



Effect of catalysts on the reduction of sub-bituminous coal by the co-steam process
by Stephen Scott Bodner

A thesis submitted in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE
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Abstract:

The research presented in this thesis is a scoping study of the effects of various catalytic materials on the reduction of Colstrip sub-bituminous coal using the carbon monoxide-water shift reaction.

The purpose was to identify catalysts which would increase conversion to a benzene soluble product and possibly decrease sulfur and nitrogen content. The test runs were made in a 500 ml rocking autoclave. The operating time was five minutes when the autoclave reached 475°C.

Red mud, sodium carbonate, and sodium hydroxide were found to effectively increase conversion. Sulfur and nitrogen contents were not reduced.

Without catalysis of the shift reaction, typical hydrogenation catalysts were not found effective.

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COAL BY THE CO-STEAM PROCESS

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STEPHEN SCOTT BODNER

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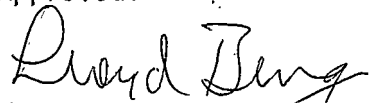
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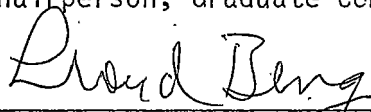
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ABSTRACT

The research presented in this thesis is a scoping study of the effects of various catalytic materials on the reduction of Colstrip sub-bituminous coal using the carbon monoxide-water shift reaction. The purpose was to identify catalysts which would increase conversion to a benzene soluble product and possibly decrease sulfur and nitrogen content.

The test runs were made in a 500 ml rocking autoclave. The operating time was five minutes when the autoclave reached 475°C.

Red mud, sodium carbonate, and sodium hydroxide were found to effectively increase conversion. Sulfur and nitrogen contents were not reduced.

Without catalysis of the shift reaction, typical hydrogenation catalysts were not found effective.

INTRODUCTION

One of the missions of the newly formed Energy Research and Development Administration (ERDA) is to develop all energy sources to meet the energy needs of present and future generations while making the nation self-sufficient in energy. One means of decreasing the dependence of the United States on foreign energy sources is to upgrade coal into a clean burning solid, liquid, or gaseous fuel. The United States has vast coal reserves with about fifty-five percent of the nation's reserves in the West (1).

Processes for upgrading coal to synthetic liquid products, originally developed in the 1930's, are being improved so as to supply a more easily transportable, nonpolluting, liquid fuel. The liquified coal products also have potential for use as synthetic crude oils and chemical feedstocks. Several processes for upgrading coal are now being developed. These include the Char-Oil Energy Development (COED) pyrolysis process, the Solvent-Refined Coal (SRC) hydrogenation process, the Synthoil catalytic hydrogenation process, and the H-Coal ebullient-bed catalytic hydrogenation process (2).

Coal is a high molecular-weight solid consisting of many unsaturated ring compounds with alkyl side chains (3). At 400°C, single carbon-carbon bonds or bonds of equivalent energy are ruptured. Progressive hydrogenation serves to stabilize the lower molecular weight fragments produced during thermal cracking. Competing with the progressive reactions are regressive reactions, polymerization of

the reactive fragments, making the product more difficult to dissolve. Reactor environment controls which reaction will predominate. Dissolution and hydrogen donor properties of the coal liquefaction solvent are also important (4).

Low-rank coals found in the sparsely populated western United States have a higher moisture content, lower sulfur content, and are more reactive when compared to coals of higher rank (5). These qualities make sub-bituminous and lignite coals especially well suited for liquefaction using the CO-Steam process. Water and carbon monoxide undergo the shift reaction to produce the hydrogen needed to stabilize thermally cracked coal molecules.

The Grand Forks Energy Research Center (GFERC) in North Dakota and the Chemical Engineering Department at Montana State University (MSU) are investigating the CO-Steam process for liquefaction of lignite and sub-bituminous coals. At MSU powdered coal slurried in phenanthrene solvent is reacted with carbon monoxide and water at approximately 5500 psig and 475°C. The liquefied product, although too hydrogen deficient to be used as a synthetic crude oil, could be used as a low ash, low sulfur utility boiler fuel. It could also be utilized as a feedstock for other catalytic hydrogenation processes being developed to increase the H:C ratio and remove sulfur and nitrogen (6).

BACKGROUND

Declining conversions have been noted in the continuous fixed bed reactor of the Synthoil process due to catalyst aging. This aging is a result of coke formation, deposition of metal sulfides, and accumulation of mineral components of coal. These contaminants accumulated both on the exterior surface and in the pore mouths of the catalyst resulting in a 70% reduction in catalyst surface area and pore volume (7). Eventually pore mouth blocking and bed plugging could occur. For these reasons, research at MSU was directed toward investigating unsupported solid, liquid, or gaseous catalytic materials which would be mixed with the coal slurry flowing through the reactor. Better contacting of liquid or gaseous catalysts with the reactive mass were expected to enhance the reaction. Economics would require the separation and recovery of the catalytic material from the liquified product in most cases. However some materials are cheap enough that recovery would be uneconomical.

Catalytic materials previously tested at MSU are shown in Table I (8). York (9) states that ferric oxide and sodium carbonate contained in coal ash were effective in promoting the carbon monoxide-water shift reaction. Higher sodium contents increased conversion (9).

