

FLASH PYROLYSIS OF COAL IN REACTIVE AND
NON-REACTIVE GASEOUS ENVIRONMENTS

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ABSTRACT

The objective is to obtain process chemistry for the rapid pyrolysis of coal with reactive and non-reactive gases for the production of liquid and gaseous fuels and chemical feedstocks. The gaseous products were analyzed via an on-line GC and the heavy liquids were characterized by HPLC, IR and GC/MS. The total carbon conversion was higher in the presence of reactive gases than in the presence of non-reactive gases. The heat-up rate of the coal particles in non-reactive gasifying media followed the order $\text{He} > \text{N}_2 > \text{Ar}$. The total carbon conversion from New Mexico sub-bituminous coal in the presence of these gases also followed the same order. Experiments were performed in a down-flow, entrained tubular reactor under a wide range of process conditions: 700° to 1000°C, 20-1000 psi, 0.5 - 5 sec. coal particle residence time. The effects of these process variables and the physical properties of the pyrolyzing gases on the yield and the quality of products from the pyrolysis of New Mexico sub-bituminous coal will be discussed.

INTRODUCTION

The flash pyrolysis of coal is a fast, gas-phase, non-catalytic, direct coal conversion technique for the production of gaseous hydrocarbons and significant quantities of aromatic liquid hydrocarbons for distillate fuels and chemical feedstocks. Pyrolysis is a complex process during which several chemical reactions occur simultaneously. A general concept of the pyrolysis process is shown in Figure 1.

Considerable amount of work has been done in the past on pyrolysis of coal which was summarized recently in a classic review by Howard (1). The experimental techniques, reaction conditions and the coal used were so diversified that the information available is of limited value in a process point of view. We have, therefore, undertaken a systematic generalized study to methodically develop and build a reliable data base on the flash pyrolysis of coals with reactive and non-reactive gases. The choice of inert gas is based on covering a range of physical property effects, i.e., heat capacity, thermal conductivity and diffusivity etc. The choice of reactive gas is based on the type of gases usually formed in the pyrolytic reactions of coal. The results obtained from the pyrolysis of New Mexico sub-bituminous coal in H₂, CH₄, He, N₂ and Ar atmospheres are presented in this paper.

EXPERIMENTAL

New Mexico sub-bituminous coal ground to less than 100 mesh and vacuum dried at 70°C was used in all pyrolysis experiments. The ultimate analysis of the coal is given in Table 1.

The experimental equipment used in these studies (Figure 2) is a highly instrumented 1-in.-i.d. by 8-ft-long entrained downflow tubular reactor system, which has been described in detail (2). Coal particles 150 micrometers or less in diameter are fed by gravity into the top of the reactor from a coal feeder enclosed in a high pressure vessel. The pyrolysis gas enters through a preheater, contacting the coal particles at the

top of the 8-ft reactor. The heat transfer, pyrolysis, and secondary reactions take place as the coal and entraining gas meet and flow concurrently down the reactor. Below the reactor is a 3-ft forced air cooling section in which the product-laden gases are cooled to 250° to 300°C. The char is removed from the effluent gas in a high pressure vessel which is maintained at 250° to 300°C to prevent liquid product condensation. The heavy liquid products in the effluent gas are removed in two condensers, one water cooled (15°C) and the other Freon cooled (-40°C). The remaining gases are then reduced to atmospheric pressure, measured via a positive displacement meter, and vented to the atmosphere.

The velocity of the coal particle through the reactor is calculated by adding its free-fall velocity based on Stoke's law to the down-flow gas velocity. The residence time is determined from the length of the reactor and the total coal particle velocity.

Product yields, as a function of coal particle residence time, are determined by sampling from taps located at 2-ft intervals along the length of the reactor. The product analysis is accomplished via an on-line, programmable gas chromatograph (GC) which determines CO, CO₂, CH₄, C₂H₄, C₂H₆, and BTX (benzene, toluene, and xylene) concentrations every 8 minutes. Products heavier than BTX (>C₉) can not be measured on-line because of condensation in the sample lines and the temperature limit (230°C) of the gas chromatograph. These products are collected in the condensers and analyzed at the end of each experiment. Selected liquid samples are subjected to elemental analysis and GC/MS studies.

The duration of an experiment is at least one hour and steady state is achieved in 3 to 4 minutes. Coal feed rates are usually about 500 g/hr. To facilitate uniform flow, 10% by wt Cab-0-Sil, a silica flour, is added to the coal. The non-reactive gases used were the inert gases, argon, helium and nitrogen. The reactive gases included hydrogen, methane and carbon monoxide. Yields are based on the percent of the carbon in the feed coal which is converted to products and determined as follows:

$$\% \text{ Carbon converted to product} = \frac{\text{Carbon in Product, g} \times 100}{\text{Carbon in Feed, g}}$$

A sum of the gaseous and liquid product yields, expressed as % carbon converted, will give the total carbon conversion due to pyrolysis. From feed rates, product yields, and an elemental analysis of the feed coal and char produced, a complete material balance can be made.

