ENCLOSURE (B) 3

# EFFECT OF SIZE OF COAL ON COAL HYDROGENATION

Reference NavTechJap Document No. ND 26-0008.11 ATIS No. 4579

by

NAVAL ENGINEER
T. OGAWA

Research Period:
March 1938 - August 1939

Prepared for and Reviewed with Author by
the U. S. Naval Technical Mission to Japan

December 1945

ENCLOSURE (B)3

# LIST OF TABLES AND ILLUSTRATIONS

ENCLOSURE (B)3

## SUMMARY

To determine the influence exerted by the size of the coal on coal hydrogenation, Fushun coal was crushed to two size ranges, below 20 mesh and below 60 mesh, and tested on an autoclave scale. It was established that the size of the coal charge had no influence on the quantity or quality of the oil produced.

## I. DETAILED DESCRIPTION

### A. Test Apparatus and Procedures

The apparatus used in these experiments was a rotating type autoclave (refer to Figure 1(B)3). It was made from Mo-Ni-Cr steel and its volume was 2.4 lit.

Twenty steel balls (dia. 20mm and weight 27 gms) were put into the autoclave to assist in agitation. After introducing the oil sample and balls, air was displaced by hydrogen and the pressure was then raised to the required amount. The temperature and pressure of the autoclave were measured every five minutes. When the reaction period was over, the gas fire was turned off, but rotation was continued until the temperature dropped to 150°C. The autoclave was allowed to stand for one day, and the pressure and temperature were recorded before releasing the hydrogen. After the gas had been released, the products were removed, put into a flask, and called "Crude oil 'A'".

The residue sticking to the autoclave and balls was washed with about 100gms of benzene, and the solution was called "Crude oil 'B'".

Gas samples were analyzed by Hempel's method. Gas volumes were corrected to standard conditions (0°C, 1/atm).

Crude oil "A" was distilled in a column with an ice-cooled condenser. The oil was cut at 180°C, and the volume and weight of the oil and water measured. The solid residue was washed with benzene until the benzene was colorless. The residue was weighed and the benzene solution was added to the benzene soluble part of crude oil "B".

The water content of Crude oil "B" was determined by Deanstark's distillation method. The residue was separated by filtration and washed with benzene. The benzene soluble part of crude oil "B" was added to the benzene soluble part from crude oil "A", and the insoluble part from crude oil "B" was added to the insoluble part from crude oil "A".

The benzene and a small amount of light oil boiling below 180°C were removed by distillation and discarded. The product boiling below 180°C was blended with the oil obtained from crude oil "A".

The benzene insoluble residues obtained from crude oil "A" and "B" were dried at 80°C and reduced pressure to remove benzene, and then cooled and weighed. A portion of this residue was used for determination of benzene solubles by Soxhlet's extraction apparatus, and another portion was used for determination of ash content.

### B. Properties of Feed Stocks

Properties of the Fusnun coal used in these experiments are given below:

ENCLOSURE (B)3

Proximate Analysis (wt %)

| | |
|---|---|
| Water | 5.5 |
| Volatile matter | 41.1 |
| Fixed Carbon | 46.4 |
| Ash | 7.0 |
| Lichter's coefficient | 6.0 |

Elementary Analysis (wt %)

| | |
|---|---|
| Carbon | 79.3 |
| Hydrogen | 6.2 |
| Oxygen | 12.9 |
| Nitrogen | 1.3 |
| Sulphur | 0.3 |

Sieve Test (wt %)

| | below 20 mesh | below 60 mesh |
|---|---|---|
| 20-60 mesh | 74 | - |
| 60-120 mesh | 17 | 41 |
| 120-200 mesh | 4 | 39 |
| Below 200 mesh and less | 5 | 20 |

The paste oil was low-temperature tar oil made in a Davidson Retort operating on Shinbara coal, (30% of oil had been removed by topping).

Ferric oxide, commercial grade, was used as catalyst. Hydrogen of 99% purity was secured from the Hodogaya Chem. Co.

These items were mixed in the following proportions:

| | |
|---|---|
| Coal | 100gms |
| Tar | 50gms |
| Ferric oxide | 5gms |
| Hydrogen | 19gms(100 atm at 0°C) |

C. Experimental Results

Experimental results are summarized in Table I(B)3.

III. CONCLUSIONS

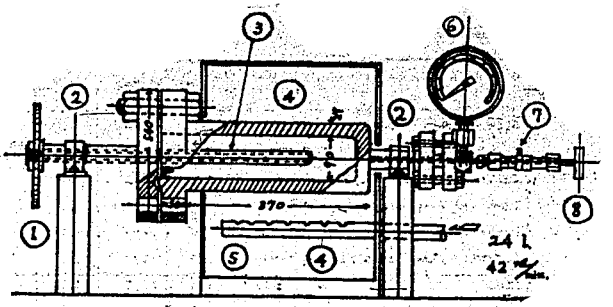
It was concluded that the size of coal charged, within the range investigated, had no influence on the yields or properties of products from coal hydrogenation on autoclave scale.

