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**Coal Dissolution Studies: Particle Size
Distribution in Coal-Derived Liquids as a
Function of Degree of Coal Conversion**

T. M. Gilliam
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COAL DISSOLUTION STUDIES: PARTICLE SIZE DISTRIBUTION
IN COAL-DERIVED LIQUIDS AS A FUNCTION
OF DEGREE OF COAL CONVERSION

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HIGHLIGHTS

Exploratory studies were conducted to assess the effect of the degree of coal conversion (i.e., a decrease in the amount of pyridine insolubles) in reacted coal--anthracene oil mixtures. Particle size distribution data on the reacted mixtures show that the percentage of particulates $<10\ \mu$ in diameter increases gradually with conversion up to a conversion of about 85%. At higher conversions, the percentage of particulates $<10\ \mu$ in diameter increases dramatically in a near-step, function-like manner.

The presence of small particulates are known to be a major cause of the low-filtration rates of coal liquefaction streams. Filtration experiments on the mixtures confirm that a relationship exists between filtration rates and conversion. Thus, the results of this work bear directly on the optimal design of two-stage liquefaction processes and could lead to improved design of processes such as SRC-I.

1. INTRODUCTION

Hydrogen required for coal liquefaction, and process heat required for liquefaction processes may be derived via gasification from the carbonaceous residues and unconverted coal present in the product of the primary liquefaction step. Typically, hydrogen and process heat requirements may demand gasification of material equivalent to 20 to 40% of the heating value of the coal fed to the process. Most primary liquefaction processes strive for a degree of coal conversion ranging from 90 to 95% of the heating value of the coal feed. On the other hand, processes based on a lower level of conversion in the primary liquefaction

step could meet the hydrogen and process heat requirements if the degree of conversion in the primary liquefaction step was as low as 60 to 80%. This program of preliminary exploratory experiments was aimed at assessing the possible merits of a process in which the degree of conversion in the primary liquefaction step was substantially lower (70 to 90%) than that achieved in most conventional processes (90 to 95%).

It has been proposed that the small particulates in coal-derived liquids, a major cause of low-filtration rates, occur significantly only at a high degree of coal conversion.¹ This hypothesis is based on the "Swiss-cheese" model of coal dissolution, shown in Fig. 1. As the dissolution of the coal particles progresses, the particles become pitted, resulting in the lattice-like structure of the partially reacted coal particles. The integrity of this structure is maintained until the conversion approaches a threshold, at which point the structure collapses and produces the large number of small particles. Thus, one benefit of a lower conversion could be an easier solid-liquid separation step.

To verify this hypothesis, the experiments were performed to determine if a relationship exists between particle size of the solids in coal-derived liquids and the degree of coal conversion. Because filtration rates are a strong function of particulate size, such a size distribution-dissolution relationship would indicate that the percent of coal dissolution is a variable that might be used to increase filtration rates.

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Fig. 1. Diagram of the Swiss-cheese model of coal conversion.

2. GENERAL EXPERIMENTAL PROCEDURE

A simplified schematic diagram of the coal conversion apparatus is shown in Fig. 2; the equipment is discussed in detail in Appendix A. The equipment consists of a rocking autoclave, a stirred autoclave, a filter feed chamber, a filter, a filtrate collector, heaters, and the necessary peripherals to control and monitor the system at the desired temperature and pressure.

The procedure involved heating three parts by weight of Allied 24CB coal tar distillate (anthracene oil) and one part of Illinois No. 6 coal in a rocking autoclave for a fixed time at the desired temperature (nominally, 750°F). To facilitate mixing, ten 0.25-in.-diam stainless steel mixing balls were added to the solution. The heatup rate was 5.6°F/min. Prior to heatup, the system was purged with argon to remove trapped air so that at the start of each experiment the overhead pressure of the solution (i.e., the vapor pressure of the oil and the argon) was 1 atm. After the solution reacted in the rocking autoclave, the resulting slurry was cooled to room temperature at a rate of 4.5°F/min and allowed to stand overnight. The slurry was then characterized by particle size distribution, percent conversion, and ash and sulfur content.