RESULTS AND DISCUSSION

Coal particles entering the reactor are heated to reaction temperature by convection from the preheated gas and by radiant heat from the reactor wall. In calculating the coal particle heat-up rate due to convection, a number of experimental configurations were considered for which heat transfer coefficients had been determined. These included heat and mass transfer from a falling water droplet (3), heat transfer to a fluidized-bed of particles (4), heat transfer to particles at Reynolds numbers greater than 500 (5), heat transfer in a distended bed of particles (6), and the theoretical heat transfer between a particle and a stagnant fluid. Since the Reynolds number for the coal particles falling through the gas in our work is very low, less than 10, neither the fluidized-bed nor the high Reynolds number work seemed appropriate. Also, the fluidized-bed work gave very low heat transfer coefficients, which were attributed to excessive bypassing of gas through the bed during the experiments (7). Since the work on distended beds showed good correlation of data from void fractions of 0.45 to 0.78, this appeared to provide the closest approach to our experimental conditions. The expression used is as follows (6):

$$Nu = 2.06 N_{RE}^{0.425} N_{PR}^{1/3}$$

where $Nu = \text{Nusselt number} = \frac{h_g D_p}{k_g}$ (1)

$$N_{RE} = \text{Reynolds number} = \frac{D_p V \rho_g}{\mu_g}$$

$$N_{PR} = \text{Prandtl number} = \frac{C_p \mu_g}{k_g}$$

h_g = heat transfer coefficient for gas film,
 D_p = diameter of particle,
 K_g = thermal conductivity of gas,
 V = velocity of particle through gas (free fall),
 ρ_g = density of gas,
 μ_g = viscosity of gas, and
 Cp_g = heat capacity of gas.

By substitution

$$h_g = \left(\frac{2.06 K_g}{D_p} \right) \left(\frac{D_p V \rho_g}{\mu_g} \right)^{0.425} \left(\frac{Cp_g \mu_g}{K_g} \right)^{1/3} \quad (2)$$

and

$$M Cp_s \frac{dT_s}{dt} = h_g A_s (T_g - T_s), \quad (3)$$

$$dT_s = \frac{h_g A_s}{M Cp_s} (T_g - T_s) dt,$$

$$t = \frac{M Cp_s}{h_g A_s} \ln \left(\frac{T_g - T_i}{T_g - T_s} \right), \quad (4)$$

Where

Cp_s = heat capacity of coal
 M = mass of coal particle
 A_s = area of coal particle
 T_g = temperature of gas
 T_s = temperature of coal particle
 T_i = initial temperature of coal particle
 t = time to raise the particle temperature from T_i to T_s

Equation (2) was used to calculate the gas-film heat transfer coefficient between the coal particle and the preheated gases hydrogen, methane, carbon monoxide, helium, nitrogen, and argon. The values of gas-film heat transfer coefficient for various pyrolyzing gases under a set of arbitrarily chosen conditions are given in Table 2. Table 2 shows that for reactive gases, the heat transfer coefficient decreased in the following order: $H_2 > CH_4 > CO$; for non-reactive gases the order is found to be $He > N_2 > Ar$. Equation (4) was used to calculate the coal particle

temperature as a function of time and is plotted in Figure 3. Figure 3 shows that hydrogen gives the highest heat-up rate and argon the lowest; among the non-reactive gases, helium gives the highest rate. Although calculations of the final temperature of the coal particle were made, these should be considered as limiting values because chemical energy exchange and devolatilization also take place.

Calculations of the radiant heat transfer from the hot reactor wall to the coal particles indicate this contribution to be about 5 to 10% of the total heat transfer. Since these calculations are probably not accurate to better than $\pm 10\%$, radiant heat transfer was not included in the coal particle heating rate or the temperature estimates.

When pyrolysis took place in the presence of non-reactive gases, the total carbon conversion followed the order: $\text{He} > \text{N}_2 > \text{Ar}$. For example, at 900°C and 200 psi pressure of He, N_2 and Ar, the total carbon conversions were: 21.0%, 20.1% and 14.5%, respectively (Table 3). This includes the yields due to hydrocarbon gases, oxides of carbon, BTX and heavy liquids. In runs using N_2 or Ar as the pyrolyzing gas, difficulties were experienced in measuring the CO produced which arose as a result of inadequate resolution of this component from the solvent peak (N_2 or Ar). Hence, the CO yields from helium pyrolysis runs under identical conditions were used to determine the total carbon conversion in these cases. This could introduce some error in the total carbon conversion data for N_2 and Ar pyrolysis runs. In the case of reactive gases, the total carbon conversions were 37.6%, 30.4% and 17.5% respectively for H_2 , CH_4 and CO atmospheres. There is a significant increase in total conversion in hydrogen and methane atmospheres over other inert gases. Though CO is a reactive gas, no enhancement in pyrolysis yields were noticed under the conditions investigated. The decrease in the gas film heat transfer coefficient for reactive and non-reactive gases and the total conversion both follow the same trend within each category. Thus, there appears to be a relationship between the heat transfer coefficient (or the heat-up rate of the coal particles) and total carbon conversion (Figure 4).

The effect of gas pressure on total carbon conversion was also investigated (Figure 5). As expected, hydrogen gave the highest carbon conversion. However, increasing the hydrogen pressure from 100 psi to 500 psi had little influence on the total conversion. The effect of pressure on total conversion was marked for all other gases. The maximum in the total carbon conversion and the pressure at which it occurs clearly seem to be dependent on the pyrolysis temperature. These differences indicate that a strong relationship exists between coal devolatilization and the transport properties of the pyrolyzing gases. More experiments are planned to understand these effects on coal pyrolysis in greater detail.

The flash pyrolysis of coal produces a wide range of gaseous and liquid products. The gas stream is mostly made up of methane, ethane, ethylene, CO and CO₂ besides the entraining gas. A typical pyrolysis product distribution is shown in Figure 6. Similar product distributions were obtained for other pyrolysis atmospheres and other conditions (8). Among these pyrolysis products, ethylene showed greatest variation in its yield. This is shown in Figure 7. Under all conditions, ethylene was produced in characteristically higher quantities in methane atmosphere (which we term "flash methanolysis") than in the presence of any other gas used. As high as 10.5% carbon in the feed coal was converted to ethylene at 825°C and 50 psi CH₄ pressure. This is approximately two times higher than that with the inert gases under similar conditions (10.5% with methane and 4.9% with helium). From several blank experiments, in the absence of coal, it was determined that homogeneous decomposition of methane does not occur in the reactor (8). A free-radical mechanism for the enhanced yield of ethylene during flash methanolysis of Douglas Fir wood, in the same pyrolysis reactor, was recently proposed (9). Figure 7 also reveals that in all cases, the ethylene yield decreased with gas pressure.

