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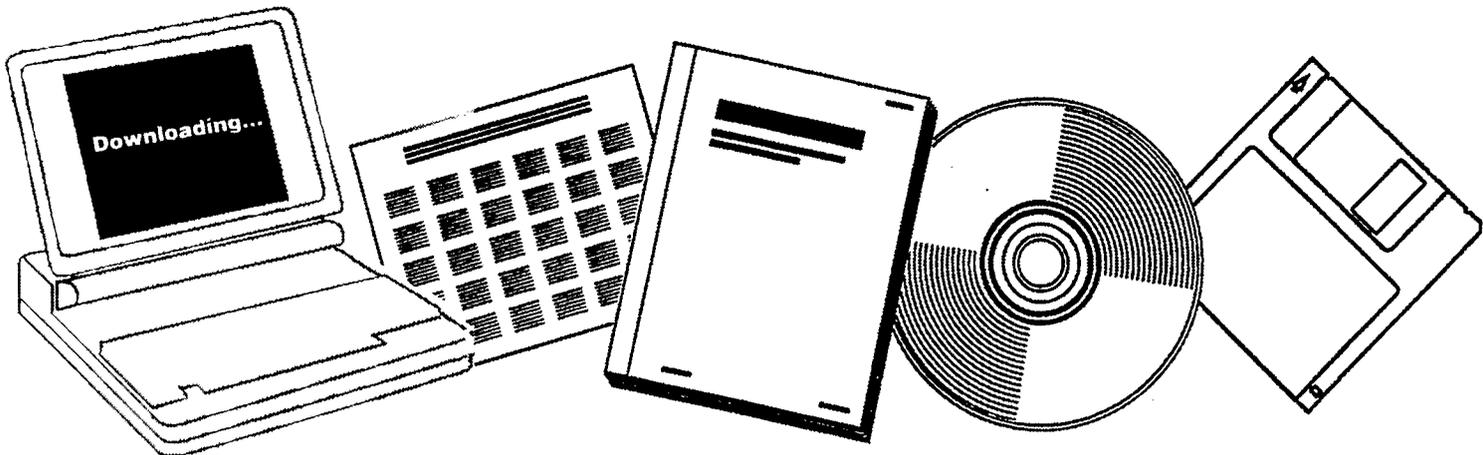
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**COAL LIQUEFACTION. INVESTIGATION OF
REACTOR PERFORMANCE, ROLE OF CATALYSTS AND
PCT PROPERTIES. QUARTERLY PROGRESS REPORT,
APRIL 1-JUNE 30, 1986**

PITTSBURGH UNIV., PA

1986



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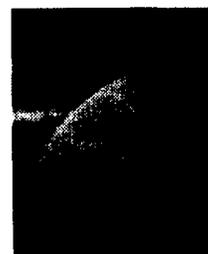
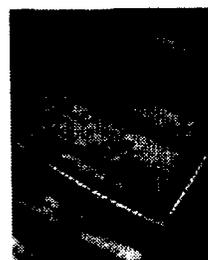
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DOE/PC/60054--T11

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Quarterly Progress Report

COAL LIQUEFACTION - INVESTIGATION OF REACTOR PERFORMANCE,
ROLE OF CATALYSTS AND PCT PROPERTIES

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Prepared for the Department of Energy
Contract No. DE-FG22-83PC60054

April 1, 1986 to June 30, 1986

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1. OBJECTIVE AND SCOPE OF WORK

The objective of this work is to investigate areas of science and technology that have been defined as being of prime interest to coal processing technology development. These areas include properties of coal liquids and slurries, reactor design and performance in relation to reaction mechanisms. The work comprises investigations of the following high priority areas:

- Reaction kinetics and reactor performance in coal liquefaction;
- Role of catalysts in indirect liquefaction and direct coal liquefaction;
- Physical, chemical and thermodynamic properties of coal liquefaction products.

Work done during the first year on this project is summarized in Technical Progress Report DOE/PC/60054-T4 dated October 1984 and work during the second year in a Technical Progress Report submitted to the Department of Energy dated November 1985.

This report summarized work done during the period April 1, 1986 through June 30, 1986. A detailed description of work follows.

