# TASK III

## Project III.2

# BETTER HYDROGEN MASS TRANSFER
# IN COAL LIQUEFACTION

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## I. Introduction

During the initial stages of coal liquefaction, it is hypothesized that free radical reactions occur rapidly and that reaction rates increase almost exponentially. These fast reactions involve the thermal disruption of the coal structure to produce free radical fragments. If the free radicals are not stabilized by either molecular hydrogen or by hydrogen donor solvents, retrogressive reactions and catalyst deactivation take place. Hydrogen starvation in the initial stages of coal liquefaction has a permanent negative impact on product yields.

In order to address these problems, a new coal liquefaction reactor system was constructed in our laboratory prior to this work year. This jet loop reactor system incorporates a novel gas-driven pumping system for use under severe operating conditions. The development of the pumping system was essential to avoid the prohibitive cost of a suitable mechanical pump.

During the year, the complete system became fully operational for coal liquefaction studies. Initial experiments, detailed later in this report, showed that the conversion of coal and selectivity for oils was very high. The reactor system incorporates reaction rate control by modifying two control parameters: (1) reaction zone temperature, and (2) the frequency of cyclic switching. The two parameters can be adjusted to regulate the gas-liquid mass transfer requirements per reaction cycle.

The pumping system consists of two modified autoclave reactors used as stirred tanks, air-operated valves, gas holding tanks, a gas booster, an air compressor and a programmable logic controller (PLC). Differential pressure between the two stirred tanks is the driving force for slurry flow through the reactor system which is placed
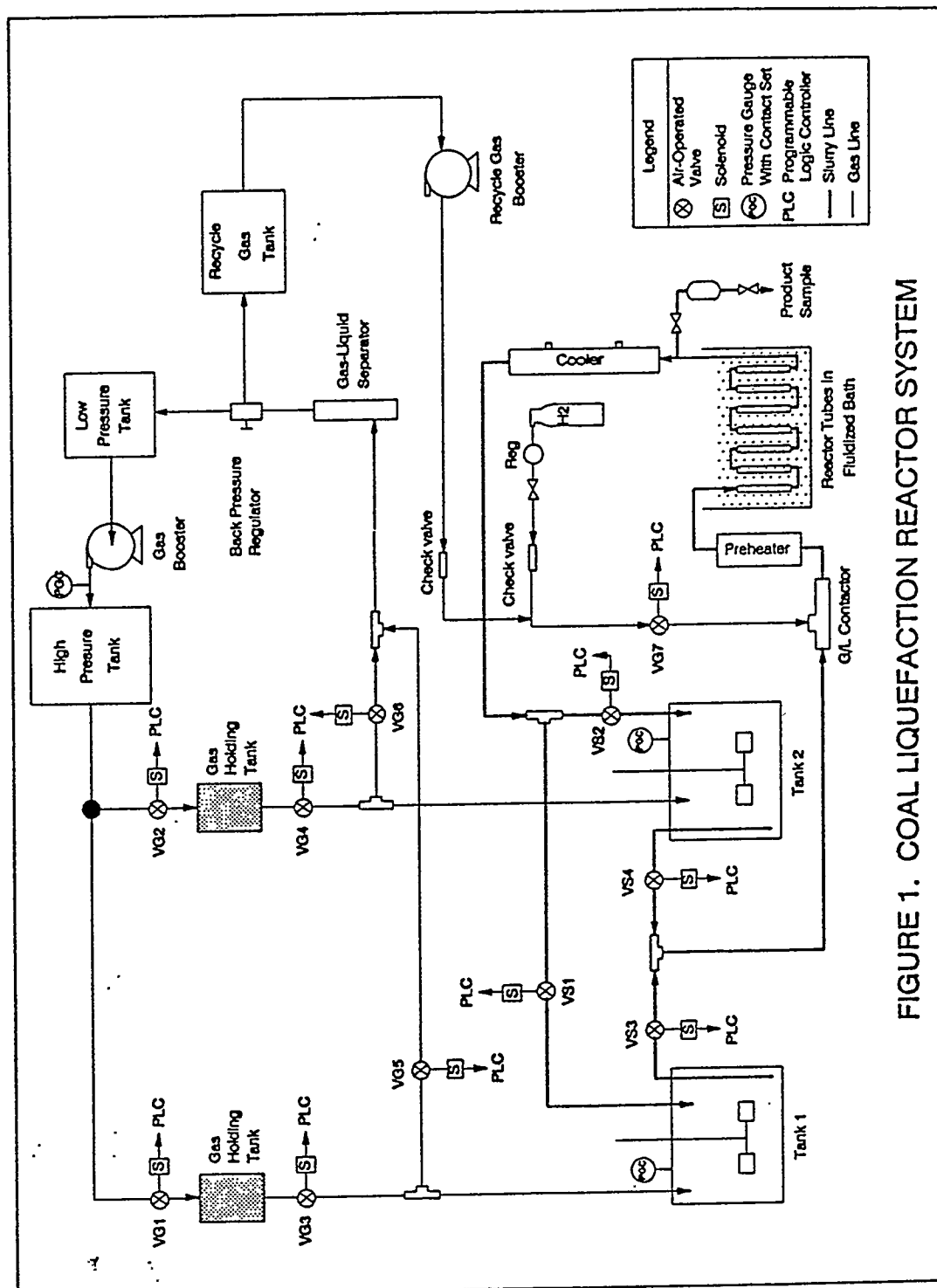
FIGURE 1. COAL LIQUEFACTION REACTOR SYSTEM

between the two tanks. The fluid flow rate can be changed simply by adjusting valve action and gas pressures, and the fluids can range from low viscosity liquids to high viscosity slurries. The complete reactor system is shown in Figure 1.

The new coal liquefaction reactor system was used in an initial study to coprocess coal with waste oil. Coprocessing coal with waste oil was also performed in conventional tubing bomb microreactors in the presence of an iron oxide precursor. Initial studies indicate the beneficial effects of coprocessing coal with waste oil, and show that the new reactor system gives better results than the tubing bomb reactor.

Coprocessing coal with waste materials such as used tires, waste plastic and waste oil, all of which have expensive disposal costs, has the potential to improve the economics of coal liquefaction. Almost 1.2 billion gallons of waste oil are generated in the United States each year, posing an environmental hazard due to its metal bearing compounds and high sulfur content. The used oil must be re-refined and hydrotreated before use as a fuel or as a lube base stock. The undissolved coal could act as a trap for the metals removed from the oil during coprocessing, while the sulfur present in the oil could produce the sulfided catalyst needed for liquefaction.

Waste oil is primarily paraffinic and is a poor hydrogen donor solvent. It does, however, contain surfactants which could be advantageous to liquefaction. Additives found in the waste oil, such as detergent/dispersant additives, oxidation inhibitors, etc., are mostly organic sulfur compounds[1]. These additives could help effectively disperse the coal and catalyst precursors throughout the coal/oil slurry during coprocessing. In addition, these additives can serve as sulfur sources to convert the catalyst precursors to the more active form. The unconverted coal could serve to trap the metals removed from the oil. The removal of metallic impurities from the oil during coprocessing of coal with a heavy oil was found to be due to deposition on the coal residue or pitch[2]. The demetallation of used oil during hydrotreatment was also found to be primarily due to the process of physical deposition on the solid catalyst bed[3].

## II. Coprocessing Coal With Waste Oil

The coal conversion to THF solubles during coprocessing with waste oil was defined as:

Conversion (Y) = 100 * (1-X) where,

$$X = \frac{W_R - W_C - W_{ash}}{W_{moisture\ ash\ free\ coal}}$$

and conversion to hexane solubles (H) was defined in a similar manner. The selectivity was defined as the ratio of conversion to hexane solubles and conversion to THF solubles i.e.,

Selectivity = 100 * S where,

$$S = \frac{H}{Y}$$

$W_R$=weight of residue remaining after THF wash,

$W_C$=weight of catalyst (it was assumed that all the iron oxide was converted to FeS)

$W_{ash}$=weight of ash in the coal

**Conversion and Selectivity.** The conversion of coal and the selectivity for the formation of oils was measured in our coprocessing studies. The selectivity as defined above is the fraction of total products from coal that are soluble in hexane. The waste oil is completely soluble in hexane before the reaction. It was assumed that the residue remaining after the solvent wash of the products was primarily unconverted coal, ash and catalyst (when used).

The catalyst precursor used in the coprocessing experiments was superfine iron oxide (SFIO) supplied by Military/Aerospace Chemicals (MACH I, INC). The amount of iron oxide used was the stoichiometric amount (2.5 wt. %) required to remove all the sulfur (1.0 wt. %) present in the waste oil before coprocessing.