As expected, high yields of gaseous hydrocarbons were obtained when pyrolysis took place in hydrogen atmosphere. The total gaseous hydrocarbon yield ranged from 8.9% C at 800°C and 50 psi to 28.0% C at 900°C and 500 psi. The yields of both ethane and ethylene decreased with

increase in the pressure of hydrogen. In non-reactive gas media, no ethane was produced in measurable quantities in most instances. Among non-reactive gases, the yield structure was comparable between helium and nitrogen atmospheres and a representative case is shown in Table 3 in which the product yields from the pyrolysis of New Mexico coal in various gasifying media, under identical conditions, are compared. From the same table, it could be seen that in argon atmosphere, the yield of gaseous hydrocarbons and other products are considerably low.

The yield of CO₂ was about 1-2% C under all conditions in the presence of all gases with the exception of CO. Higher yields of CO₂ were obtained (7-9% C) when the pyrolyzing gas was CO. This is believed to be due to disproportionation of CO under the reaction conditions.

The liquid products (excluding process water) consist of two fractions: the "light oil" fraction, boiling between 60° and 150°C, and the "heavy liquid" fraction, boiling above 150°C. This classification is arbitrary, and various distillation ranges are used by different workers. In the BNL work, usually more than 90% by weight of the "light oil" is made up of benzene, toluene, and xylene, and the "heavy liquids" or "tars" contain condensed ring aromatic structures and high molecular weight compounds with high aromaticity (10).

"Light Oil" from coal is a potential source of some key chemicals such as benzene-toluene-xylene (BTX). Even though the exact mechanism of their production is not known, it is generally thought to consist largely of secondary pyrolytic reactions. Prolonged heating of the primary devolatilization products induces cracking and if hydrogen is available, stable lighter products are formed. Thus, the yield of light oil depends on the residence time of the tar vapor in the pyrolysis reactor and on the temperature. With longer residence times, more light oil is formed at the expense of the heavy liquids (tar). Too high a temperature (>900°C) results in an increased yield of gases at the expense of both light oil and tar.

BTX yield ranged 1-3% C in the case of CO and non-reactive gases; up to 7% carbon in the feed was converted to BTX during flash hydrolysis and flash methanolysis experiments. The IR spectra of light oils, free from process water, obtained from a series of flash hydrolysis experiments with New Mexico sub-bituminous coal show (i) strong absorption in the low frequency range between 900 and 675 cm^{-1} , typical of aromatic ring C-H out-of-plane bending, (ii) strong absorption in the medium frequency range between 1600 and 1580 cm^{-1} due to C=C stretching, and (iii) moderate absorption between 3100 and 3000 cm^{-1} characteristic of the aromatic C-H stretch. Weak absorptions at 2926, and 2853 cm^{-1} ($\nu_{\text{C-HCH}_3}$, $\nu_{\text{as CH}_2}$, and $\nu_{\text{s CH}_2}$, respectively) indicate that the extent of alkyl substitution is small.

Phenols, if any, were present only in very small amounts in the light oils produced from the flash pyrolysis of New Mexico sub-bituminous coal. The IR spectra of light oils produced at 900°C at 20, 60, and 200 psi showed absorption due to -OH groups to be present only in the 20 psi sample, and this spectrum was similar to the one obtained at 800°C and 200 psi. An increase in the hydrogen pressure or residence time of the volatiles tends to decrease the amount of phenols in the product stream, and temperatures above 900°C and hydrogen pressures above 50 psi favor the dehydroxylation/decomposition reactions of phenols. The light oil from the inert helium pyrolysis at 900°C and 50 psi had a composition qualitatively similar to that from flash hydrolysis under the same conditions, but for different reasons: In the presence of external hydrogen, the phenols initially formed undergo decomposition, but during inert gas pyrolysis the conditions are not favorable for initial phenol formation.

Heavy liquid or tar is an inherent product of coal devolatilization. Its composition is much more complex than that of light oils. Tar accounts for only a small amount of the carbon in the feed coal that is converted (0-5% C). Tars from selected runs were subjected to detailed investigation, but the data available at present are limited.

A basic problem in identifying the individual components in a tar is that compounds of widely varying polarities are present, precluding GC/MS analysis of the tar as such. By fractionating the tars on the basis of solubility in selected solvents and analyzing the fractions for individual compounds, valuable information could be obtained.

In one scheme of solvent fractionation, the tar is separated into oil (hexane soluble), asphaltenes (hexane insoluble but benzene soluble), and pre-asphaltenes (benzene insoluble). The yields of total tar and tar fractions from selected pyrolysis runs are shown in Table 4. The pre-asphaltenes, which include compounds of high polarity, comprise only a very small part of the tar. Except when the pyrolysis gas is methane, an increase in tar yield is accompanied by an increase in oil yield. The tar yield from flash methanolysis is between those from helium pyrolysis and from hydroxyrolysis. In the case of helium pyrolysis, the tar yield tends to increase with increasing pressure.

The individual components in an oil sample from the flash hydroxyrolysis of New Mexico sub-bituminous coal at 1000°C and 25 psi hydrogen pressure were identified with capillary GC/MS which is shown in Figure 8. The peaks are identified in Table 5. Naphthalene is the most abundant product in this tar; higher-ring aromatic compounds and heteroatom containing specie account for less than 10% of the total. No sulfur containing compounds were detected. The reason for the presence of relatively large amount of acenaphthylene in this tar is not known at present.

CONCLUSIONS

The conclusions which can be drawn from this work thus far are as follows:

1. Under the same conditions, the total carbon conversion was higher in the presence of reactive gases than in the presence of non-reactive gases. Among the reactive gases, the total carbon conversion followed the order: $H_2 > CH_4 > CO$. Among the non-reactive gases, the total carbon conversion followed the order: $He > N_2 > Ar$.