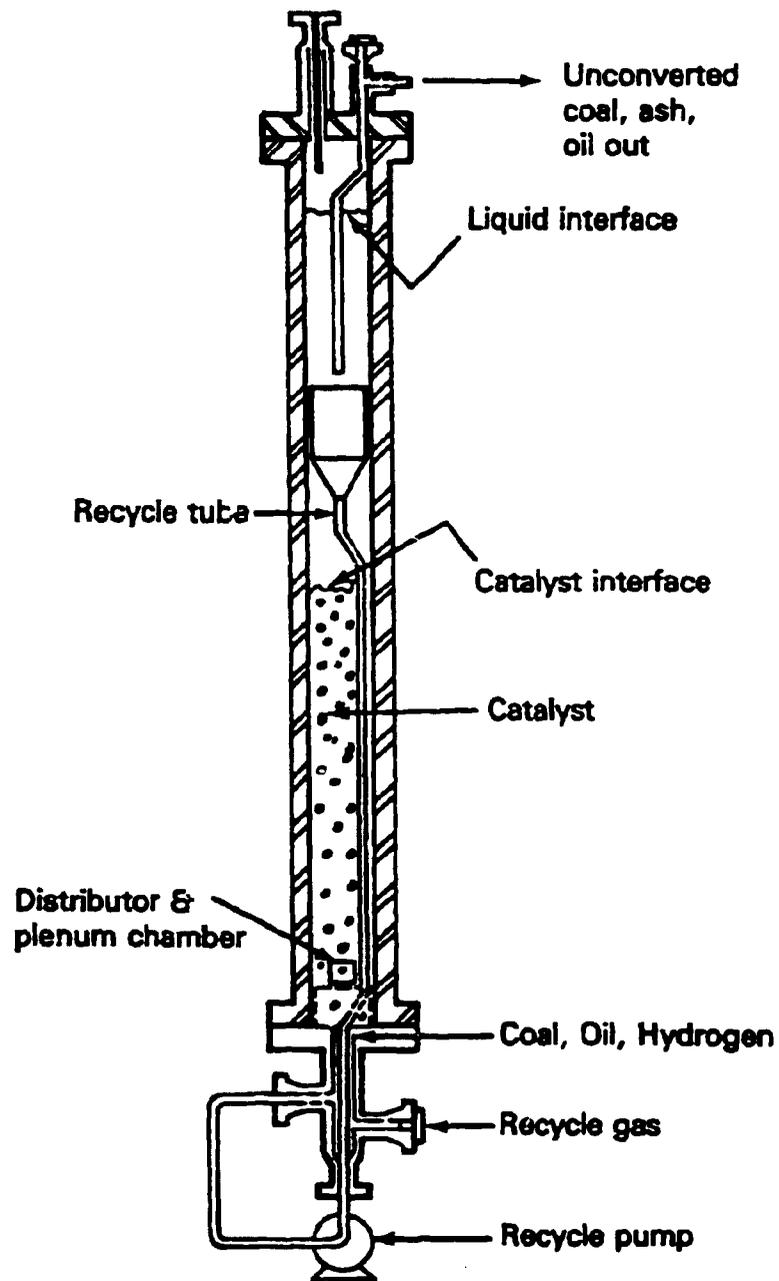
2. MODELING OF COAL LIQUEFACTION PROCESSES

In the last quarter, major emphasis was placed on obtaining hydrodynamic, mass transfer and mixing parameters for the description of the ebullated bed reactor. The ebullated bed reactor, which was developed and patented by Hydrocarbon Research Inc. is the heart of the second stage of the Wilsonville Integrated Two Stage Liquefaction (ITSL) Process. A schematic of the ebullated bed reactor is shown in Figure 1. The catalyst particles are maintained in the form of an expanded bed by the continuous flow of slurry and hydrogen through the reactor. The liquid phase forms a continuous medium and the hydrogen is dispersed in the form of bubbles. A part of the slurry is recycled in order to obtain the necessary liquid velocity to fluidize the bed. The major advantages in using ebullated beds in place of fixed beds are: on stream catalyst replacement, fouling-free operation and a small reactor temperature gradient.