**Tubing Bomb Microreactor (TBMR). (i) Effect of tetralin and catalyst.** The conversion of coal and selectivity for oils was found to be very high (85% and 70% respectively) without the addition of a donor solvent (tetralin) and catalyst precursor. This is attributed to efficient dispersion of coal fragments, due to the presence of dispersant additives in waste oil. The conversion increased slightly with the addition of either the catalyst precursor or tetralin, but the selectivity change was not significant (Figure 2).
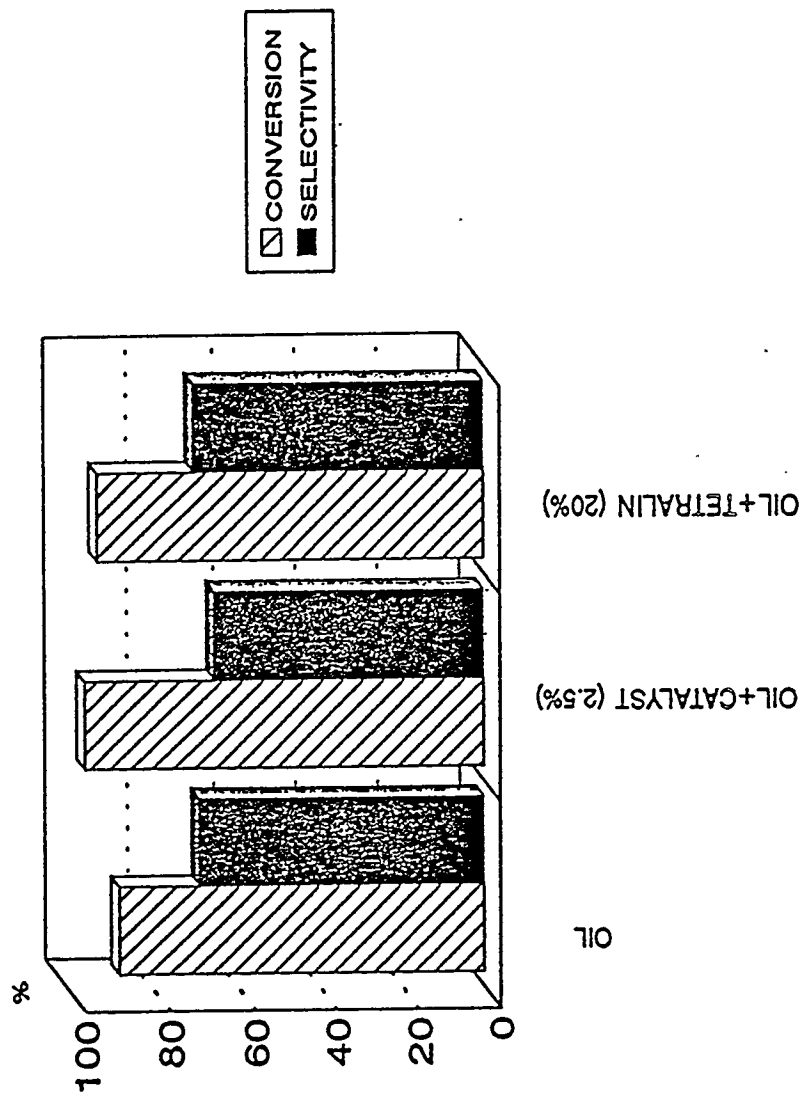
FIGURE 2.  CONVERSION AND SELECTIVITY OF COAL IN THE TUBINB BOMB REACTOR
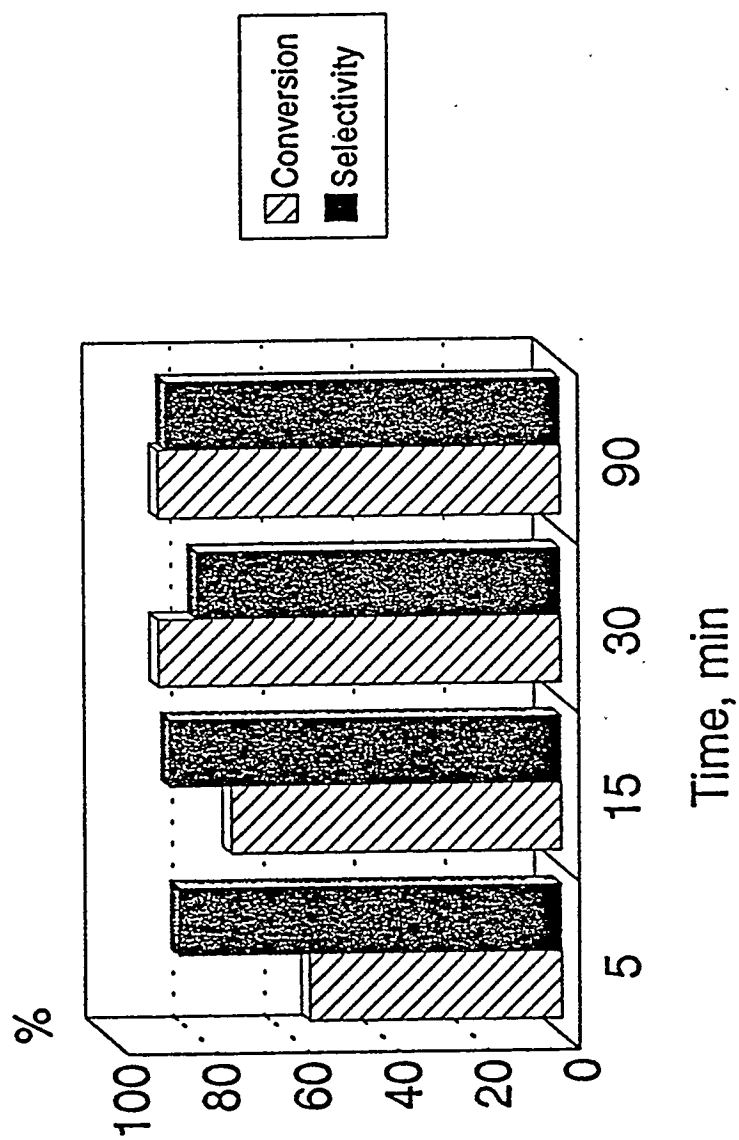
FIGURE 3. EFFECT OF TIME ON COAL CONVERSION DURING COPROCESSING (TUBING BOMB REACTOR)

**(ii) Effect of time.** Coprocessing experiments were conducted in order to study the effect of time on yields. No donor solvents or catalyst precursors were used. The conversion at the end of five minutes was only 50%, but the selectivity was relatively higher. The results are shown in Figure 3. The figure shows that the conversion and selectivity do not change significantly after a reaction time of 30 minutes. The selectivity in all cases is consistently high (more than 75%), and remains fairly constant, indicating that the coal is primarily converted to oils during coprocessing.

**(iii) Effect of dispersion.** The role of a dispersant in coal liquefaction was evaluated using a mineral oil (paraffinic oil with no additives) as the solvent and with different concentrations of a commercial dispersant additive added to the reaction mixture. The conversions were higher in the presence of the additive and increased with the concentration of the additive, but the selectivity decreased. However, a relatively lower increase in the conversion to hexane solubles was observed with the additive concentration (Figure 4). We believe the dispersant additive's function is to increase coal dispersion and dissolution. This can be seen by the increased conversion with the additive concentration, but a relatively lower increase in the conversion to hexane solubles. The selectivity and the conversion to hexane solubles can be increased by enhancing the gas-liquid mass transfer rate and by better control of the free-radical propagation rate. The jet-loop reactor system was designed for this purpose. The coprocessing results from the loop reactor system are discussed in the next section.

The typical concentration of a dispersant additive in a waste oil is a maximum of 3-6%. The shaking speed (425 rpm) of the TBMR during coal liquefaction with mineral oil and the dispersant additive was lower than that used with waste oil (720 rpm). The gas-liquid mass transfer limitations are lowered as the shaking speed is increased. A lower speed was used during liquefaction with the mineral oil to study the effect of the dispersant under conditions which could be mass transfer limiting. The conversion of coal in the mineral oil with a dispersant additive is comparable to the conversion with waste oil. However, the conversion to hexane solubles and selectivity are higher with the waste oil. These results are attributed, in part, to the higher shaking speed used while evaluating coal coprocessing with waste oil. This
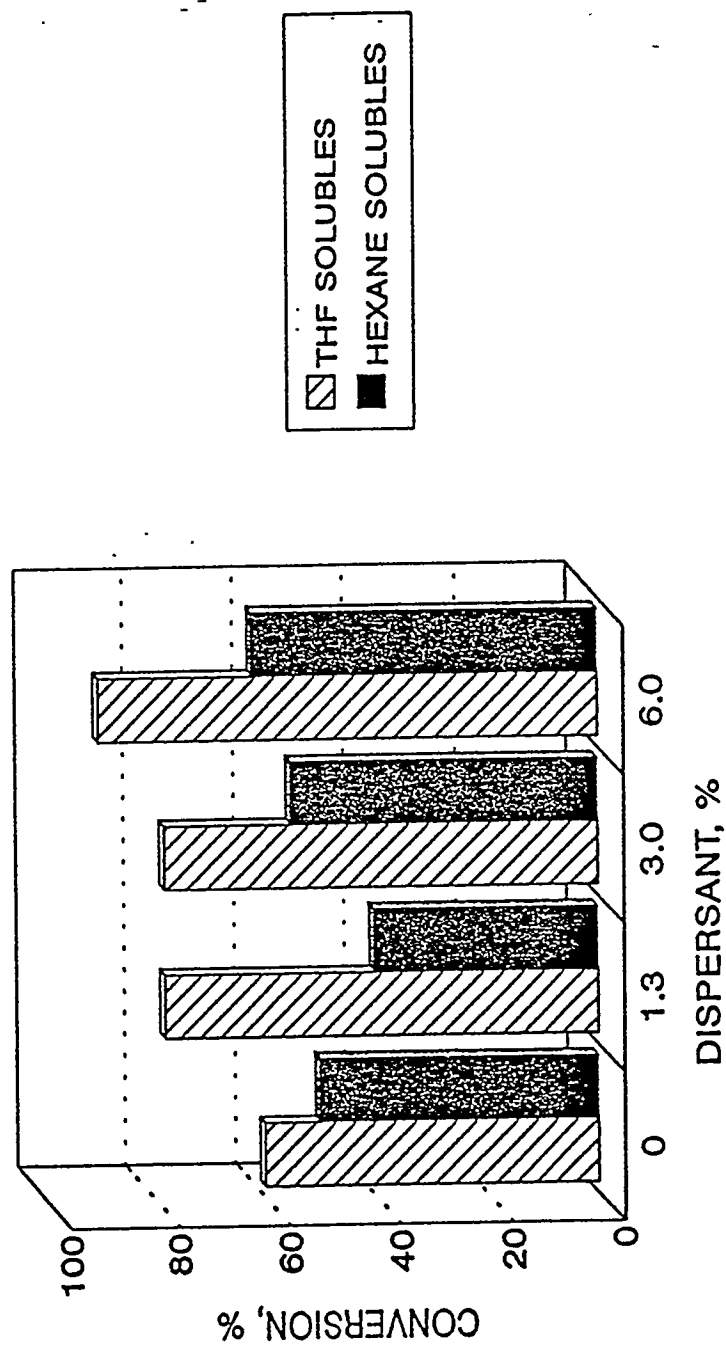
FIGURE 4. EFFECT OF DISPERSANT ON COAL CONVERSION
(TUBING BOMB REACTOR)

