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ENCLOSURE (B) 23

STUDIES ON THE EXTRACTION OF COAL

by

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SUMMARY

A study was made of the extraction of Fushun coal with basic oil obtained from shale oil. It was found that this basic oil was an effective solvent and 77% of the coal was dissolved by 4-stage extraction at atmospheric pressure. By extracting under hydrogen pressure, the amount of coal dissolved was increased. By distilling off basic oil from the final solution, an ashless coal can be precipitated which is suitable for carbon electrodes.

I. INTRODUCTION

In April, 1940, orders were given to study this project. After experiments on laboratory scale, a pilot plant of 10 liters/hr capacity was designed and constructed, but was not operated because the basic oil could not be obtained from MANCHUKUO in 1944. A flow sheet of the pilot plant is given in Plate I (B)23. No large-scale plants using this process have been built.

The key research personnel working on the project were Chem. Eng. Lt. M. MOCHIZUKI and Chem. Eng. Lt. Cmdr. M. KUMAMOTO.

II. DETAILED DESCRIPTIONA. Apparatus and Procedure

A rotating-type autoclave of 5 liters capacity was used for this project. The coal was ground and sized to pass a 60 mesh screen. Basic oil produced at FUSHUN was used as a solvent.

One part (by weight) of dried coal and three parts of oil were mixed to a paste in the autoclave.

After air in the autoclave was replaced by hydrogen, the pressure of hydrogen was raised to 100 kg/cm². The rotating autoclave was heated gradually and held at the desired reaction temperature for the required time. After cooling and releasing the pressure, the products were removed from the autoclave and filtered through a glass filter. The residue of filtration was washed, first with pyridine until the filtrate was colorless, and then with benzene until the odor of pyridine disappeared.

The washed residue was then dried and weighed. The percentage of extraction was calculated by the following formula:

$$\text{Percentage of Extraction} = \frac{(\text{weight of sample coal}) - (\text{weight of residue})}{\text{weight of sample coal}} \times 100$$

B. Experimental Results

The properties of Fushun coal and the basic oil obtained from shale oil are given in Tables I(B)23 and II(B)23. The basic oil was obtained by caustic neutralization of the sludge from acid treatment of shale oil. This oil was rich in quinoline and related compounds.

The effect of temperature, time, and pressure on the extraction were investigated and the results are given in Tables III(B)23, IV(B)23 and V(B)23.

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It was found that the extraction of coal is possible under atmospheric pressure at boiling temperature of the solvent. Consequently, further experiments were made to investigate extraction of coal at atmospheric pressure. The basic oil was fractionated into several cuts.

At first the extraction of coal was carried out with the lowest boiling solvent fraction, then the residue was extracted with the next higher boiling range fraction, and so on.

Experimental results are shown in Table VI(B)23 for 4-step extraction and in Table VII(B)23 for 3-step extraction. Both series of extractions were carried out under atmospheric pressure with no hydrogen present.

III. CONCLUSIONS

1. The influence of temperature, time and hydrogen pressure on the solvent extraction was investigated and it was found that the temperature effect was the most significant. Optimum conditions for extraction in presence of hydrogen were found to be 1-2 hours, 350-400°C and 50-100 kg/cm².

2. Extraction of the coal in the presence of hydrogen under pressure increased the percentage of coal dissolved. This is believed to be due to the peptization of the coal being facilitated as a result of slight hydrogenation. Up to 89% of the coal was dissolved under 100 atm. of hydrogen pressure, whereas at atmospheric pressure, using 4-stage extraction, a 77% extraction was obtained.

3. The solution of basic oil and dissolved coal was not considered as a source of boiler fuel due to scarcity and poisonous nature of the solvent. By distilling off the basic oil, an ashless coal can be obtained which is suitable for manufacture of carbon electrodes.

Table I(B)23
ANALYSIS OF FUSHUN COAL

Proximate Analysis (%)		Ultimate Analysis (%)	
Moist	4.16	C	73.7
Vol. H.	47.52	H	6.6
Fix. C.	41.67	S	0.7
Ash	6.65	N	1.7
		O	17.3

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Table II(B)23
DISTILLATION OF BASIC SHALE OIL

Boiling Range (°C)	Yield (%)	Remarks
From I.P. to 200	2	"Distillation above 250°C was made under vacuum of 10mm Hg" Distillation temperatures were corrected to atmospheric pressure.
From I.P. to 225	8	
From I.P. to 250	14	
From I.P. to 275	26	
From I.P. to 300	42	
From I.P. to 325	60	
From I.P. to 350	85	

Table III(B)23
EFFECT OF TEMPERATURE*

Reaction Temp. (°C)	Percentage Extract (%)	Final Pressure (kg/cm ²)
250	8.0	99.8
300	21.3	99.4
350	87.1	98.6
400	88.5	91.3
450	68.2	83.7

* Sample in each case contained 115gm coal, 310gm solvent, and was reacted for 2 hours under an initial pressure of 100 kg/cm².

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Table IV(B)23
EFFECT OF PRESSURE*

Initial Pressure with H ₂ (kg/cm ²)	Percentage of Extract (wt.%)
0	67.7
30	83.3
50	88.1
80	89.0
100	87.0

* Sample in each case contained 100gm coal, 300gm solvent and was reacted for 2 hours under a reaction temperature of 350°C.