2. Pyrolysis in methane was characterized by high yields of ethylene and liquid products. As high as 10.5% carbon in the feed coal was converted to ethylene at 825°C and 50 psi. The ethylene yield during pyrolysis of coal with methane was approximately two times higher than that with the inert gases under similar conditions (10.5% CH₄ vs 4.9% with He at 825° to 850°C and 50 psi).

3. Phenols, if any, were present only in very small amounts in the light oil fraction of the liquid products. In the presence of external hydrogen, the phenols formed undergo decomposition, but during the inert gas pyrolysis the conditions are not favorable for initial phenol formation. An increase in the hydrogen pressure or residence time of the volatiles tends to reduce the amount of phenols in the product stream, and temperatures above 900°C and hydrogen pressures above 50 psi favor the dehydroxylation/decomposition reactions of phenols.

4. In the case of helium pyrolysis, the tar yield tends to increase with increasing pressure. The tar yield from methane pyrolysis is between those from helium pyrolysis and from hydrolysis.

5. The pre-asphaltenes, which include compounds of high polarity, comprise only a very small part of the tar. Except when the pyrolysis gas is methane, an increase in tar yield is accompanied by an increase in oil yield.

6. A correlation appears to exist between the total carbon conversion to gaseous and liquid products and the heat-up rate of coal particles for inert gases in the order of He > N₂ > Ar. Uncovering other effects of heat-up rate of coal particles on the pyrolytic behavior of coals require further investigation.

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Table 1

Ultimate Analysis (Wt % Dry) of New Mexico Sub-Bituminous Coal
Used in Pyrolysis Experiments

Carbon	59.3
Hydrogen	4.2
Oxygen ^a	16.8
Nitrogen	1.2
Sulfur	0.8
Ash	17.7

a) By difference.

Table 2

Calculation of Gas Film Heat Transfer Coefficients
for Various Pyrolyzing Gases

Coal Particle Size - 100 micrometers
Coal Particle Initial Temp. - 20°C
Gas Conditions - 900°C and 1000 psi

Gas	h_g (Cal/cm ² -K ⁰ -sec)
Hydrogen	2.21×10^{-1}
Methane	2.02×10^{-1}
Helium	1.73×10^{-1}
Carbon Dioxide	9.9×10^{-2}
Carbon Monoxide	7.7×10^{-2}
Nitrogen	7.16×10^{-2}
Argon	4.96×10^{-2}

Table 3

Product Yields from the Flash Pyrolysis of New Mexico Sub-bituminous Coal at 900°C and 200 psi Coal Residence Time: 2.4-4.6 sec.			
Entraining Gas	He	N ₂	Ar
<u>% Carbon Conv. to Prod.</u>			
CH ₄	8.1	10.2	2.6
C ₂ H ₄	3.5	0	3.3
C ₂ H ₆	0	0.7	0
Total Gaseous H.C.	<u>11.6</u>	<u>10.9</u>	<u>5.9</u>
BTX	1.8	2.7	2.0
>C ₉	1.2	0.4	0.8
Total Liquid H.C.	<u>3.0</u>	<u>3.1</u>	<u>2.8</u>
CO	4.4	(4.4)	(4.4)
CO ₂	2.0	1.7	1.4
Total CO _x	<u>6.4</u>	<u>6.1</u>	<u>5.8</u>
Total Carbon Converted	<u>21.0</u>	<u>20.1</u>	<u>14.5</u>

() CO could not be resolved from N₂ or Ar on the on-line GC.
The values in parenthesis are CO yields from inert helium
pyrolysis runs under identical conditions. This could
introduce some error in the determination of the total
carbon conversion.

Table 5

Composition of Heavy Liquids from the
Flash Hydropyrolysis of New Mexico Coal

Peak No.	Component	% Peak Area
1	xylene	0.54
2	xylene	0.27
3	phenol	3.07
4	indene	6.45
5	naphthalene	60.80
6	quinoline	1.15
7	isoquinoline	0.29
8	C ₁ -naphthalene	1.25
9	C ₁ -naphthalene	0.32
10	biphenyl	0.03
11	acenaphthylene	14.98
12	acenaphthene	1.66
13	C ₁ -biphenyl/acenaphthene	1.79
14	fluorene	2.64
15	phenanthrene/anthracene	3.82