supports the hypothesis that the dispersant additives present in the waste oil can play a significant role in improving the coal conversion.

The results from this initial study with waste oil in the TBMR indicate the potential beneficial effects of coprocessing. The improved dispersion of the coal particles in the TBMR resulted in higher conversions (> 80%). The ratio of coal to waste oil was limited to 10 wt. % for most of this study. A few runs with higher coal concentrations (20 and 25%) resulted in a relatively lower conversion (60%) and selectivity (50%).

**Reactor System.** The samples collected in the reactor system during heat-up were used to determine the conversion of coal and selectivity for oils. The results are shown in Figure 5. At relatively low temperatures (275°C), the conversion was lower, but the conversion to hexane solubles was negative as indicated by the negative selectivity. The reactant hydrogen was introduced in this run after the temperature of the slurry at the reactor outlet reached 240°C. This was attributed to significant solvent (waste oil) incorporation into the coal. Solvent incorporation at lower temperatures has been reported by a number of investigators[4]. However, when the temperature was increased to approximately 305°C, the coal conversion was 15%, but the conversion to hexane solubles was higher as indicated by the high selectivity (90%). As the temperature increased to 320°C, the conversion increased to nearly 50% with a selectivity of over 95%.

The conversion to THF and hexane solubles from another coprocessing experiment in the loop reactor system is shown in Figure 6. The reactant hydrogen in this run was introduced after the slurry temperature at the reactor outlet reached 255°C. The results from this run confirm significant solvent incorporation into the coal at lower temperatures, which results in negative conversions. The conversion to THF solubles decreased in this run as the temperature increased from 275 to 335°C. This is believed to be due to increased solvent incorporation and the effect is evident with a further decrease in the conversion to hexane solubles for the same temperature range. The effect of solvent incorporation into coal was reduced as the temperature increased above 335°C. The conversions to THF and hexane solubles are very close at 360°C, indicating high selectivity for the formation of oils from coal.
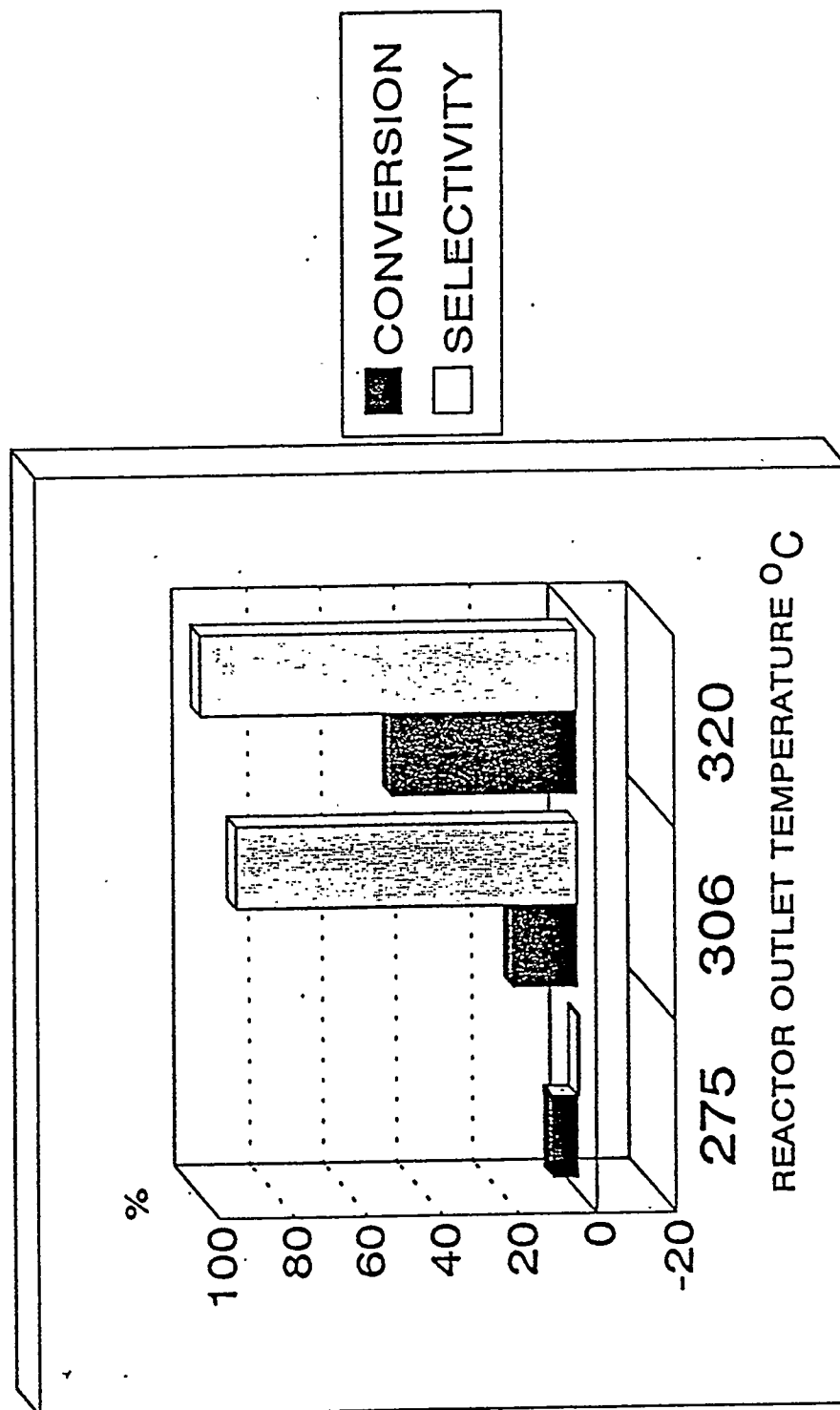
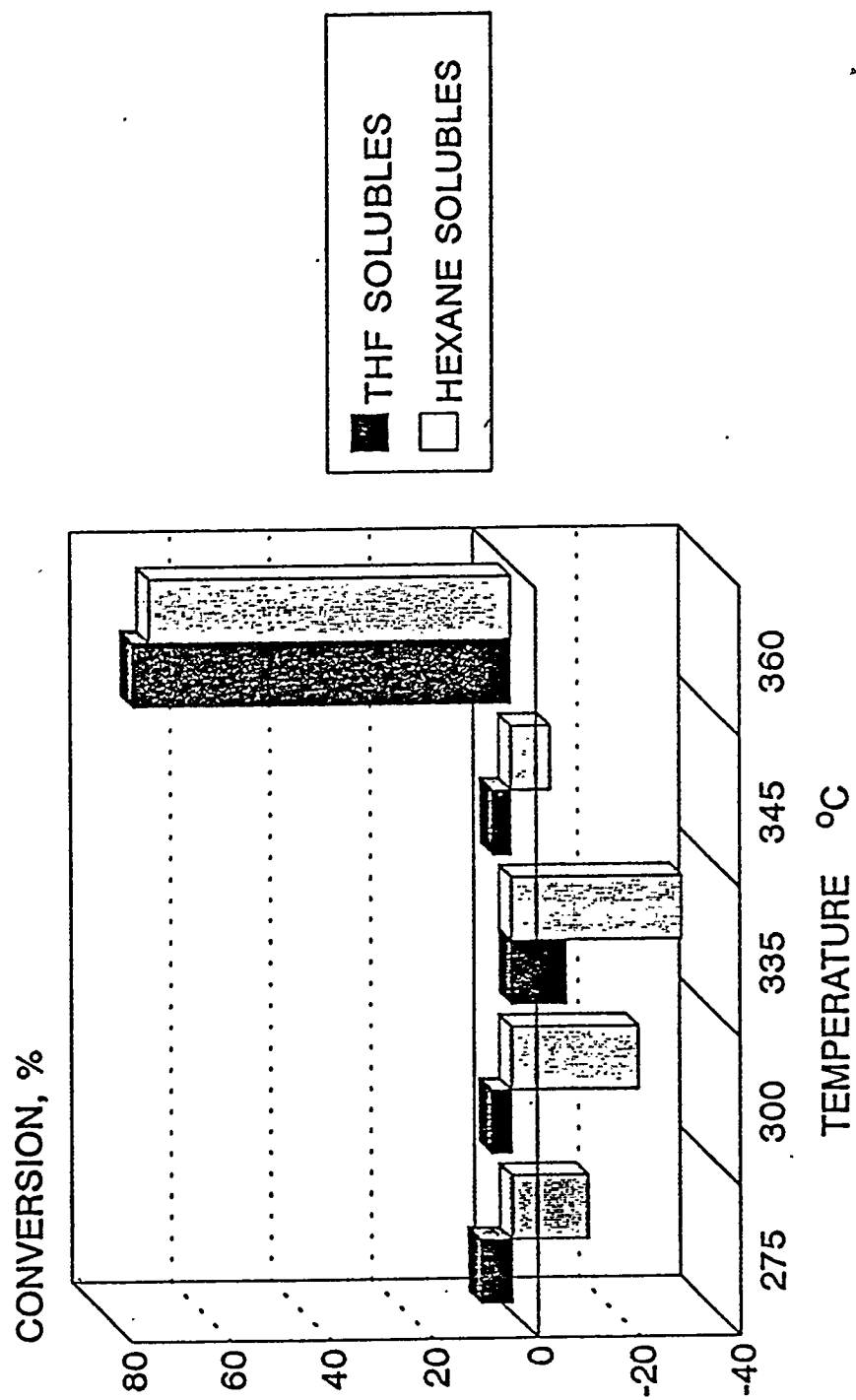FIGURE 5. CONVERSION AND SELECTIVITY DURING COPROCESSING