Modeling of the ebullated bed requires a detailed understanding of the fluid dynamics of the complex reacting system. Factors affecting the performance of the reactor and information needed for the design and modeling include the following:

- A) Catalyst bed expansion
- B) Phase holdups of gas, slurry and catalyst
- C) Mixing parameters, axial dispersion of gas, slurry and catalyst phases.
- D) Transport parameters: gas-liquid and liquid-solid mass transfer coefficients and gas-liquid interfacial area.
- E) Reaction data: kinetics, conversion and product yields.
- F) Thermodynamic parameters: vapor-liquid thermodynamic data, specific heats, thermal conductivities.

Figure 1
H-Coal PDU Reactor



Liquid Phase Dispersion

Liquid phase dispersion measurements were carried out by Schaefer et al. (1983) and they have proposed the following correlation:

$$Pe_L = 5.05 + 42.91 \exp(-35.48 U_G) \quad U_G \text{ in ft/sec}$$

$$\text{where } Pe_L = \frac{U_L L}{D_L}$$

Gas-Liquid Mass Transfer Coefficient

Muroyama and Fan (1985) do not recommend any specific correlation and note that literature correlations for $k_L a$ lack generality and can be employed only for very limited conditions. Lee et al. (1978) have shown that for the SRC dissolver, mass transfer of hydrogen was not the rate-controlling step, rather, the SRC reactor was found to operate in a kinetically limited regime. In the case of catalytic liquefaction, however, Feldman et al. (1972) concluded from their studies on the hydrogenation of coal tar and a coal-coal tar slurry using a Co-Mo catalyst, that the rate of hydrogenation was limited by the diffusion of hydrogen from the gas phase to the liquid phase rather than by inter or intraphase diffusion involving the catalyst. Thus a knowledge of gas-liquid mass transfer coefficient is needed for an accurate model of the ebullated bed. In the absence of directly applicable correlations, the correlations developed by Dhanuka and Stepanek (1980) for the system $CO_2/N_2/Na_2CO_3-NaHCO_3$ buffer will be used. A sensitivity analysis with respect to $k_L a$ will also be performed.

Phase Holdups:

Models to predict phase holdups in three phase fluidized bed reactors have been reviewed by Muroyama and Fan (1985). They recommend the generalized wake model developed by Bhatia and Epstein (1974) to be used in describing the individual phase holdups. The generalized wake model, which takes into account the volume fraction of the wake and the solids holdup in the wake, was used by Vasaios et al. (1980) and Schaefer et al. (1983) to describe holdup data both for the cold-model runs and the PDU runs. The Bhatia-Epstein model was selected by these workers due to the following advantages over other models:

- i) It is able to predict both catalyst bed expansions and bed contractions when gas is introduced into a liquid-fluidized bed.
- ii) It is able to describe operation both in the ideal bubbly and churn turbulent regimes.
- iii) Its parameters (bubble rise velocity, wake structure and wake composition) are amenable to a physical interpretation and can be checked by such experimental approaches as tracer methods.

The Bhatia-Epstein model envisages the fluidized bed to comprise of three component phases: gas bubbles, wakes behind the bubbles and particulate (catalyst plus liquid slurry) phases. The wake behind the bubbles is assumed to entrain both slurry and catalyst. The amount of catalyst carried by the wake is described by the parameter X_k . If $X_k = 1$, the ratio of solid to liquid content in the wake is the same as that existing in the particulate phase. Darton and Harrison (1975) used a similar type of wake model with $X_k=0$ in conjunction with Wallis' drift flux approach to correlate gas and liquid holdups.

Liquid-Solid Mass Transfer Coefficient

Lee et al. (1974) neglected liquid-solid mass transfer in modeling a three phase fluidized bed. Ermakova et al. (1977) have given a correlation for the liquid-solid mass transfer coefficient for a liquid fluidized bed.

Model Development

The following assumptions are made in the development of the model for ebullated bed reactor:

- 1) The ebullated bed operates at steady-state conditions. Nalitham et al. (1984) have shown that the relatively slow deactivation kinetics makes it feasible to maintain essentially steady performance in a discrete addition-withdrawal mode.
- 2) The gas phase moves in plug flow (Muroyama and Fan, 1985).
- 3) The slurry phase can be modeled using a one-dimensional axial dispersion model.
- 4) The reactor operates in an isothermal mode. This is a reasonable assumption in view of the observation of Li and Lin (1981) of not more than a 8⁰C rise in axial reactor temperature. Van Driesen and Stewart (1964) also reported a virtually uniform temperature distribution throughout the H-Oil fluidized-bed reactor even when the heat of reaction was sufficient to raise the temperature of the entire feed by 56⁰C. The uniform temperature distribution within the H-Oil or H-Coal reactor is due to the relatively high degree of internal backmixing and the large recycle flow of the liquid.