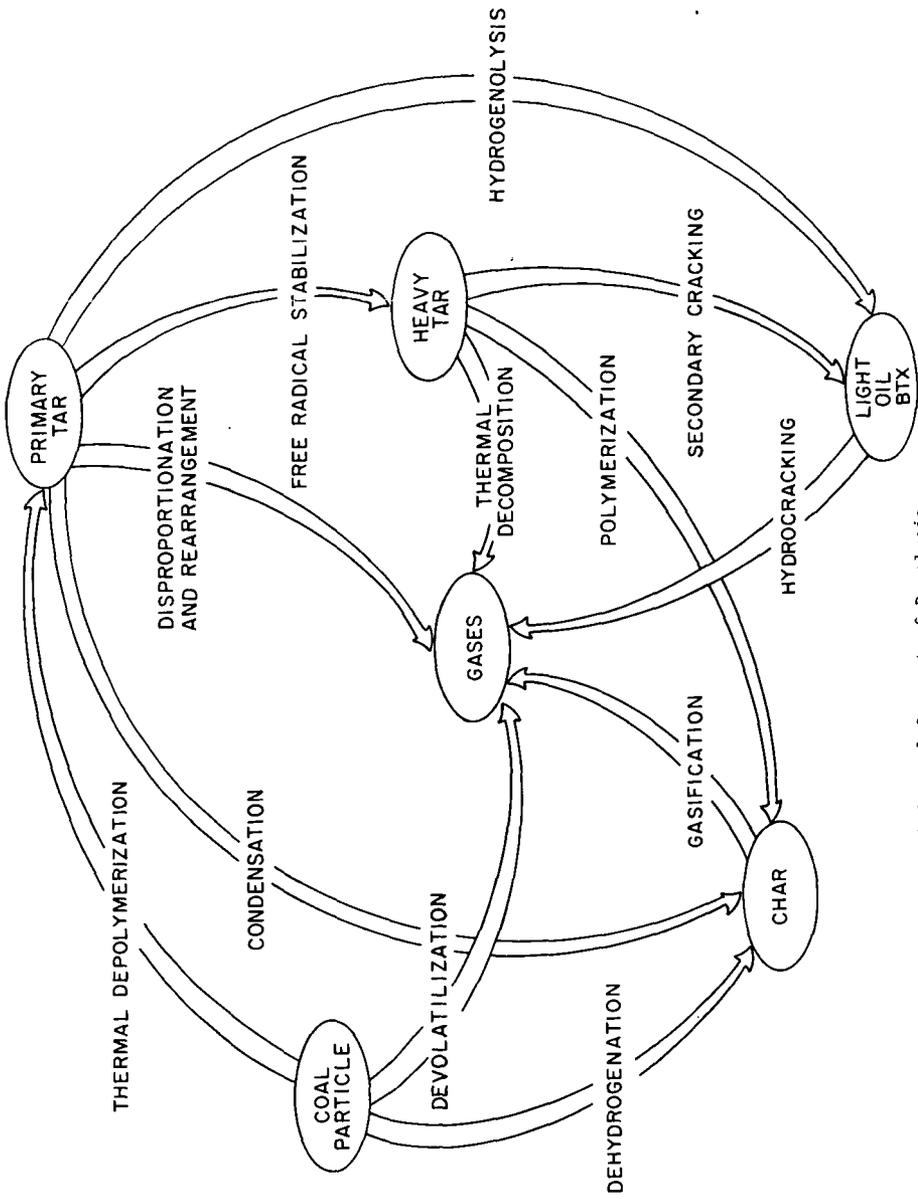


Figure 1. A General Concept of Pyrolysis

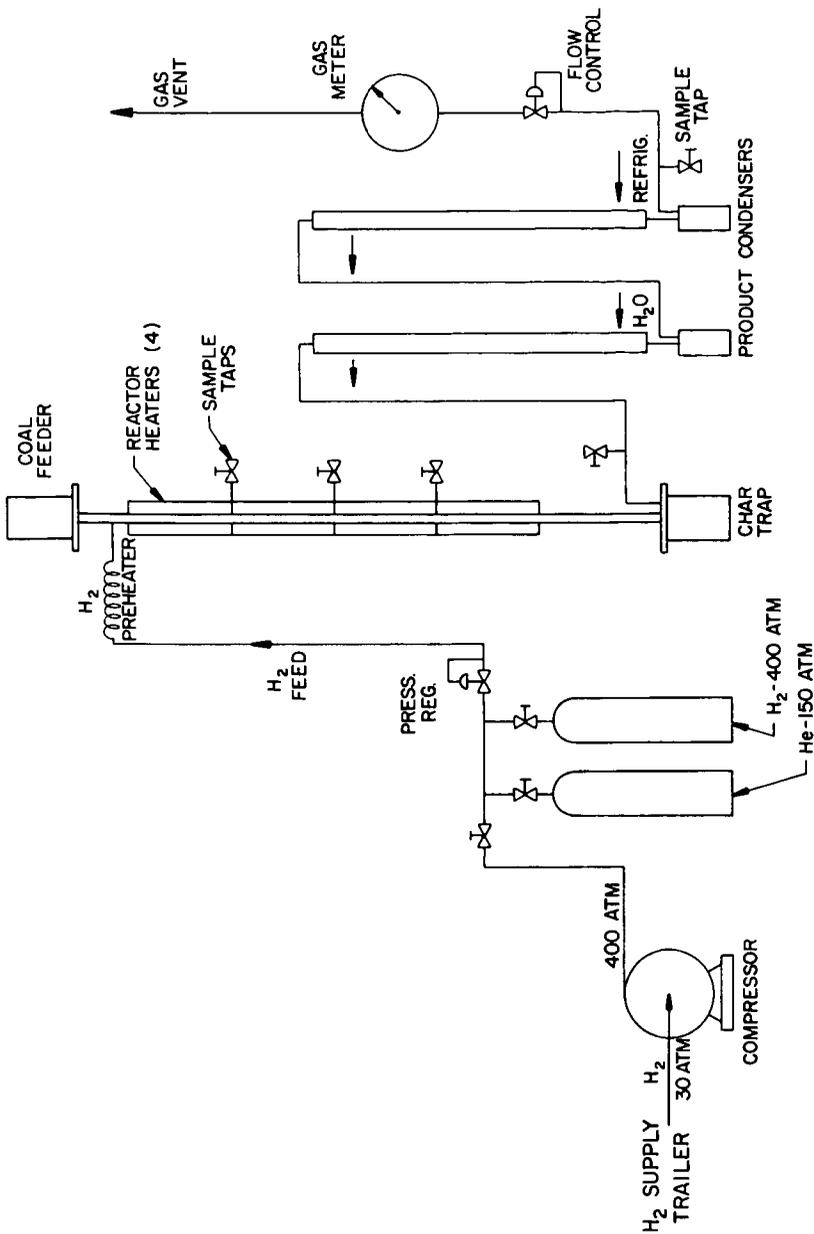


Figure 2. SCHEMATIC FLOWSHEET OF ENTRAINED TUBULAR REACTOR EXPERIMENT

COAL PARTICLE TEMPERATURE vs. TIME
PARTICLE SIZE - 100 μ