FIGURE 6. CONVERSION OF COAL DURING COPROCESSING IN THE REACTOR SYSTEM

In one coprocessing experiment, the temperature of the slurry coming out of the reactor was increased to 390°C and resulted in coal conversion of 83%, with a selectivity of 95%.

The conversion of coal and the high selectivity in the loop reactor system at relatively lower temperatures (< 350°C) is significant. Shaw and co-workers[5, 6] have reported recently that coal particle disintegration is characterized by mass transfer limited erosive breakage where molecular hydrogen is the principal reacting species. They found that the dissolution of coal was initiated at a lower temperature in the presence of molecular hydrogen and that the initiation temperature decreased with increased hydrogen pressure. Higher gas-liquid mass transfer rates were observed in model reactions using the loop reactor system[7, 8]. Coal conversion and high selectivity at relatively lower temperatures in the loop reactor system is attributed, in part, to the high mass transfer rates.

The results from the TBMR and jet-loop reactor system indicate very high conversions of coal in the presence of a paraffinic waste oil. The dispersion of coal fragments by the additives present in the waste oil is believed to be the primary reason for this behavior in a very poor hydrogen donor solvent. Earlier results have shown that at lower agitation rates, the undissolved coal particles adhere to one another, preventing dispersion and fragmentation of the particles[9]. The presence of a dispersant reduces agglomeration and enhances dispersion which could result in increased coal dissolution and conversion.

The selectivity was higher in the jet-loop reactor system compared to the TBMR. The loop reactor system was designed to provide enhanced gas-liquid mass transfer rates, better control of free-radical propagation, and improved dispersion characteristics as reported in the model reaction studies[7, 8]. The high selectivity in the loop reactor system is believed to be due to a combination of better control of free-radical propagation and improved mass transfer characteristics.

**Sulfur and Ash Removal.** The hexane soluble (oils) fraction was analyzed for sulfur and ash content. The reduction in the sulfur content (compared to the original waste oil) was higher (over 40%) when tetralin was present in the reaction system. This is probably due to the increased availability of hydrogen in the liquid

phase when tetralin is present, leading to increased removal of sulfur by forming hydrogen sulfide. The reduction in the ash content was greater than 70% in most of the runs. The ash reduction is believed to be due primarily to the deposition of metals on unreacted coal and residue[2, 3].

# References

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# TASK III

## Project III.3

# DIRECT LIQUEFACTION OF COAL TO CLEAN TRANSPORTATION FUELS; OPTIMIZATION OF PROCESS VARIABLES AND DETAILED CHARACTERIZATION OF PRODUCTS

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I.   Catalytic Hydroliquefaction of Coal, Catalysts, and Liquid Product Quality

(Larry L. Anderson and Win-H. Yuen)

During the year a matrix of experiments were performed to optimize reaction variables

for hydroliquefaction of coals to liquid fuels. Following our earlier work, which showed that

catalysts that performed well were those able to penetrate the coal particles during the

reaction. Water-soluble compounds containing molybdenum, tungsten, iron, cobalt, and nickel

were used as catalyst precursors. Coals tested included Blind Canyon (Utah high volatile

bituminous) from the Penn State sample bank and Wyodak (Wyoming, subbituminous).

Previous studies showed that good conversion without high gas yields could be obtained at

400°C so that temperature was used for most of the hydroliquefaction runs.

In typical runs the coal sample was impregnated with catalyst to the required loading

by using an aqueous solution of catalyst precursors. For the bituminous coal samples the

loading required water in the amount of about fifty per cent of the weight of the coal. For

the subbituminous coal the weight of water was only about thirty six per cent of the coal

weight. After impregnation of the catalyst precursor the coal was dried at 55°C under a

vacuum (-60 kPa) for six hours. The catalyst/coal mixture was then placed into a tubing

reactor, sealed, pressurized with hydrogen, and immersed into the sand bath used to heat the

reactor.

After the hydroliquefaction reaction tubing reactors were taken from the sand bath, quenched, gases were vented for analysis, and the liquids and solids were extracted with tetrahydrofuran (THF). THF was stripped from the liquids after which they were extracted with toluene. Toluene-solubles were further extracted with cyclohexane to determine oil and asphaltene yields.

Table II.1.1 gives analyses for the coals tested during the year and Table II.1.2 shows the catalyst precursors used, which included compounds of iron, cobalt, nickel, molybdenum, and tungsten. Previous reports have given the effects of pressure, temperature, demineralization, reaction time, and catalyst concentration [especially molybdenum] on the conversion and liquid yields.

Of the many catalyst precursors used the highest yields were obtained with nickel (II) hexahydrate and ammonium tetrathiomolybdate which both gave liquid yields near 80 per cent. Ammonium iron (II) sulfate hexahydrate and cobalt(II) nitrate hexahydrate gave similar results with liquids near 70 per cent. Results from runs with all of the catalyst combinations used are shown in Figures II.1.1-11. Further characterization of the liquids, especially the oil fractions, are being done using simulated distillation and chromatographic methods.

This work will be presented at the International Conference on Coal Science to be held in Canada September 12-17, 1993. The paper was accepted for oral presentation. A paper summarizing the earlier work was also presented at the American Chemical Society Meeting in Denver, Colorado on March 30, 1993. The paper was included in the Fuel Chemistry Division Preprints[1].

## REFERENCES

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## II. Coliquefaction of Coal and Waste Polymers to Produce Clean Liquid Fuels.
(Larry L. Anderson and Wisanu Tuntawiroon)

Preliminary experiments have been done to determine the suitable conditions such as temperature, pressure, reaction time and other variables on the conversion of mixtures of coal and waste polymers such as polystyrene, polyethylene, tire rubber, and commingled waste plastics. Initial experiments were thermal runs done on pure polymers, polystyrene and polyethylene. Reactions were done using the same reactors as was used in the hydroliquefaction of coal. Runs were also done using coal and rubber and coal and commingled plastic, mostly polyethylene, which was obtained from a local recycling center. Results are shown in Figures II.12-15. Although good conversion was found for pure compounds the results were much less satisfactory for commingled plastic. More appropriate conditions and/or catalysts for the coliquefaction of coal/polymer feeds are being determined. A paper describing this work has been accepted for presentation at the American Chemical Society National Meeting in Chicago, August 22-27, 1993. The paper is titled, "Coliquefaction of Coal and Polymers To Liquid Fuels.

## Table II.1.1
## Properties of Coals from the Penn State Coal Sample Bank

| Coal Sample | DECS-6 (PSOC-1543) | DECS-17 (PSOC-1554) | PSOC-1520 |
|---|---|---|---|
| Seam | Blind Canyon | Blind Canyon | Smith-Roland (Wyodak) |
| County | Emery | Emery | Campbell |
| State | UTAH | UTAH | Wyoming |
| Province | Rocky Mountain | Rocky Mountain | Northern Great Plains |
| Apparent Rank | HVA | HVA | Subbit C |
| Moisture Content (wt%, as received) | 4.73 | 3.74 | 26.74 |

Ultimate Analysis (wt% dry coal)

| | DECS-6 | DECS-17 | PSOC-1520 |
|---|---|---|---|
| Ash | 5.84 | 6.57 | 12.61 |
| C | 76.53 | 76.25 | 64.68 |
| H | 5.88 | 5.80 | 4.24 |
| N | 1.46 | 1.29 | 0.95 |
| S | 0.40 | 0.44 | 1.21 |
| 09.89 | 9.65 | 16.31 | |