TABLE I. Catalysts Previously Tested at MSU

<u>Catalyst</u>	<u>Conversion %</u>
Sodium carbonate	72
Sodium bicarbonate	60
Sodium formate	45
Stannous chloride	60
Calcium carbonate	50
Zinc chloride	40

Conversions of 65 to 75 percent have been obtained at the University of Utah using zinc chloride catalyst. At operating conditions the catalyst vaporizes and diffuses into the coal to catalyze desulfurization. A 99% catalyst recovery was obtained using a nitric acid wash (10). Nickel nitrate and stannous chloride are also reported effective (10).

Rock Springs, Wyoming sub-bituminous coal was reacted with hydrogen in the presence of red mud, stannous chloride, and ammonium molybdate catalysts by the Bureau of Mines at Pittsburgh. Ammonium molybdate was found to have a higher catalytic activity than stannous chloride (11, 12).

Halogen acids have been reported effective as having a catalytic effect on the thermal splitting of the coal into reactive fragments.

The fragments can be stabilized to form benzene soluble products by the addition of hydrogen or can polymerize to form benzene insoluble products (12, 13). The hydrogenation stabilization is catalyzed by tin (13).

Several metal chloride catalysts were tested by Sire and Alzheimer in a hydrogen plus hydrochloric acid atmosphere (14,15). Stannic chloride, nickel chloride, and stannous chloride were found to be the most active metal chlorides for coal hydrogenation using this technique.

RESEARCH OBJECTIVES

The objective of this research was to determine the catalytic effects of various materials on the CO-Steam process for the liquefaction of Montana sub-bituminous coal. The purpose of this research was to find a "good" catalyst which would reduce the operating costs of a full scale liquefaction plant.

A "good" catalyst is defined as a material enhancing conversion to a benzene soluble product with a possible side benefit of reducing sulfur and nitrogen content of the product. The catalyst should be either inexpensive so as to be throw-away, or easily recovered, regenerated, and recycled.

The catalytic effects of materials are unpredictable and therefore empirical data is needed for catalyst comparison.

EXPERIMENTAL PROCEDURE

COAL PREPARATION AND ANALYSIS

Large variations in Colstrip sub-bituminous coal composition were realized when small samples were taken from different lumps of a coal sample. In an attempt to minimize these variations, approximately ten pounds of coal were ground to minus 38 mesh in a porcelain ball mill. The batches of ground coal were then thoroughly mixed and stored in glass containers to prevent further water loss and oxidation. Water and ash analyses were performed on samples from each reactor charge. The ground coal size distribution is shown in Table II.

TABLE II. Coal Particle Size Distribution

Size: Tyler Standard

<u>mesh/inch</u> <u>Through</u>	<u>On</u>	<u>Wt. %</u>
-	50	30
50	100	37
100	200	18
on pan		15

Ash analyses were performed on two samples, each approximately one gram, from each reactor charge. The samples were heated in covered porcelain crucibles over Bunsen burners until all of the volatile matter was driven off. Then the lids were removed and the

flame was increased to burn off the remaining fixed carbon. The weight of the remaining ash divided by weight of the coal sample determined the weight fraction of the ash. The average of the two samples was used in the conversion calculations.

A water analysis was performed on a ten gram sample of coal from each reactor charge. A diagram of the apparatus is shown in Figure 1. Coal, toluene, and boiling chips were placed in a boiling flask attached to a 5 ml distilling receiver with a reflux condenser. The mixture was allowed to boil for 10-12 hours as condensed water and toluene collected in the graduated receiver. The volume of water collected times the density of water divided by the weight of the coal sample determined the weight fraction of the water.

York's thesis contained a typical analysis of Colstrip coal which is shown in Table III (16).

REACTOR CHARGING

First 30 ml of distilled water was poured into the reactor. Next 60 g of phenanthrene (Aldrich, 90%) and 30 g of coal were carefully added. The layers of phenanthrene and coal served to isolate the catalyst charge, which was then added, from the water. In some cases the catalyst would have reacted violently with the water had this precaution not been taken.