INITIAL TEMPERATURE - 20°C
GAS TEMPERATURE - 900°C

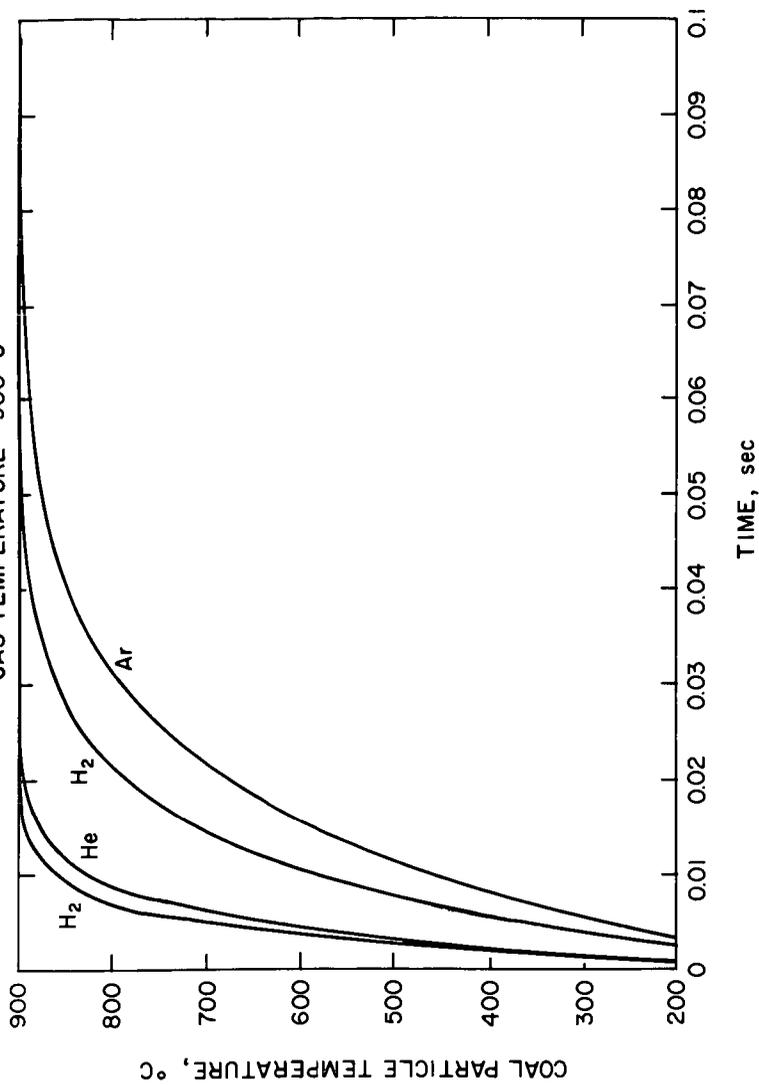


Figure 3. Coal Particle Temperature vs Time

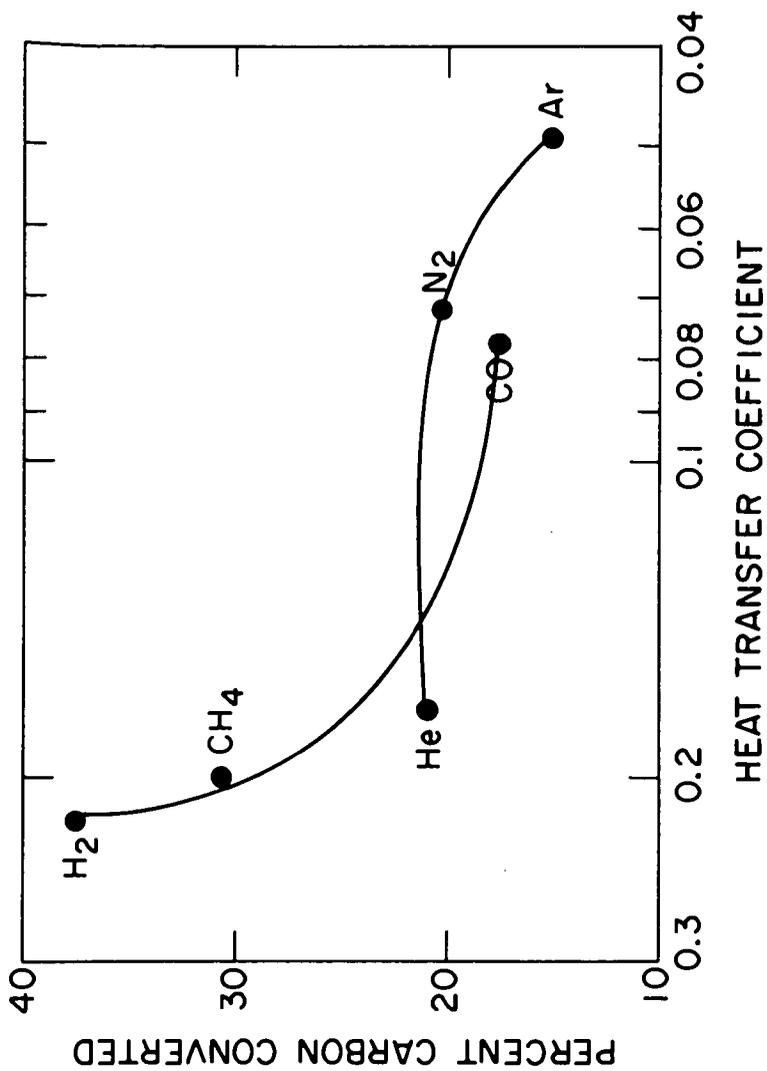


Figure 4. Percent Carbon Conversion at 900°C and 200 psi vs Heat Transfer Coefficient