Based on the above assumptions, the following mass balance equations can be written:

Gas Phase:

$$-\frac{dy_i}{dz} = \frac{St}{m_i} (y_i - m_i c_i)$$

Liquid Phase:

$$\frac{1}{Pe_L} \frac{d^2 c_i}{dz^2} - \frac{dc_i}{dz} + \frac{St}{m_i} \cdot \frac{1}{U_r} (y_i - m_i c_i) - Da \cdot C_i = 0$$

The solid-liquid mass transfer has been neglected in the balance on liquid phase components.

$$\text{Here } y_i = \frac{C_{G,i}}{C_{G,in}} \quad c_i = \frac{C_{L,i}}{C_{G,in}} \quad z = \frac{x}{L}$$

$$Pe_L = \frac{LU_L}{D_L \epsilon_L} \quad U_r = \frac{U_L}{U_G} \quad St = \frac{L k_L a}{U_G} \quad Da = \frac{L k_i G_L}{U_L}$$

The above set of differential equations constitute a boundary value problem. The boundary conditions for the liquid phase are not straightforward as can be seen from examining Figure 1. The liquid phase entering the reactor consists of fresh feed and a recycle slurry. Thus, a trial and error procedure has to be resorted to in order to obtain the correct boundary conditions. The software package, COLSYS, which we have previously used, will be used for the solution of the boundary value problem.

Notation

$C_{G,i}$	gas phase concentration of component i , kmol/m^3
$C_{G,in}$	inlet gas phase concentration, kmol/m^3
C_i	dimensionless concentration of component i in liquid phase
$C_{L,i}$	liquid phase concentration of component i , kmol/m^3
Da	Damkohler number
D_L	liquid phase dispersion coefficient, m^2/s
k_i	reaction rate constant (first order, $1/\text{s}$)
$k_L a$	gas-liquid mass transfer coefficient, $1/\text{s}$
L	effective reactor height, m
m_i	solubility coefficient for component i
Pe_L	liquid phase Peclet number
St	Stanton number
U_G	superficial gas phase velocity, m/s
U_L	superficial liquid phase velocity, m/s
U_z	linear liquid phase velocity, m/s
x	distance along reactor, m
y_i	dimensionless gas phase concentration of component i
z	dimensionless reactor distance

Greek

ε_L	liquid phase holdup
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3. ROLE OF CATALYSTS IN INDIRECT LIQUEFACTION AND DIRECT COAL LIQUEFACTION

The objective of this work is to review and discuss key advances in direct coal liquefaction catalysis, especially for the innovative use of certain catalysts in direct coal liquefaction. This study will provide an overview of streams of knowledge which have contributed to the development of coal liquefaction catalysis and which may have potential for contributing in the future. Newer types of catalysts, discovered after World War II, will constitute the main subjects of this report.

Scope of Work

The use of catalysts in direct coal liquefaction provides several advantages including activating molecular hydrogen, minimizing hydrogen consumption, permitting milder operating conditions, and increasing distillate production. Possibly included among the catalyst systems to be reviewed and whose potentials are to be evaluated are: the use of liquid clathrate systems, homogeneous catalysts such as metal carbonyls and transition metal complexes, iodine and hydrogen iodide, and superacids such as fluorosulfonic acid (FSO_2OH) and the less corrosive solid acids such as $\text{Fe}_2\text{O}_3\text{-SO}_4^{2-}$ and $\text{TiO}_2\text{-SO}_4^{2-}$.