In most cases 0.5 g of catalyst were charged. Catalysts were

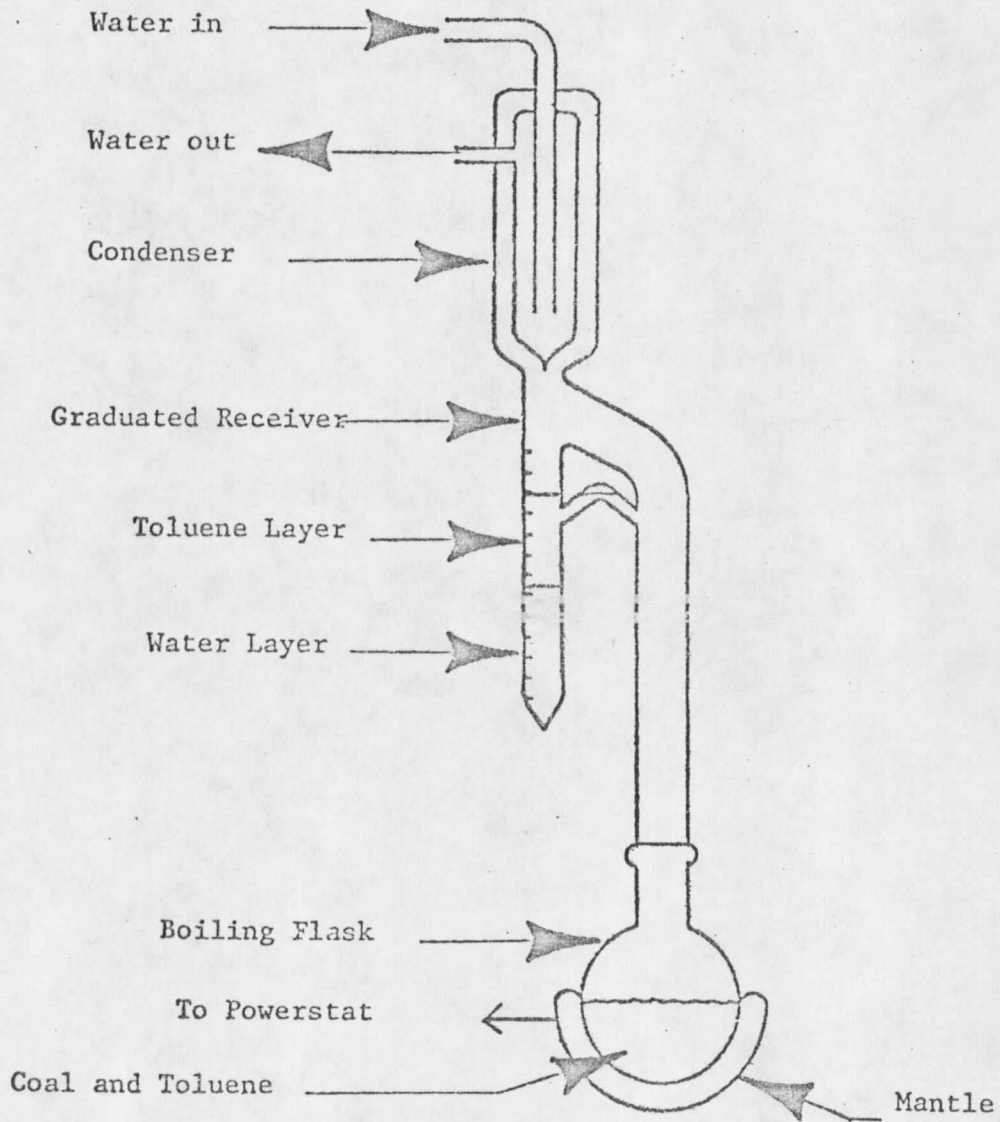


FIGURE 1. APPARATUS USED TO DETERMINE WATER CONTENT OF THE COAL

TABLE III. Analyses for Colstrip Sub-bituminous Coal

<u>Proximate Analysis</u>	
Moisture	23.9%
Volatile matters	30.9
Fixed carbon	37.6
Ash	7.6

<u>Ultimate Analysis</u>	
Moisture	23.9
Carbon	50.3
Hydrogen	3.4
Nitrogen	0.7
Sulfur	0.4
Oxygen	13.7
Ash	7.6

<u>Ash Analysis</u>	
SiO ₂	36.4
Al ₂ O ₃	17.6
Fe ₂ O ₃	4.6
TiO ₂	0.4
P ₂ O ₅	0.8
CaO	22.7
MgO	9.4
Na ₂ O	0.3
K ₂ O	0.5
SO ₃	14.9

compared on a weight basis rather than on a molecular or atomic basis since the "active" ion or ions in the catalyst molecule were unknown.

Next the head of the reactor was secured and the gauge block was installed. The reactor was then pressurized to 1500 psig \pm 50 psi with carbon monoxide.

REACTOR OPERATION

The reactor used for this study was a 500 ml Parr T316 stainless steel rocking autoclave. The Parr Series 4000 pressure reaction apparatus is shown in Figure 2. The reactor head was equipped with an 8000 psig rupture disk. The reactor will withstand working pressures up to 7000 psig at 400°C.

The reactor was placed into the heater-rocker apparatus and heated to 475°C \pm 4°. The temperature was recorded on a chart recorder using an iron-constantan thermocouple installed in the thermowell in the base of the reactor. As the reactor temperature neared 465°C, power input to the heaters was reduced to allow the temperature to line out at 475°C. After the reaction time of five minutes at 475°C, the heater-rocker apparatus was turned off and the reactor was removed.

After cooling, the reactor gases were bled off slowly under a vented hood. Then the reactor was disassembled and the unused water was poured off. The remaining sticky, solid material was removed and placed into a dried, weighted, cellulose, 43 x 123 extraction