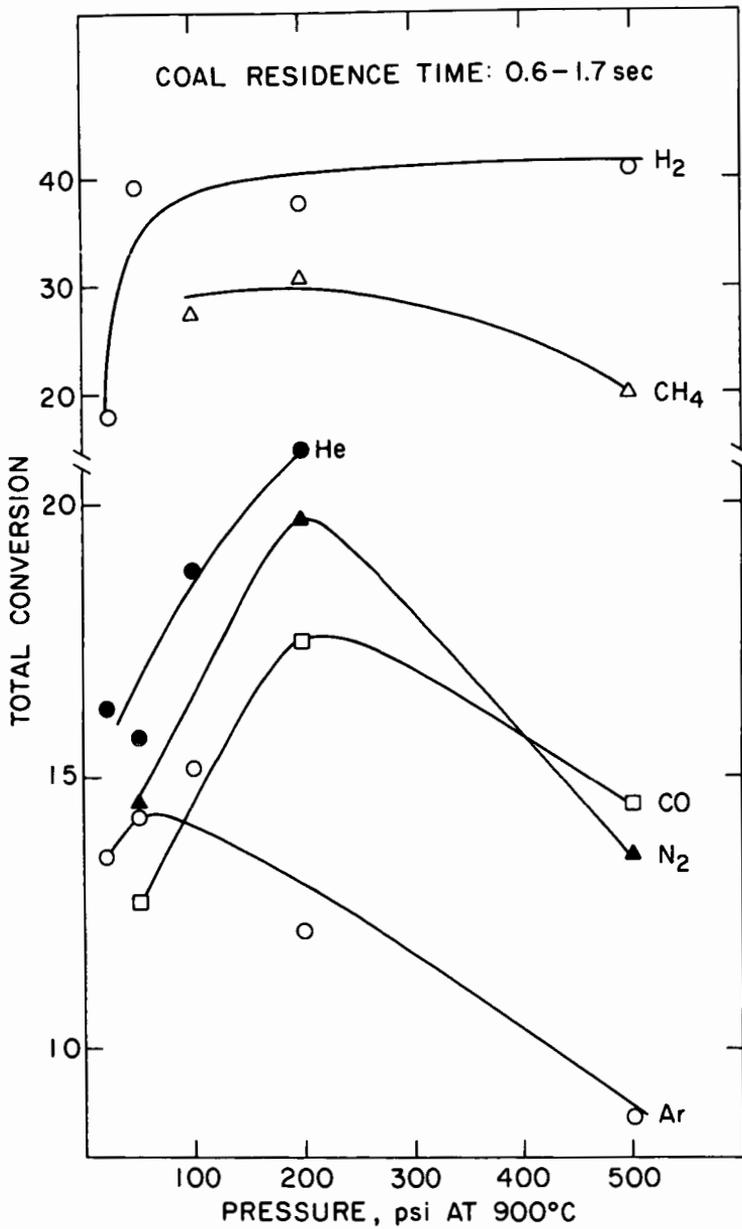


Figure 5. Total Conversion vs Pressure, at 900°C

FLASH PYROLYSIS OF NEW MEXICO SUBBITUMINOUS COAL WITH HELIUM
 PERCENT CARBON CONVERTED vs. TEMPERATURE
 PRESSURE - 20 psi
 RES. TIME - 0.5 to 0.6 sec.