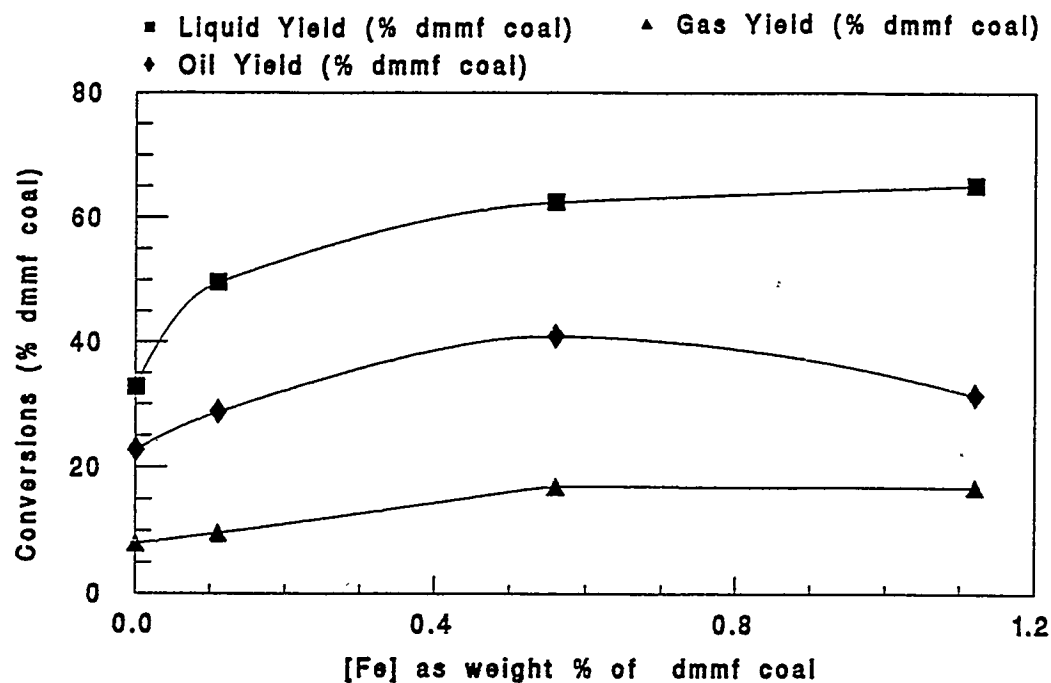| Table II.1.2 Catalysts Used in Hydroliquefaction of Coals | | | |
|---|---|---|---|
| Fe (III) & Fe (II) | Ni (II) | Co (II) | Mo & W |
| 1. Ammonium iron (III) sulfate dodecahydrate $(NH_4)Fe(SO_4)_2$ | | | 1. Ammonium tetrathio-molybdate $(NH_4)_2MoS_4$ F.W.=260.27 |
| 2. Iron (III) nitrate nonahydrate $Fe(NO_3)_39H_2O$ F.W.=404.00 | 1. Nickel (II) nitrate hexahydrate $Ni(NO_3)_26H_2O$ F.W.=290.81 | 1. Cobalt (II) nitrate hexahydrate $Co(NO_3)_2\ 6H_2O$ F.W.=291.03 | 2. Ammonium tetrathiotung-state $(NH_4)_2WS_4$ F.W.-348.18 |
| 3. Iron (III) sulfate pentahydrate $Fe_2(SO_4)_35H_2O$ F.W.=489.76 | 2. Nickel (II) sulfate hexahydrate $NiSO_4\ 6H_2O$ F.W.=262.86 | 2. Cobalt (II) sulfate hydrate $CoSO_4\ X\ H_2O$ F.W.=155.00 | |
| 4. Ammonium iron (II) sulfate hexahydrate $(NH_4)_2Fe(SO_4)$ F.W.=392.14 | 3. Ammonium nickel (II) sulfate hexahydrate $(NH_4)_2Ni(SO_4)_2$ $6H_2O$ F.W.=395.00 | | |