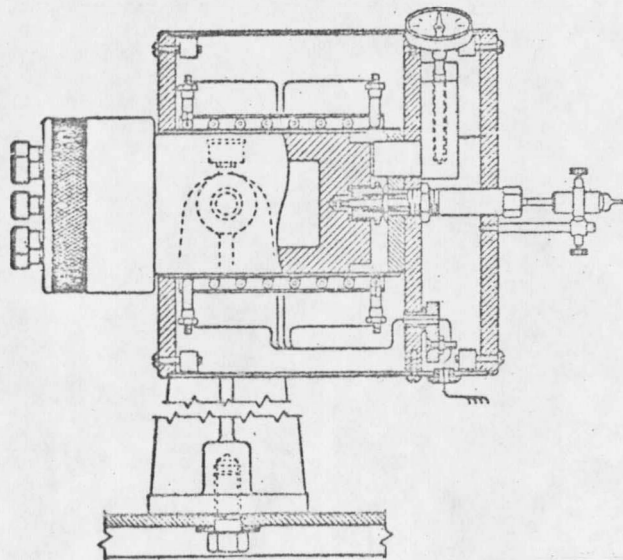
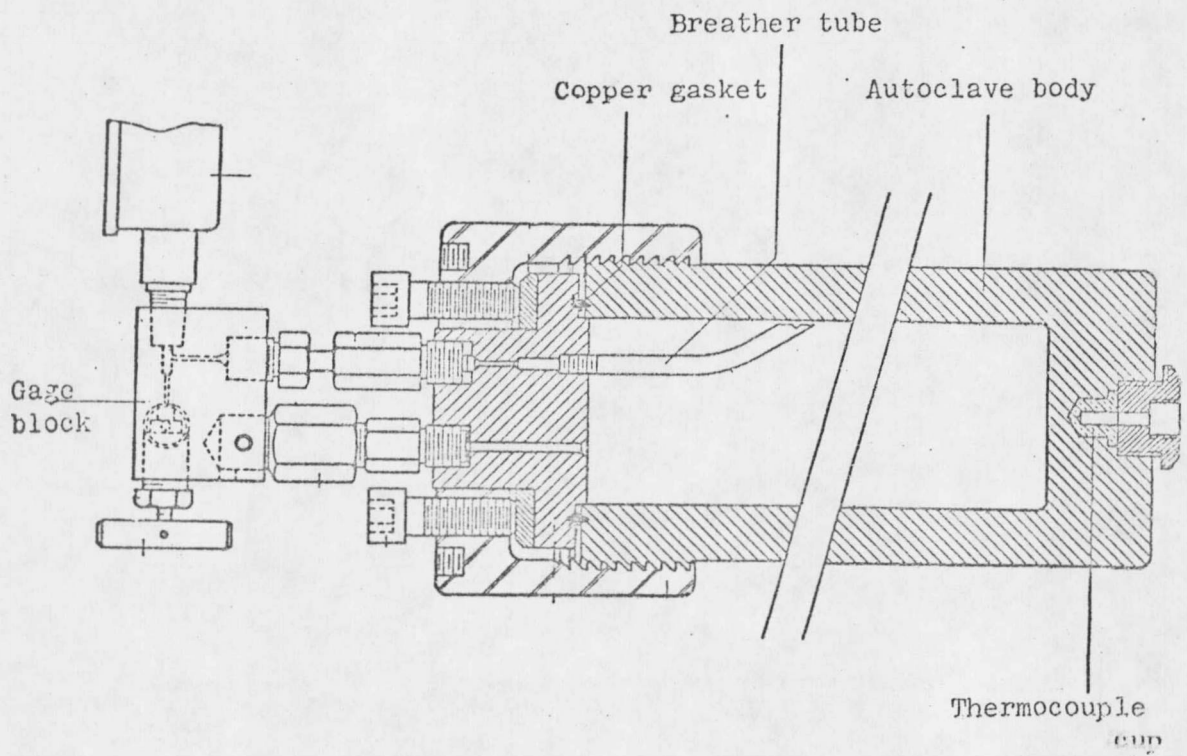


FIGURE 2. AUTOCLAVE AND HEATER DETAILS

thimble. Benzene was used to wash out the reactor and saved for the extraction step.

EXTRACTION

A Soxhlet extraction apparatus (Figure 3) was used to separate the benzene soluble material from the insoluble material. The extraction was run until the circulated benzene appeared to be clear, normally 24 hours, depending on the reflux rate.

The extract was then filtered through #2 Whatman filter paper to remove entrained insoluble material. Both the filter paper and extraction thimble were dried in a 110°C oven for at least 24 hours and weighed.

CONVERSION CALCULATION

Conversion to benzene soluble material was calculated using the formula:

$$x = \frac{\text{MAF(in)} - \text{MAF(out)}}{\text{MAF(in)}}$$

where

x = fractional conversion

MAF(in) = (wt. of coal charged) x (1-ash frac. - water frac.)

MAF(out) = (wt. of insoluble material - wt. ash - wt. catalyst)

In most cases the weight of the catalyst charged was neglected or assumed to be dissolved in the unused water that was poured off.