Coal Liquefaction Using Transition Metal Carbonyl Complexes as Catalyst Precursors

A review of coal liquefaction with transition metal carbonyls has shown that there are two ways in which these complexes have been used as catalysts or catalyst precursors. As catalytic agents, transition metal carbonyl complexes are usually effective at temperatures below 250°C to hydrogenate

coal constituents or coal-derived liquid products, depending on the carbon monoxide pressure needed to prevent the complexes from decomposing to reduced metals. In Table 1, a number of polynuclear aromatic hydrocarbons are compared under synthesis gas ($H_2:CO=1:1$) conditions using $Co_2(CO)_8$ as the catalyst. The data indicates that linear aromatic compounds such as anthracene and naphthalene are more reactive and more easily hydrogenated to form products with isolated benzene rings than non-linear aromatic compounds such as phenanthrene and chrysene.

The second major method of using transition metal carbonyl complexes is as a catalyst precursor. In this case, at the usual temperatures of direct coal liquefaction, the complex decomposes to finely divided, well-dispersed zerovalent metal which, in many cases, proves to be an effective catalyst for coal hydroliquefaction. The carbonyl compounds of molybdenum, tungsten, iron, cobalt, and nickel that provide highly active catalyst precursors for the liquefaction of Illinois No. 6 coal are shown in Table 2. High coal conversions and high oil yields were obtained using transition metal carbonyl complexes as catalyst precursors. In particular the decomposition products from $Mo(CO)_6$ gave a coal conversion of 92.1 wt% and an oil yield of 57.7% compared to 56.7 and 22.1%, respectively, without using a catalyst.

Table 1

Hydrogenation of Polynuclear Aromatic Hydrocarbons
Using Dicobalt Octacarbonyl^a

Compound	Structure	[Sub]/[Cat.]	Temp. (°C)	(CO/H ₂) Pressure (psi)	Hydrogenated Yield (%)
Naphthalene		26.7	200	3100	16
2-Methylnaphthalene		25.8	200	3500	43
1, 1-Dinaphthyl		0.7	200	3500	ND ^c
Acenaphthene		4.4 8.7 ^b	200 185	3500 2800	45 82
Fluorene		4.1	200	3600	ND ^c
Anthracene		95.8 7.5 ^b	135 185	2900 2800	99 93
Phenanthrene		4.6 7.5 ^b	200 185	3500 2800	8 31
Naphthacene		20.5	140	3000	70
Chrysene		0.4 0.7	150 200	3500 3000	ND ^c 24
Fluoranthene		1.2	200	3200	54
Triphenylene		0.2	200	3200	ND ^c
Pyrene		0.9	150	3600	ND ^c
Perylene		1.1 0.3	200 150	3000 3000	69 72
Coronene		0.2	200	2800	ND ^c

a. Reference from Friedman et al. (1959).

b. Reference from ARCO SEACOKE Project (1966).

c. Not detectable.

Table 2
 Hydroliquefaction of Illinois No. 6 Coal
 Using Various Transition Metal Carbonyl Complexes as Catalyst Precursors^a
 (Yamada et al., 1985)

Catalyst ^b	Conversion (%)	Oil (%)	Distribution of Products		H ₂ added to Coal (wt% of coal)
			Asphaltenes (%)	Preasphaltenes (%)	
None	56.7	22.1	19.2	11.0	0.5
MnCpMe(CO) ₃ ^c	75.8	20.0	27.7	22.4	1.3
Cr(CO) ₆	77.3	25.9	25.3	19.8	1.4
W(CO) ₆	91.6	33.0	33.0	19.4	2.2
Mo(CO) ₆	92.1	57.7	26.0	3.8	3.1
Fe(CO) ₅	92.2	44.0	30.4	17.8	2.2
[FeCp(CO) ₂] ₂ ^d	92.9	32.6	39.0	14.5	2.5
Co ₂ (CO) ₈	93.3	37.9	32.8	16.6	2.3
Ni(CO) ₄	94.1	32.4	35.3	21.1	2.2

a. Experimental Conditions: 2 gm Illinois No. 6 coal, 4.0 ml 1-methylnaphthalene, initial H₂ pressure 710 psi, 425°C and 60 min.

b. 1 wt% of coal as metal.

c. Methylcyclopentadienylmanganese tricarbonyl.

d. Cyclopentadienyliron dicarbonyl dimer.

