below 350°C	10.6 %

(11) In these data, the content of solids is of particular importance, because it controls the content of solids of the coal paste when the preparation of coal remains unchanged. The centrifuged oil is the carrier of the solids; it is therefore important that the making of this centrifuged oil and its addition be kept strictly uniform.

FINENESS OF THE COAL

Remaining over screen of	3.	2.	1.	.5	.3	.1	.06
On dry coal as received	10.	24.	46.	60.	71.		

HIGH SPEED SCREEN

52 % through	-	.3	9.	25.	43.5		
48 % above	21.	49.5	86.1	98.	100.		
Doreaker outlet	.8	1.8	25.2	45.7	64.8		
Mill inlet (paste)	-	-	-	3.5	13.	16.7	36.

(12) Loading of the mill. The maximum capacity of the mill corresponds to a production of twenty-five (25) tons/hours of paste at forty-one (41) percent dry coal. The bottleneck comes from the passage through the perforated plate between the preliminary and the main chamber. But the conveyor belt does not . . . . . (words missing here on micro-film) . . . . . either permit a capacity of twenty (20) ton/hours. To get a better fineness the weight of milling bodies

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I. COAL MILL UNIT. (b) (Cont'd.)

was enlarged; in the preliminary chamber, the balls were increased from nine (9) to twelve (12) tons, and in the main chamber from fifty-five (55) to sixty (60) tons. The wear of the balls was .65 kilograms per hour of operation and the wear of the bars .75 kilograms per hour, meaning about four (4) percent loss of balls and one (1) percent loss of such as the liners, made of harder material than the balls, is low and after three (3) years of operation does not come into the picture.

IMPORTANT FIGURES

Use factor of the mills	60.1 %
Number of mills operating	2,445.
Average load of all mills	54. tons/hour dry coal
Hours of operation	21,416. tons/hours/month

COAL PASTE

Fineness through the .3 m/m screen	10.6 %
Solids	41. %
Viscosity (500 cm <sup>3</sup> /100°C/6m/m orifice)	48 - 60 sec.
Ash in solids	13.4 %
Fine coal in the paste	32.4 %

DILUENT OIL

Coal stall effluent heavy oil	34.2 %
Centrifuged oil and low temp. distillation oil	56.7 %
Outside oil	9.1 %
Portions boiling below 340°C	10.5 %
Solid	4.05%
Asphalt	8.9 %
H <sub>2</sub> O in the dry coal	7.86%
Ash in the dry coal	5.83%
Red Earth (% of pulverized coal)	5.88%
Fe in Red Earth	35.5 %
H <sub>2</sub> O in Red Earth	9.3 %
Sulfur (% of pulverized coal)	1.19%

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I. COAL MILL UNIT. (b) (Cont'd.)

QUANTITIES

Dry coal	48.98 tons/hour
Red Earth	2.88 tons/hour
Sulfur	.58 tons/hour
Diluent Oil	79.67 tons/hour
Coal paste produced	130.57 tons/hour