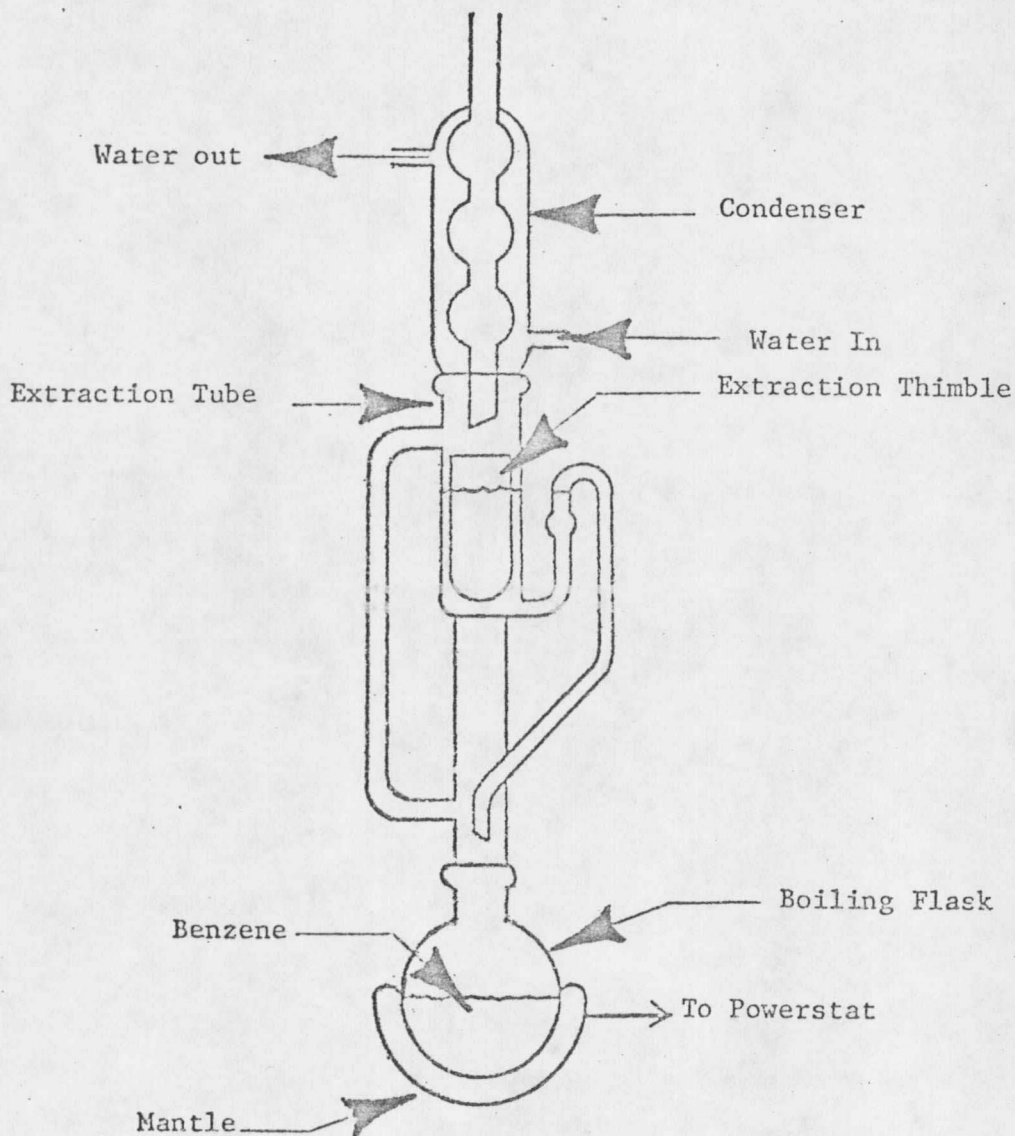


FIGURE 3. SOXHLET EXTRACTION APPARATUS USED TO DETERMINE COAL CONVERSION

When large quantities of catalyst were charged, the catalyst weights were included in the conversion calculation.

RESULTS AND DISCUSSION

Data for this scoping study were generated by 66 experimental runs during which 27 candidate catalysts were tested. Table V lists catalysts in order of decreasing conversion. Conversions of duplicate runs are averaged.

CATALYTIC EFFECTS ON CONVERSION

The combination of red mud and sodium carbonate gave the highest conversion of the catalysts tested. The composition of red mud is shown in Table IV. The 81 percent conversion obtained in this study (Appendix, Table VIII. No. 61) compares with the maximum conversion of 80.6 percent obtained by Sherick (17) for a 60-minute residence time run. The apparent maximum reached is probably due to the 11.5% fusinite and semifusinite content in the coal which is resistant to liquefaction, as well as equipment limitations.

TABLE IV. Red Mud Composition (18)

Fe_2O_3	40.8%
Al_2O_3	15.2
SiO_2	9.8
TiO_2	5.8
Na_2O	9.3
CaO	4.1
P_2O_5	1.3

TABLE V. Catalysts and Conversions

<u>Catalyst</u>	<u>Conversion %</u>
Red Mud (0.033:1)* Na ₂ CO ₃ (0.01:1)	81
Red Mud (0.133:1)	80
Na ₂ CO ₃ (0.01:1)	71
NaOH	71
Red Mud (0.0033:1)	70
NiCO ₃	64
SnCl ₄ · 5H ₂ O	61
AlCl ₃ · 6H ₂ O	61
Cr(C ₅ H ₇ O ₂) ₃	60
Ni(NO ₃) ₂ · 6H ₂ O	59
(NH ₄) ₆ Mo ₇ O ₂₄ · 4H ₂ O	59
NaCl	58
NH ₄ FSO ₃	58
AlBr ₃ · 6H ₂ O	58
HCl	57
H ₂ WO ₄	57
FeCl ₃ · 6H ₂ O	57
SbF ₃	57
PCl ₅	57
MoCl ₅	56

TABLE V. Catalyst and Conversions (Cont'd)

<u>Catalyst</u>	<u>Conversion %</u>
SnCl ₂	56
Mn(NO ₃) ₂	56
W	55
PCl ₃	55
POCl ₃	55
SbCl ₅	55
SbCl ₃	54
None	52
ZnCl ₂	49
TiCl ₄	6

* (Grams catalyst per gram coal)

Red mud, a waste product from the ore leaching step in the alumina process, when used in large quantities also appears to reach a maximum conversion of 80 percent. Data showing the effect of red mud concentration on conversion to a benzene soluble product is plotted in Figure 4. In general, as conversion increased, water consumption increased.

A conversion of 70 percent, using red mud (0.33%), is comparable to the 71 percent conversion obtained using sodium carbonate (1%) and the 71 percent conversion obtained using sodium hydroxide. Although sodium is contained in the most active catalysts, the form in which sodium is charged influences conversion. A comparison of Tables I and V shows sodium bicarbonate, sodium formate, and sodium chloride to be much less effective.