Coal Liquefaction with Liquid Clathrates

Coal liquefaction with liquid clathrates occurs at or near room temperature and requires neither application of heat nor consumption of hydrogen. The unique characteristics of coal dissolution with liquid clathrates lies in that the clathrate acts as a host and does not itself react with the coal. A comparison of coal solution in different liquid clathrates for various ranks of coals is summarized in Table 3. The weight percent of coal dissolved in liquid clathrates depends mainly on temperature, contact time, and coal rank. In general, higher extraction temperature and longer contact time will dissolve more coal in the liquid clathrate.

Another unique property of the liquid clathrates is that they dissolve substances which act as homogeneous catalysts. A significant increase of coal dissolution, up to 40%, was found by incorporation of a catalyst such as $(C_6H_6)Cr(CO)_3$ or $(C_5H_5)Mn(CO)_5$ in the liquid clathrate $[NEt_4][Al_2Me_6I] \cdot 6.4$ toluene with hydrogen gas bubbling through the phase. It is believed that the mechanism of catalytic hydrogenation of coal dissolution in liquid clathrates involves coal reactive fragments, derived immediately after dissolution, being hydrogenated with an immobilized homogeneous catalyst. Meanwhile, saturated hydrocarbons are preferentially transferred from the clathrate into the solvent; then, subsequent equilibrium attainment would extract a further amount of aromatic type of material from the coal into the clathrate, resulting in higher yields and better product qualities.

Table 3
Coal Solvation in Liquid Clathrates
(Atwood, 1982)

Run	1	2	3	4	5	6	7
Sample ^a	Bituminous, Mary Lee Seam	Bituminous, Mary Lee Seam	Bituminous, Mary Lee Seam	Subbituminous, Walker Co.	Lignite, East Central	Tar Sands	Bituminous, Mary Lee Seam
Liquid Clathrate							
Parent Compounds	[N(C ₃ H ₇) ₄] [Al ₂ Me ₆ I]	[N(C ₃ H ₇) ₄] [Al ₂ Me ₆ I]	K[Al ₂ Me ₆ N ₃]	[N(C ₃ H ₇) ₄] [Al ₂ Me ₆ I]	[N(C ₂ H ₅) ₄] [Al ₂ Et ₆ I]	[N(C ₂ H ₅) ₄] [Al ₂ Me ₆ NU ₃]	[K(18-Crown-6)] [Al ₂ Me ₆ N ₃]
Guest solvent	Toluene	Toluene	Toluene	Toluene	Benzene	Toluene	Toluene
Coal/Liquid Clathrate (gm/ml)	10/100	10/100	10/100	10/100	10/100	10/100	10/100
Temperature, °C	80	Room Temp.	60	60	Room Temp.	Room Temp.	Room Temp.
Contact Time, hr.	12	48	96	6	4	1	1
Conversion (% Based on Coal Feed)							
Coal Dissolved, %	19	12	22	--	--	--	--
Toluene Soluble, %	4	3	5	6	10	18	3
Toluene Insoluble, %	9	5	9	--	--	--	--

^a = All samples are from Alabama.

Several limitations or restrictions associated with coal dissolution in liquid clathrates must be overcome before a continuous phase or a large-scale operation can be considered. Some of these are listed below:

- (1) The overall yield of coal dissolution in liquid clathrates is about 20 to 40%. This seems close to, but not better than, the limit of coal extracted or dissolved in most good coal solvents. The amount of coal dissolved seems to be controlled by the breakage of hydrogen bonds or of cleavage of oxygen-containing groups in the coal structure.
- (2) The aluminum alkyls used in the liquid clathrates are thermally unstable at high temperatures. Beyond 190°C, the aluminum alkyl will decompose. In addition, the clathrates discussed here are sensitive to moisture and oxygen. Moisture or oxygen will attack the aluminum alkyl component of the salt, although the reactivity of the parent compounds in the liquid clathrate is, in general, much less than that of the aluminum alkyls themselves.
- (3) The particle size of coal will affect the period of contact time needed with the liquid clathrate. Agitation or stirring must be applied during the contact time to reduce diffusion limitations. Fine coal particles with large surface areas enable maximum contact with the liquid clathrate.
- (4) The liquid clathrate phase, by continued operation, could accumulate materials which cannot be regenerated due to the various ionic components on the coal surface. The liquid clathrate will then gradually lose its solvating power. The primary requirements for the components in the liquid clathrate are low cost, easy regeneration, and hydrolytic stability.

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