## FIGURE CAPTIONS

Figure II.1.1. Total liquids, oils and gases from hydroliquefaction of -60 mesh Blind Canyon (DECS-17) coal at 400 C and 2000 psi for 1 hour with ammonium iron (III) sulfate dodecahydrate as catalyst precursor.
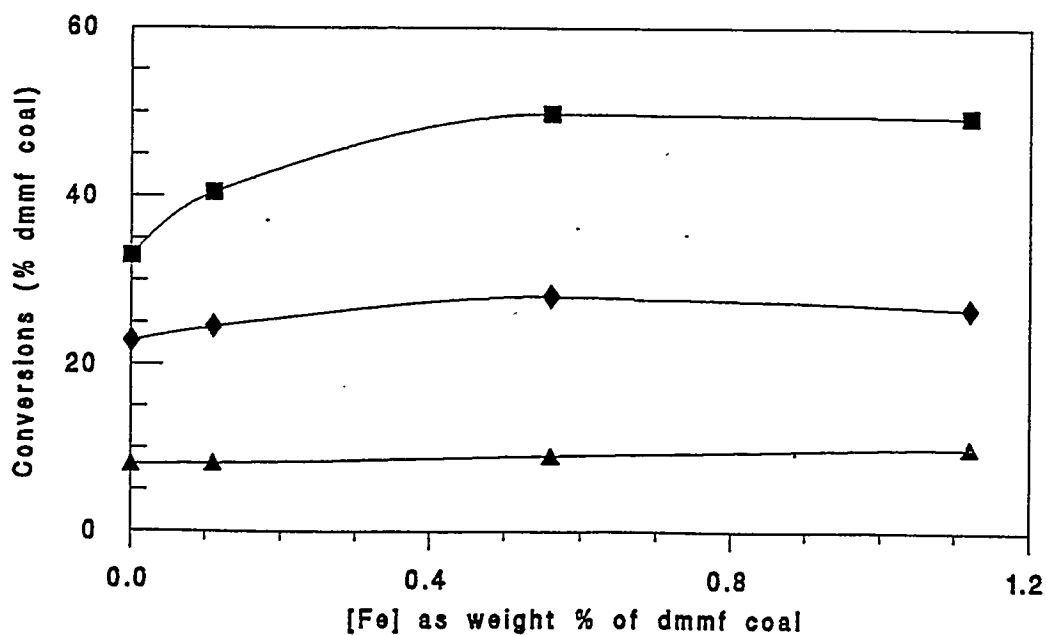
Figure II.1.2. Total liquids, oil and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C, 2000 psi for 1 hour. Catalyst used was iron (III) nitrate nonahydrate.
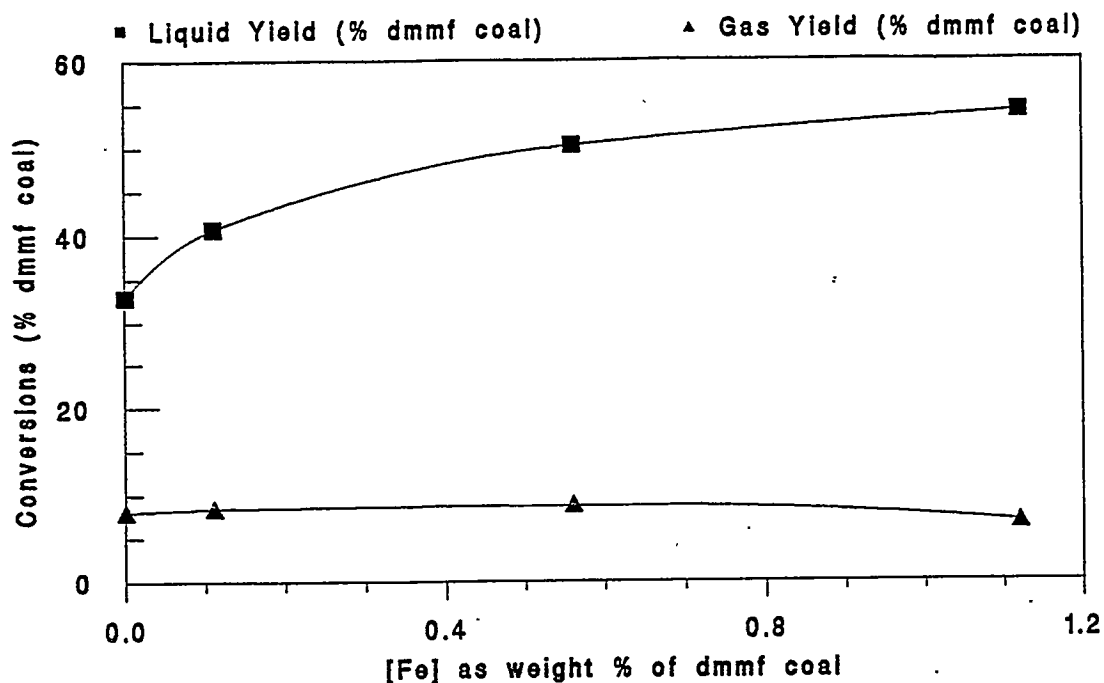
Figure II.1.3. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using iron (III) sulfate pentahydrate as catalyst.
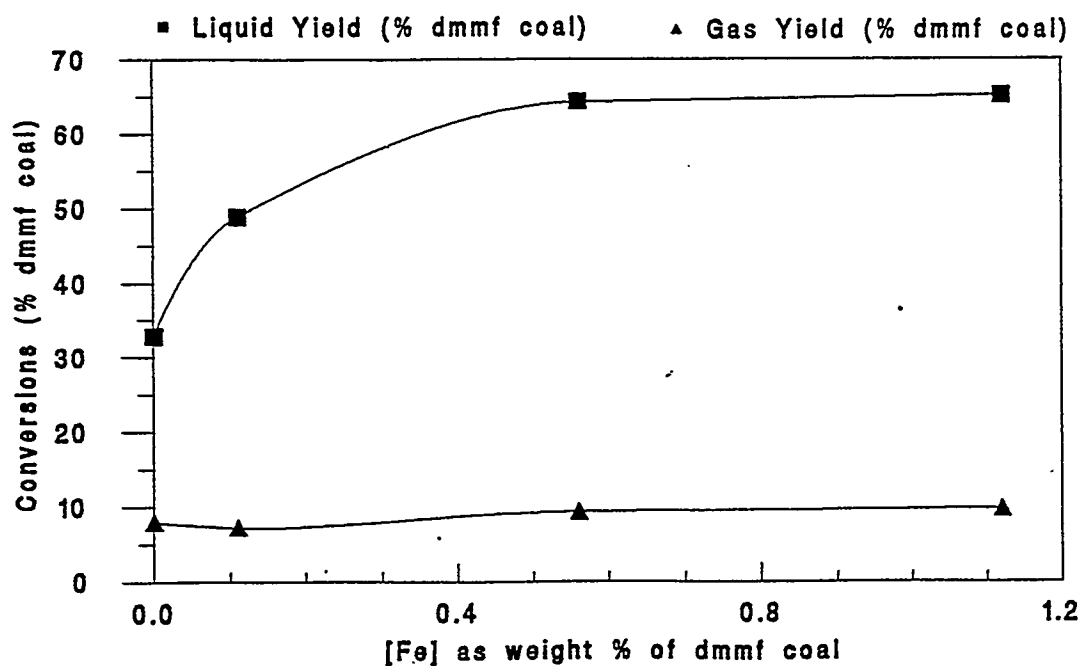
Figure II.1.4. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using ammonium iron (II) sulfate hexahydrate as catalyst.
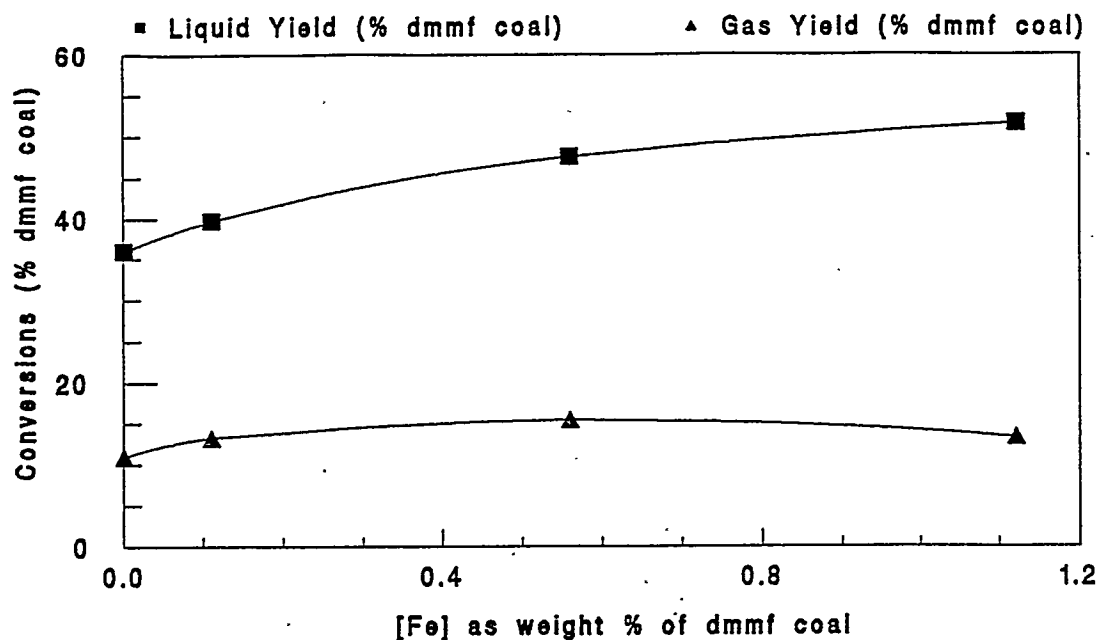
Figure II.1.5. Total liquids and gases from hydroliquefaction of -60 mesh Wyodak subbituminous coal (PSOC-1520) at 400 C for 1 hour at 2000 psi using ammonium iron (III) sulfate dodecahydrate as catalyst.
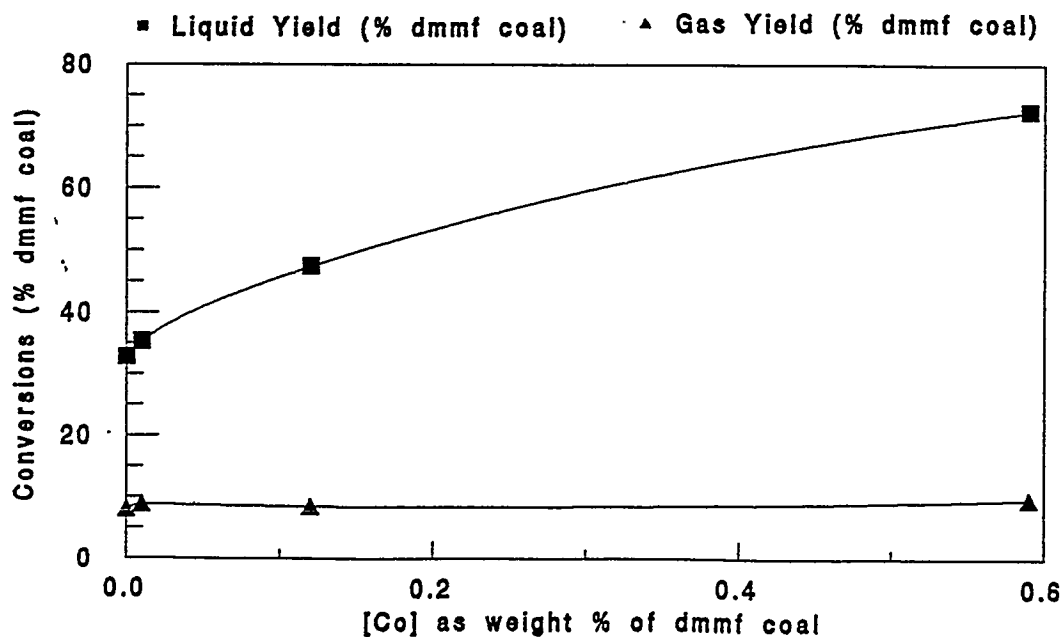
Figure II.1.6. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using cobalt (II) nitrate hexahydrate as catalyst.
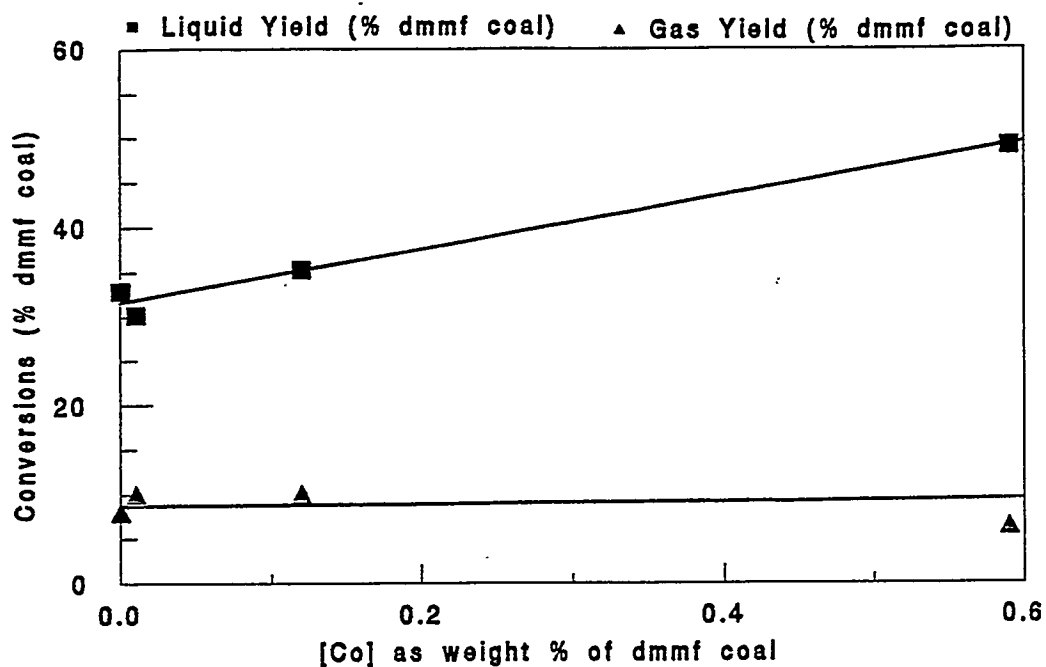
Figure II.1.7. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using cobalt (II) sulfate hydrate as catalyst.

Figure II.1.8. Total liquids, gases and oil from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using nickel (II) nitrate hexahydrate as catalyst.

Figure II.1.9. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using nickel (II) sulfate hexahydrate as catalyst.

Figure II.1.10. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using ammonium tetrathiotungstate as catalyst.

Figure II.1.11. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using ammonium tetrathiomolybdate as catalyst.

Figure II.1.12. Conversion of mixtures of coal and polyethylene in hydroliquefaction weight percent (liquid + gases) of feed material.

Figure II.1.13. Conversion of mixtures of coal and polystyrene at 400 C in hydroliquid faction reactions. Value are weight percent (liquid + gases) of feed material.