Nickel carbonate was relatively effective, however stannous chloride (SnCl_2) and zinc chloride were ineffective. Stannic chloride (SnCl_4) and nickel nitrate were more effective than stannous chloride and zinc chloride in this study using the CO-Steam process. Alzheimer's (19) catalytic-acid hydrogenation showed that stannous chloride was better than stannic chloride. Obviously different mechanisms are involved. Less water was consumed during the SnCl_2 and ZnCl_2 runs, indicating that less hydrogen from the shift reaction was available for stabilization of fragments. While some catalysts may be effective as hydrogenation catalysts, the shift reaction has

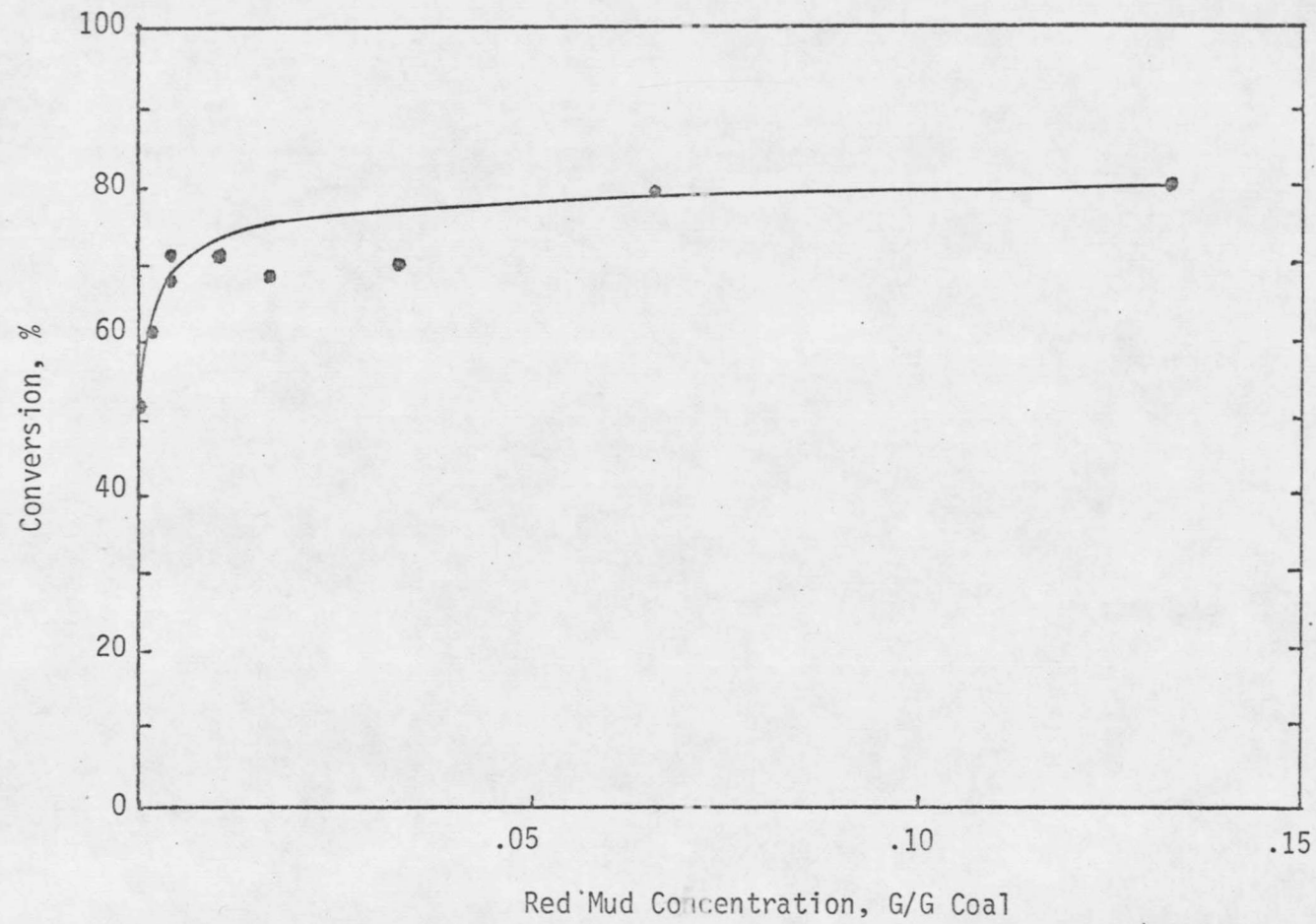


FIGURE 4. EFFECT OF RED MUD CONCENTRATION ON CONVERSION

to be catalyzed before hydrogenation can occur in this system.

QUALITATIVE PRODUCT PROPERTIES

Products from runs using $TiCl_4$, PCl_3 , and $POCl_3$ as catalysts were sticky masses together with a solid layer of charred material. Solid, crumbly product was found in all other runs. Apparently these catalysts were effective in hydrogenating part of the coal while coking of the remainder occurred.

GRINDING TECHNIQUE

York reported decreased conversion when sub-bituminous coal was ground in a ball mill rather than in a mortar and pestle (20). His solvent was ANP (50-50 mixture of alpha-naphthol and phenanthrene), residence time was one hour, and the temperature was $390^\circ C$. No catalyst was used.