Table V(B)23
EFFECT OF TIME*

Reaction Time (hr)	Percentage of Extract (wt.%)	Final Pressure (kg/cm ²)
½	65.6	99.4
1	84.5	99.0
2	85.4	98.3
4	78.0	90.8

* Samples in each case contained 105gm coal, 301gm solvent, and was reacted under a temperature of 350°C with initial pressure (H₂) of 100 kg/cm².

Table VI(B)23
4-STEP EXTRACTION*

Step	Matter Extracted (gm)	Solvent		Reaction Temp. (°C)	Percentage of Extract (wt.%)
		Volume (cc)	Boiling Range (°C)		
1	400	2000	I.D. to 250	230	7.5
2	235	1012	250 to 300	280	11.9
3	213	1065	300 to 350	330	23.0
4	119	342	350 to 400	380	35.0
				Total	77.4

Table VII(B)23
3-STEP EXTRACTION*

Step	Matter Extracted (gm)	Solvent		Reaction Temp. (°C)	Percentage of Extract (wt.%)
		Volume (cc)	Boiling Range (°C)		
1	400	2000	230 to 280	250	11.4
2	203	745	280 to 330	300	13.3
3	214	592	330 to 360	350	27.0
				Total	51.7

* In each case, reaction time was 1 hour.

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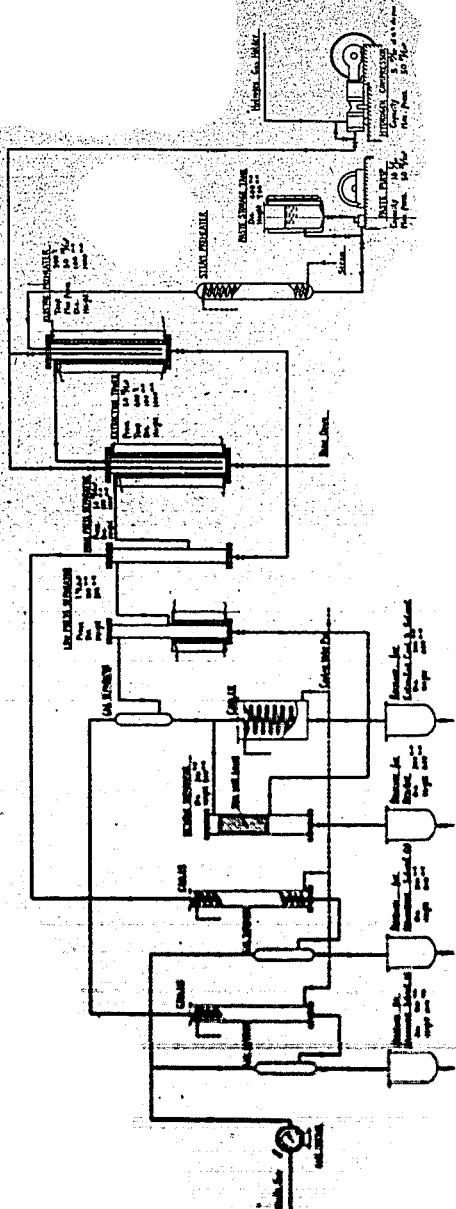


Figure 1(B) 23
FLOW SHEET OF PILOT PLANT FOR SOLVENT EXTRACTION OF COAL

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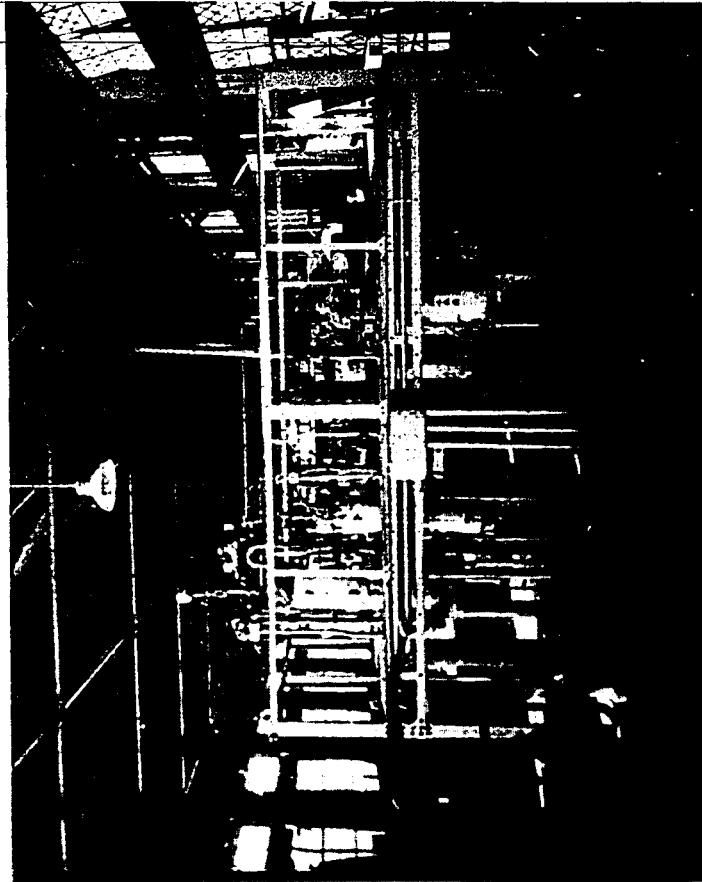


Figure 2 (b) 23
SOLVENT EXTRACTION PILOT PLANT OF BASIC OIL FROM SHALE