Figure II.1.14. Conversion of mixtures of coal and polystyrene at different temperatures. Value are weight percent (liquid + gases) of feed material.

Figure II.1.15. Conversion of mixtures of coal commingled plastic. Values are weight percent (liquid + gases) of feed material.

Figure II.1.1. Total liquids, oils and gases from hydroliquefaction of -60 mesh Blind Canyon (DECS-17) coal at 400 C and 2000 psi for 1 hour with ammonium iron (III) sulfate dodecahydrate as catalyst precursor.



Figure II.1.2. Total liquids, oil and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C, 2000 psi for 1 hour. Catalyst used was iron (III) nitrate nonahydrate.

Figure II.1.3. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using iron (III) sulfate pentahydrate as catalyst.
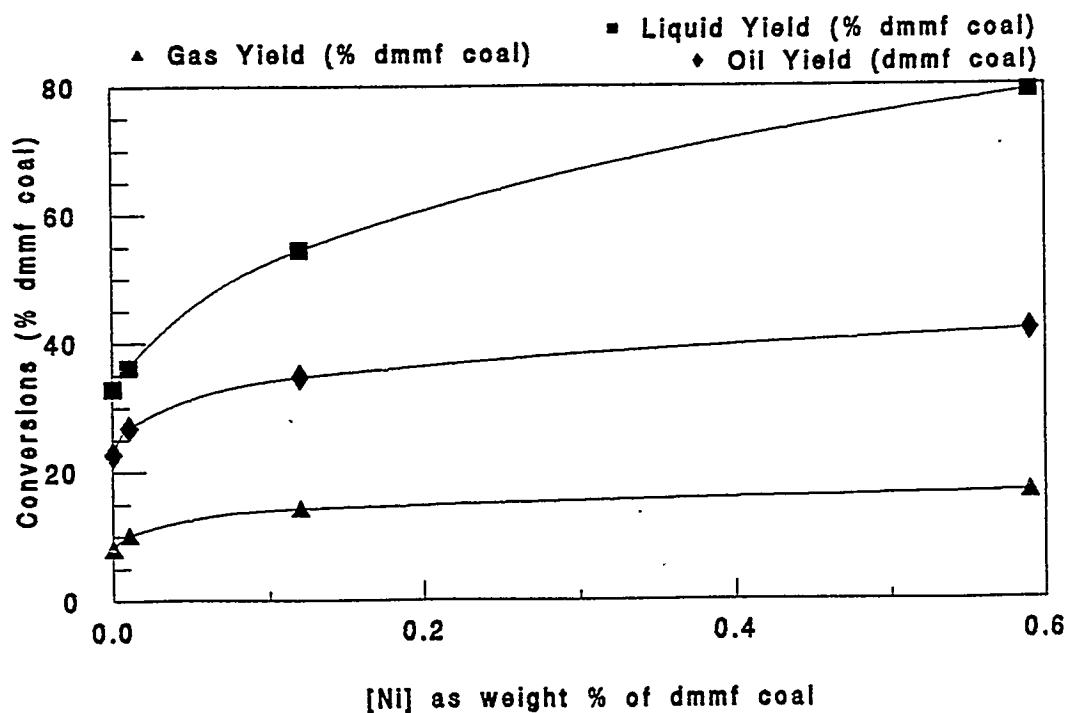


Figure II.1.4. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using ammonium iron (II) sulfate hexahydrate as catalyst.

Figure II.1.5. Total liquids and gases from hydroliquefaction of -60 mesh Wyodak subbituminous coal (PSOC-1520) at 400 C for 1 hour at 2000 psi using ammonium iron (III) sulfate dodecahydrate as catalyst.



Figure II.1.6. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using cobalt (II) nitrate hexahydrate as catalyst.

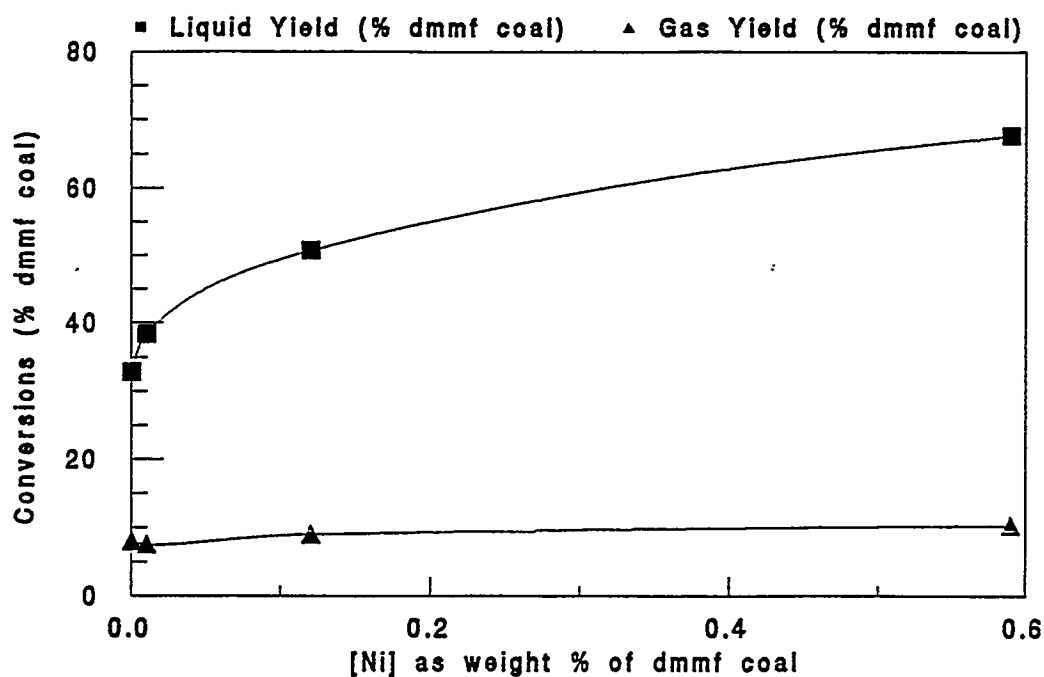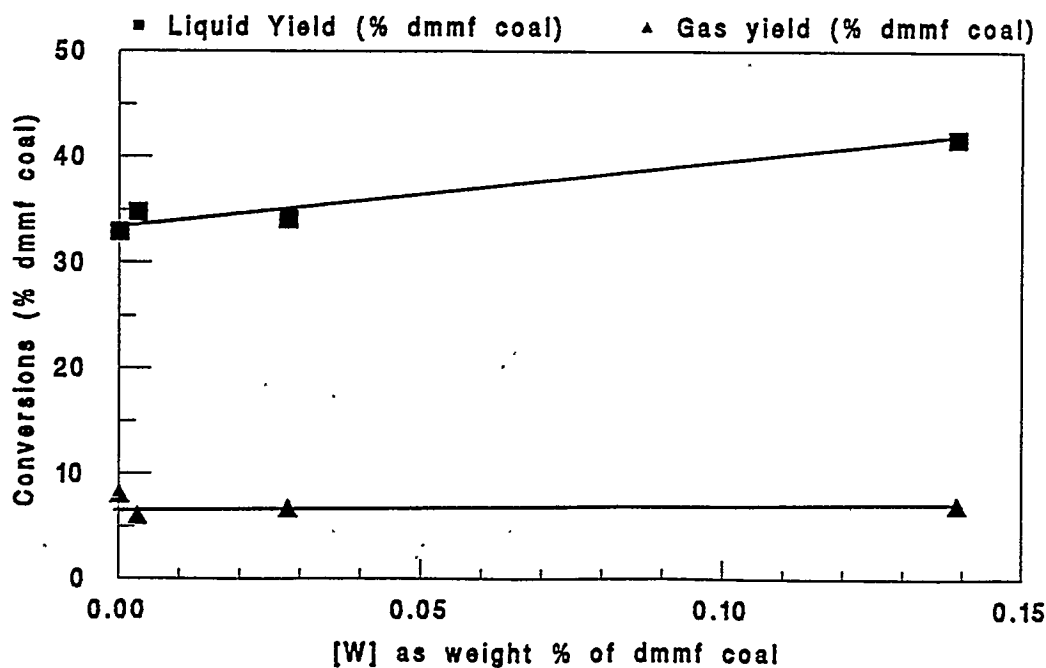Figure II.1.7. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using cobalt (II) sulfate hydrate as catalyst.



Figure II.1.8. Total liquids, gases and oil from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using nickel (II) nitrate hexahydrate as catalyst.

Figure II.1.9. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using nickel (II) sulfate hexahydrate as catalyst.