ENCLOSURE (B)3

## Table I(B)3
## EXPERIMENTAL RESULTS

| | | Run Number | | | | |
|---|---|---|---|---|---|---|
| | | 11 | 12 | 15 | 25 | 9 |
| Experimental Conditions | Size of Coal (mesh) | <60 | <20 | <20 | <20 | <60 |
| | Reaction Temperature (°C) | 500 | 500 | 480 | 450-470 | 460-480 |
| | Initial Pressure ($kg/cm^2$) | 100 | 100 | 101 | 98 | 97 |
| | Pressure Drop ($kg/cm^2$) | 27 | 26 | 11 | 18 | 10 |
| | Preheating Time (hr-min) | 2-10 | 1-50 | 2-55 | 2-10 | 1-55 |
| | Reaction Time (hr-min) | 2-50 | 3-0 | 1-0 | 1-0 | 1-0 |
| Yield of Products(gm) | Gas | 56.9 | 55.1 | 41.8 | 28.7 | 46.1 |
| | Water | 12.5 | 14.0 | 11.0 | 13.7 | 10.0 |
| | Oil | 62.4 | 65.2 | 75.1 | 78.7 | 79.5 |
| | Residue | 26.3 | 20.5 | 23.9 | 25.6 | 28.6 |
| | Total | 158.1 | 154.8 | 151.8 | 146.7 | 164.2 |
| Gas Analysis(gm) | $CO_2$ | 0.6 | 0.6 | 1.3 | 0.6 | 5.5 |
| | $C_nH_{2n}$ | 1.6 | 1.6 | - | - | 1.6 |
| | $O_2$ | - | - | - | - | - |
| | CO | 2.7 | 2.3 | 2.3 | 2.0 | 4.5 |
| | $H_2$ | 10.5 | 10.5 | 12.5 | 13.9 | 16.5 |
| | $C_nH_{2n+2}$ | 41.3 | 40.1 | 25.7 | 12.7 | 18.0 |
| | $N_2$ | - | - | - | - | - |
| | Total | 56.9 | 55.1 | 41.8 | 28.7 | 46.1 |
| | No. of C of Sat. Hydrocarbon | 1.8 | 1.5 | 1.5 | 1.0 | 1.4 |
| Yield of Crude Oil(gm) | Crude Oil "A" | 72.5 | 78.0 | 83.0 | 111.5 | 57.0 |
| | Crude Oil "B" | 28.7 | 21.7 | 27.0 | 6.5 | 61.1 |
| | Total | 101.2 | 99.7 | 110.0 | 118.0 | 118.1 |
| Yield of water(gm) | Crude Oil "A" | 12.5 | 14.0 | 11.0 | 11.0 | 10.0 |
| | Crude Oil "B" | - | - | - | 2.7 | - |
| | Total | 12.5 | 14.0 | 11.0 | 13.7 | 10.0 |
| Fractional Distillation (gm) | Below 180°C | 17.9 | 18.4 | 7.8 | 8.9 | 6.7 |
| | 180-230°C | 9.8 | 8.4 | 8.5 | 8.5 | 6.5 |
| | 230-280°C | 8.9 | 10.2 | 17.3 | 14.4 | 14.3 |
| | 280-360°C | 12.8 | 13.4 | 19.9 | 21.2 | 22.4 |
| | Pitch and Loss | 12.5 | 19.3 | 21.0 | 23.3 | 27.7 |
| | Total | 61.7 | 64.7 | 74.5 | 78.3 | 77.6 |
| Yield of Residue(gm) | Crude Oil "A" | | | | | |
| | Crude Oil "B" | | | | | |
| | Total | 27.0 | 21.0 | 24.1 | 24.0 | 30.3 |
| Analysis of Residue(gm) | Soluble in Benzene | 0.7 | 0.9 | 0.6 | 0.4 | 1.9 |
| | Organic Residue | 16.8 | 11.1 | 12.0 | 13.7 | 18.2 |
| | Ash | 9.5 | 9.4 | 11.9 | 11.9 | 10.4 |
| | Total | 27.0 | 21.0 | 24.5 | 24.0 | 30.5 |

ENCLOSURE (B)3




① Gear-Wheel
② Bearing
③ Protective Tube of Thermocouple
④ Furnace
⑤ Gas burner
⑥ Pressure gauge
⑦ Inserting and Evolving Pipe of Gas
⑧ Stopcock

Figure 1(B)3
ROTATING TYPE AUTOCLAVE

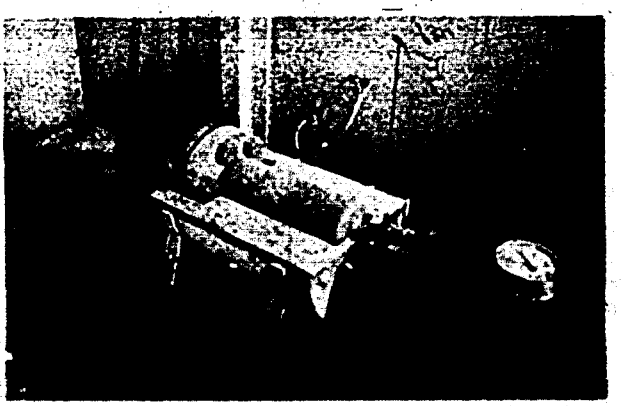


Figure 2(B)3
AUTOCLAVE USED IN THE COAL-HYDROGENATION EXPERIMENT