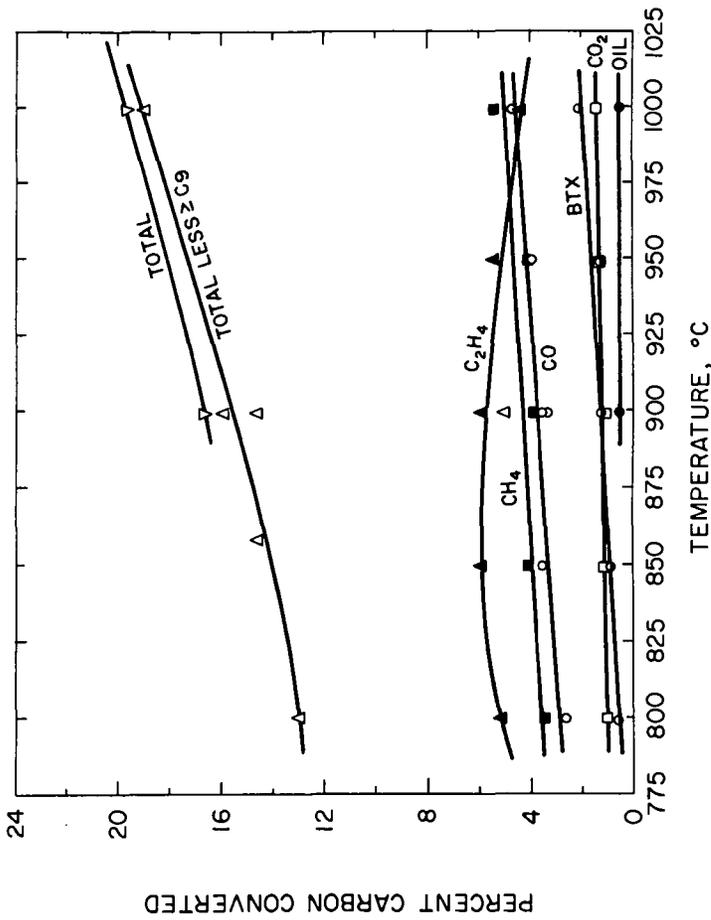


Figure 6. Product Yields vs Temperature at 20 psi He

ETHYLENE PRODUCTION BY FLASH PYROLYSIS OF
 NEW MEXICO SUBBITUMINOUS COAL
 TEMPERATURE - 800 - 1000°C

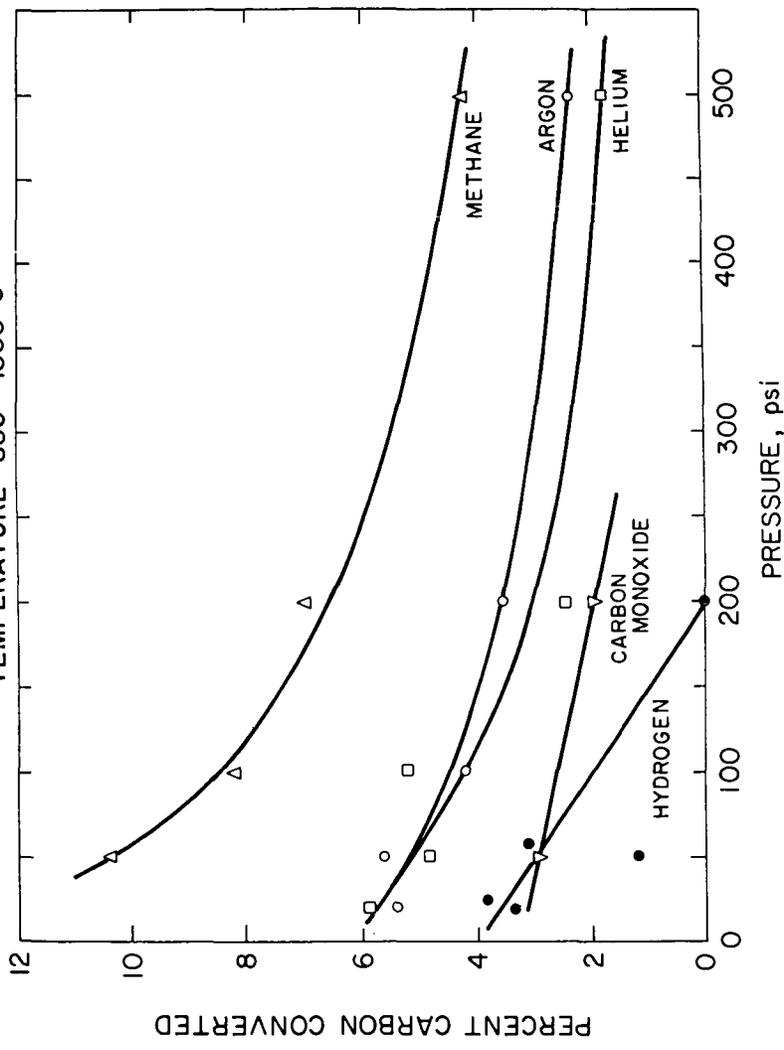


Figure 7. Ethylene Yield vs Pressure at 800° to 1000°C

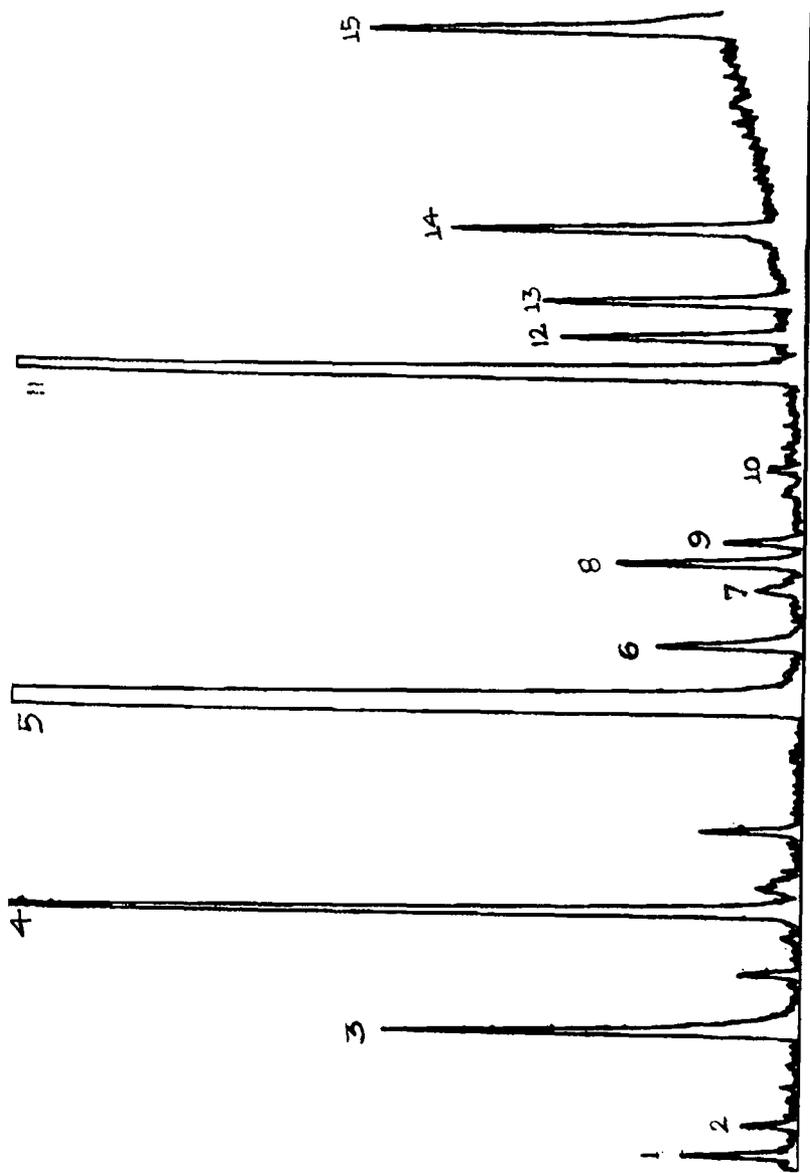


Figure 8. GC/MS Total Ion Chromatogram of a Pyrolysis Oil