Figure II.1.10. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using ammonium tetrathiotungstate as catalyst.
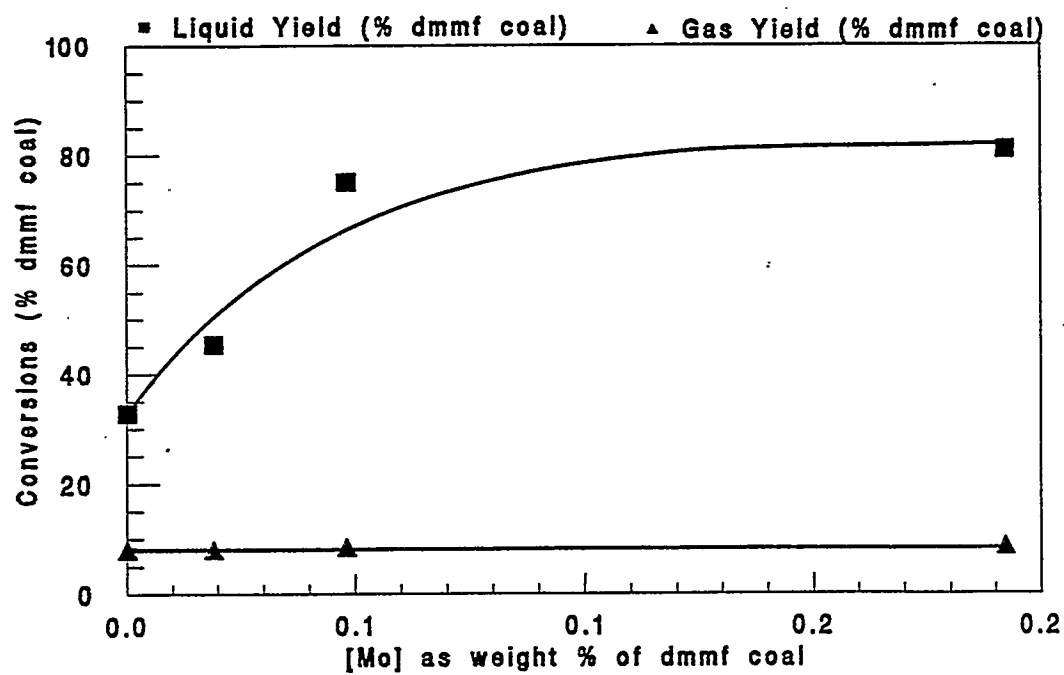
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Figure II.1.11. Total liquids and gases from hydroliquefaction of -60 mesh DECS-17 coal at 400 C for 1 hour at 2000 psi using ammonium tetrathiomolybdate as catalyst.
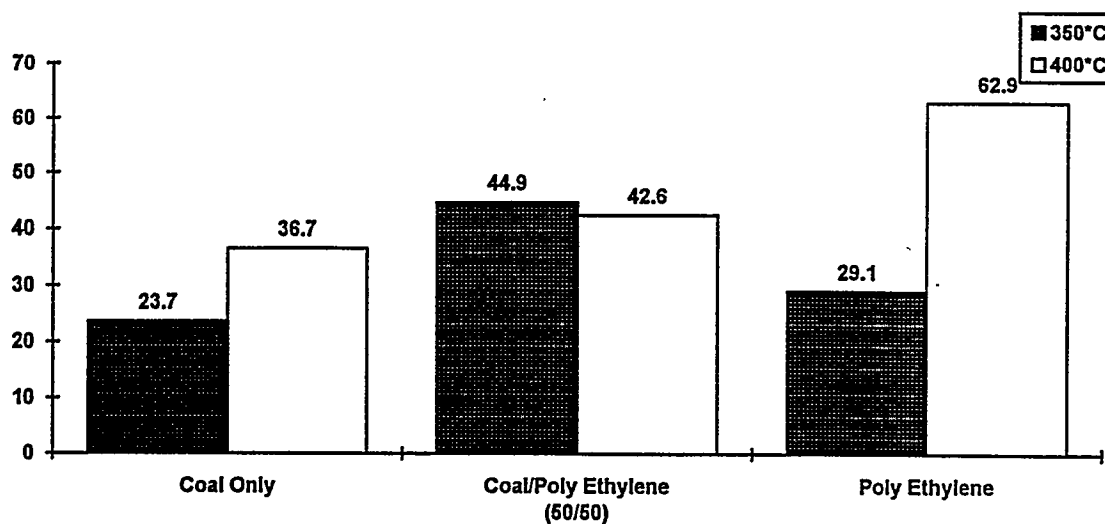
Figure II.1.12. Conversion of mixtures of coal and polyethylene in hydroliquefaction weight percent (liquid + gases) of feed material.
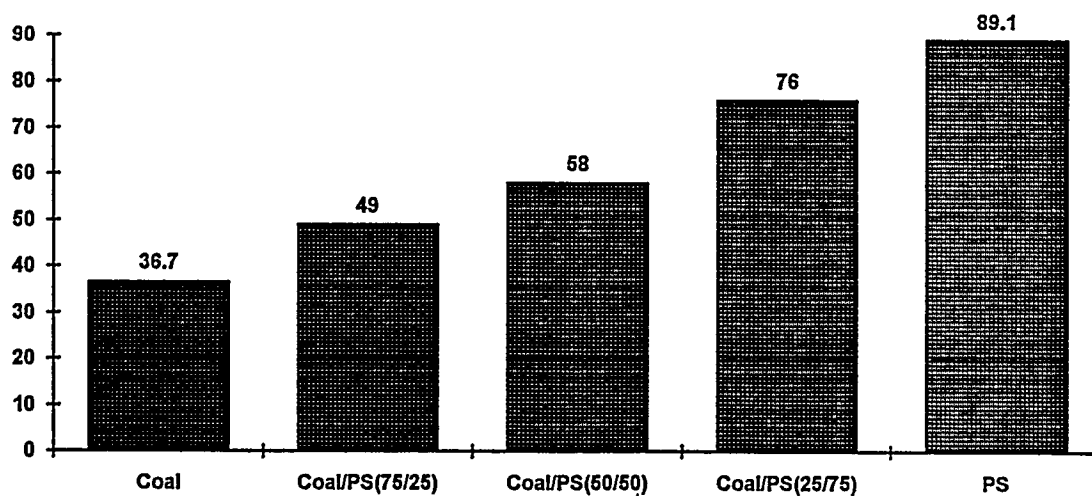


Figure II.1.13. Conversion of mixtures of coal and polystyrene at 400 C in hydroliquid faction reactions. Value are weight percent (liquid + gases) of feed material.
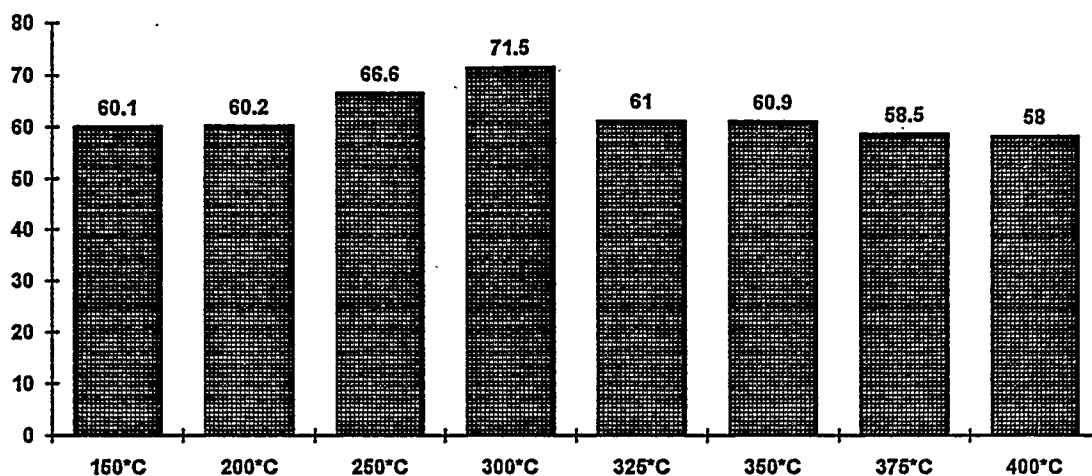
Figure II.1.14. Conversion of mixtures of coal and polystyrene at different temperatures. Value are weight percent (liquid + gases) of feed material.
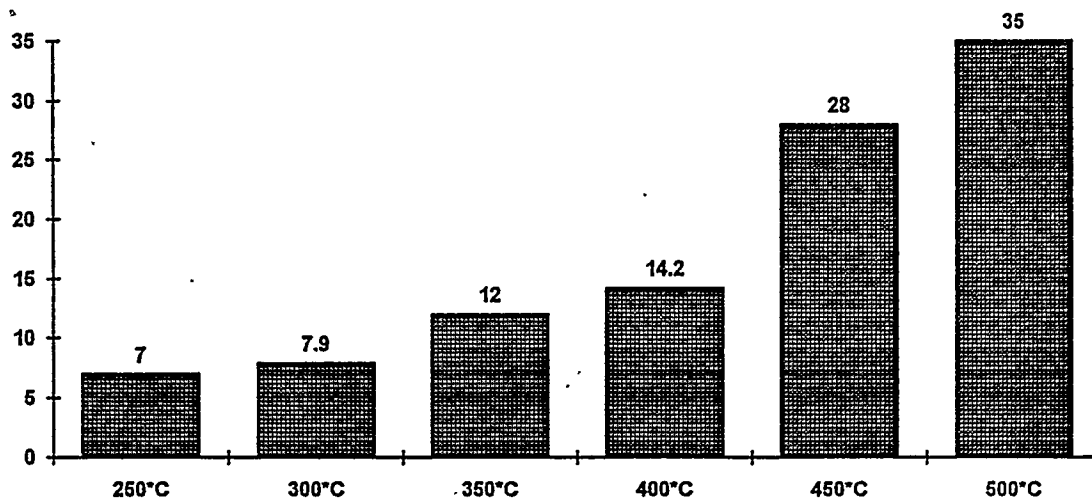


Figure II.1.15. Conversion of mixtures of coal commingled plastic. Values are weight percent (liquid + gases) of feed material.