Ball milled coal had a slightly increased conversion in this study where the solvent was phenanthrene, residence time was five minutes, and the temperature was $475^\circ C$. The coal for the first run was ground in a mortar and pestle and the conversion was 68 percent. This agrees with Sherick's (21) average conversion for five-minute runs using the mortar and pestle grinding technique. This research yielded an average conversion of 71 percent over three runs (Table VIII No.'s 02, 16, 45) using the ball mill techniques. One percent sodium

carbonate was used as in Sherick's work.

SULFUR AND NITROGEN ANALYSES

One of the objectives of this research was to produce a liquid product low in sulfur and nitrogen. Table VI shows the sulfur and nitrogen contents of sub-bituminous coal and the product obtained from the red mud-sodium carbonate run, No. 61. This was the only product chosen for analysis as conversion was the highest, and the availability of the analyses was limited.

TABLE VI. Sulfur and Nitrogen Analyses

	Sub-bituminous Coal Wt %	Coal MAF	Product Incl. Phenanthrene Wt %	Product W/O Phenathrene Wt %
Sulfur	0.4	0.6	0.23	1.0
Nitrogen	0.7	1.0	0.905	4.0

As shown in Table II, the coal contained both pyritic (in the ash) and organic sulfur. Apparently some of the pyritic sulfur ended up with the product thereby increasing the sulfur content. The reason for the high nitrogen content is unknown. These experimental results agree well with Huffman Laboratories' analysis of York's product shown in Table VII (22).

TABLE VII. York's Product Analysis

	<u>Product Incl. Solvent Wt %</u>
Ash	.02
Hydrogen	7.71
Carbon	84.10
Sulfur	.24
Nitrogen	1.86
Oxygen	6.07

CATALYST ECONOMICS

The use of red mud as a throw-away catalyst instead of sodium carbonate will decrease the operating cost of a coal liquefaction plant. Red mud could be added to coal in a 0.0033:1 ratio, compared to a 0.01:1 ratio for sodium carbonate, to give essentially the same conversion. The cost of red mud is approximately 1 dollar per ton (23) compared to 47 dollars per ton for sodium carbonate (24). The use of sodium carbonate rather than red mud would increase catalyst costs from 0.3 cents to 47 cents per ton of coal processed. Liquefaction plants of 1000 tons/day throughput are envisioned.

While enhanced conversions may be possible with combinations of shift reaction catalysts (eg. red mud or sodium carbonate) and hydrogenation catalysts (eg. metal chlorides), the cost of recovery

and regeneration of these materials may be prohibitive. Also the added problem of chloride corrosion in an already abrasive situation would complicate materials specifications for the liquefaction plant. It is felt by the author that if further hydrogenation, desulfurization, and denitrogenation are desired it would better be done in a subsequent catalytic process on the liquefied coal from this process.

CONCLUSIONS

1. Approximately 80 percent conversion of Colstrip sub-bituminous coal to a benzene soluble product was obtained using a combination of red mud and sodium carbonate catalysts. A similar conversion was obtained using a weight ratio of 0.13:1, red mud to coal.
2. Three times more sodium carbonate is needed than red mud to obtain a conversion of 70 percent.
3. The use of red mud instead of sodium carbonate as a throw-away type catalyst would decrease catalyst costs of a liquefaction process from 47 cents to 0.3 cents per ton of coal processed.
4. Organic sulfur and nitrogen were not reduced by the CO-Steam process.
5. Further hydrogenation, desulfurization, and denitrogenation would better be done in a subsequent catalytic hydrogenation process.
6. Ball milling of coal enhanced conversion as compared to use of a mortar and pestle.
7. The shift reaction should be catalyzed before hydrogenation can occur in this system.

RECOMMENDATIONS FOR FUTURE STUDY

1. Studies using a continuous reactor must be made to provide the data necessary for scale-up of the process. The effects of mixing and reactor size and the optimum operating conditions, using trends shown in previous batch studies as guidelines, must be established.
2. Production of a large quantity of uniform product from a continuous reactor is necessary for subsequent hydrogenation, desulfurization, and denitrogenation studies.
3. Quantities of extraction solvent and the residence times needed for a continuous extraction unit must be found.
4. A means of separating the process residue from the product must be demonstrated using either filters or centrifuges.
5. A use for the process residue such as carbon monoxide production should be established.
6. The ability to recover and recycle both the liquefaction solvent and the extraction solvent from the product must be demonstrated.
7. The feasibility of treating and recycling reactor off-gases should be determined.

APPENDIX

TABLE VIII. Experimental Run Data

Run No.	Catalyst Charge (g)	Coal Analyses		P _f (psig)	Water left(ml)	Conversion %
		% H ₂ O	% Ash			
01	0.3 Na ₂ CO ₃	22.3	7.3	5850	-	68
02	0.3 Na ₂ CO ₃	22.4	6.6	5950	-	72
03	None	22.9	8.0	5800	24	52
04	* HCl	23.0	7.3	5300	27	59
05	* HCl	22.8	7.3	5830	24	56
06	* HCl	22.8	7.3	5900	25	57
07	1.0 AlCl ₃ · 6H ₂ O	22.8	7.0	5550	25	59
08	1.0 AlCl ₃ · 6H ₂ O	22.8	7.5	5800	24	62
09	0.5 SnCl ₄ · 5H ₂ O	23.0	7.4	5900	21	60
10	1.0 SnCl ₄ · 5H ₂ O	23.0	7.7	6300	-	62
11	0.5 H ₂ WO ₄	23.1	7.1	5500	24	57
12	0.5 H ₂ WO ₄	23.0	7.7	5500	-	57

TABLE VIII. Experimental Run Data (Cont)

Run No.	Catalyst Charge (g)	Coal Analyses		P _f (psig)	Water left(ml)	Conversion %
		% H ₂ O	% Ash			
13	0.5 (NH ₄) ₆ Mo ₇ O ₂₄ · 4H ₂ O	22.9	7.3	5700	23	57
14	0.5 (NH ₄) ₆ Mo ₇ O ₂₄ · 4H ₂ O	22.3	7.7	5700	25	60
15	0.5 Na ₂ CO ₃	23.0	8.4	5600	10	77
16	0.3 Na ₂ CO ₃	22.9	7.4	5300	17	73
17	0.5 AlBr ₃ · 6H ₂ O	22.6	7.6	-	23	57
18	0.5 AlBr ₃ · 6H ₂ O	22.9	8.0	5700	-	60
19	0.5 FeCl ₃ · 6H ₂ O	23.0	7.2	5800	17	56
20	0.5 FeCl ₃ · 6H ₂ O	22.8	6.6	5400	20	57
21	0.5 SbCl ₃	22.8	8.3	4600	25	52
22	0.5 SbCl ₃	22.9	7.8	4500	24	55
23	0.5 NH ₄ FSO ₃	22.8	7.9	4900	26	58
24	0.5 NH ₄ FSO ₃	22.1	7.0	6080	22	57

TABLE VIII. Experimental Run Data (Cont)

Run No.	Catalyst Charge (g)	Coal Analyses		P _f (psig)	Water left(ml)	Conversion %
		%H ₂ O	% Ash			
25	0.5 NiCO ₃	22.4	7.6	4700	21	64
26	0.5 NiCO ₃	22.5	7.7	5100	25	65
27	0.5 SbF ₃	22.3	7.5	4800	25	58
28	0.5 SbF ₃	22.4	7.4	4750	25	56
29	0.5 MoCl ₅	22.3	7.4	4900	20	57
30	0.5 MoCl ₅	22.3	7.7	5000	22	55
31	* TiCl ₄	22.2	7.3	3900	11	12
32	* TiCl ₄	22.2	7.3	4300	10	0
33	0.5 W	21.5	7.7	4700	20	54
34	0.5 W	21.0	7.4	5400	13	56
35	* PCl ₃	19.6	7.8	5100	16	58
36	* PCl ₃	22.6	7.1	4900	16	52

TABLE VIII. Experimental Run Data (Cont)

Run No.	Catalyst Charge (g)	Coal Analyses		P_f (psig)	Water left(ml)	Conversion %
		%H ₂ O	% Ash			
37	* POCl ₃	22.6	7.3	5000	18	52
38	* POCl ₃	22.5	7.6	4900	15	59
39	0.5 PCl ₅	22.6	6.8	5000	25	58
40	0.5 PCl ₅	22.5	7.6	4800	-	57
41	* SbCl ₅	21.9	7.5	5100	26	56
42	* SbCl ₅	21.5	7.5	4300	24	54
43	0.5 SnCl ₂	19.0	6.4	5300	26	55
44	0.5 SnCl ₂	19.3	7.0	4900	23	58
45	0.3 Na ₂ CO ₃	22.0	7.4	5300	16	69
46	0.5 Red Mud	21.8	7.4	5450	14	68
47	1.0 Red Mud	21.6	6.7	5800	16	70
48	0.5 NaOH	21.5	7.1	5000	12	71

TABLE VIII. Experimental Run Data (Cont)

Run No.	Catalyst Charge (g)	Coal Analyses		P _f (psig)	Water left(ml)	Conversion %
		%H ₂ O	% Ash			
49	0.5 NaOH	21.2	7.2	4600	12	71
50	0.5 NaCl	21.5	7.5	4800	26	56
51	0.5 NaCl	21.4	6.7	4600	25	61
52	0.5 ZnCl ₂	21.3	6.8	5200	21	49
53	0.5 ZnCl ₂	21.4	7.3	5300	23	48
54	0.5 Cr(C ₅ H ₇ O ₂) ₃	21.4	7.2	4700	14	59
55	0.5 Cr(C ₅ H ₇ O ₂) ₃	21.5	7.5	4300	24	61
56	* Mn(NO ₃) ₂	21.3	7.2	4600	19	55
57	* Mn(NO ₃) ₂	21.2	7.4	4700	24	57
58	0.5 Ni(NO ₃) ₂ · 6H ₂ O	21.6	7.9	4700	20	60
59	0.5 Ni(NO ₃) ₂ · 6H ₂ O	21.3	7.4	5700	24	58
60	2.0 Red Mud	21.3	7.2	4500	10	79

TABLE VIII. Experimental Run Data (Cont)

Run No.	Catalyst Charge (g)	Coal Analyses		P _f (psig)	Water left(ml)	Conversion %
		%H ₂ O	% Ash			
61	1.0 Red Mud 0.3 Na ₂ CO ₃	21.5	7.0	4400	10	81
62	0.3 Red Mud	21.5	7.4	5400	22	71
63	0.1 Red Mud	21.4	7.0	5400	17	71
64	0.05 Red Mud	21.3	7.3	5300	26	61
65	0.1 Red Mud	21.3	7.0	5300	20	68
66	4.0 Red Mud	21.3	7.2	5100	14	80

* Approximately 1.0 ml of liquid catalyst charged

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