After characterization, the slurry was placed in a stirred autoclave that acted as a preheater for the filter. (It should be noted that the stirred autoclave was not used as the coal dissolution reaction vessel because of pressure limitations in that vessel's design.) The stirred autoclave, the filter, and the filter feed chamber were then heated to the preselected filtration temperature (350°F). An argon gas cylinder

Fig. 2. Simplified schematic of experimental apparatus.

was attached to the autoclave and the cylinder discharge pressure was regulated (at the manifold) to ensure a constant pressure in the equipment upstream of the filter. The pressure downstream of the filter is 1 atm, and the argon addition thus ensured a constant pressure drop of 35 psi across the filter. After the system reached the desired filtration temperature, the slurry was routed to the filter feed chamber by manually opening a 1-in. ball valve located at the base of the autoclave. The slurry was then filtered through two pieces of No. 42 Whatman ashless filter paper supported on a 200-mesh stainless steel screen. The 4-in.-diam filter has an effective surface area of 0.0873 ft^2 . The filtrate was collected in a beaker while the cumulative filtrate weight was continuously monitored by a precision digital balance with analog output, which simultaneously drove a strip chart recorder. The resulting cumulative filtrate weight curves (i.e., weight of the filtrate collected as a function of time) and the weight of the filter cake collected were used to determine the overall effective filtration rate as a function of time.

3. DATA ANALYSIS AND DISCUSSION

3.1 Coal Conversion Studies

The anthracene oil used in these experiments was supplied by the Solvent Refined Coal pilot plant at Wilsonville, Alabama, which is operated by Catalytic, Inc. The oil was characterized and the results are shown in Fig. 3 and Table 1. The coal was prepared by grinding it in a micropulverizer. The coal was characterized by composition and particle size, and the results shown in Tables 2 and 3.

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Fig. 3. Distillation curve for anthracene oil.

Table 1. Principal analysis of anthracene oil
used in the experiments

Component	Weight (%)
Moisture	<0.01
Ash	0.1
Pyridine insolubles	0.46
C	86.65
H	9.16
N	0.84
S	0.57

Table 2. Principal analysis of Illinois No. 6 coal
used in the experiments

Component	Weight (%)
Moisture	2.48
Ash	11.1
Pyridine insolubles	77.0
C	78.33
H	4.93
N	1.10
S	4.55
O ^a	11.09

^aBy difference.

Table 3. Particle size analysis of Illinois No. 6 coal
used in the experiments

Sieve No.	Sieve opening (μ)	Total mass on each sieve (%)
140	105	0
170	88	36.0
200	74	15.3
230	63	17.6
270	53	11.0
325	44	15.9
400	37	4.1

Data collected during successful coal conversion experiments are shown in Tables 4 and 5. As shown in Table 4, there is a significant weight loss between the coal and oil introduced into the autoclave and the slurry that was removed after the reaction. Subsequent tests have shown that less than a 4% weight loss occurs due to material lost in transferring the solution from one vessel to another. The majority of the weight loss is caused by volatile reaction products that were lost when the cooled, rocking autoclave was vented before it was opened.

A partial chemical analysis of the slurries is shown in Table 5. The percent of pyridine insolubles, assumed to be the solids content, was determined by extracting the slurry with pyridine in a Soxhlet extraction system using a 43- by 123-mm Whatman cellulose double-thickness extraction thimble.

The degree of conversion was calculated on a moisture- and ash-free (maf) basis according to the following:

$$\% \text{ CON} = 100 \frac{[M_c (W_{CI} - W_{CA}) - M_s (W_{SI} - W_{SA})]}{M_c (W_{CI} - W_{CA})} \quad (1)$$

where

CON = conversion,

M_c = mass of coal added, g,

W_{CI} = weight fraction of pyridine insolubles in coal,

W_{CA} = weight fraction of ash in coal,

M_s = mass of slurry produced, g,

W_{SI} = weight fraction of pyridine insolubles in slurry, and

W_{SA} = weight fraction of ash in slurry.

Table 4. Rocking autoclave coal conversion data

Experiment No. ^a	Weight of coal added (g)	Weight of anthracene added (g)	Ratio of oil to coal	Reaction temperature (°F)	Reaction time (min)	Slurry removed from autoclave (g)	Weight loss (%)
89-104	166.3	498.5	3:1	750	10	522.3	21
91-101	166.2	498.9	3:1	750	60	559.4	16
95-97	168.8	506.5	3:1	730	60	327.9	51
103-106	229.5	688.4	3:1	390	90	848.2	8
108-110	271.1	813.2	3:1	750	20	759.6	30
119-122	167.2	501.5	3:1	750	50	518.0	23
126-124	179.3	540.2	3:1	750	85	649.3	10
130-132	197.0	589.4	3:1	750	110	608.5	23
134-136	199.2	596.1	3:1	750	135	633.9	20
138-139	195.7	587.9	3:1	750	195	668.8	15
141-142	197.8	592.2	3:1	750	25	734.8	7
144-150	178.1	534.8	3:1	750	280	563.2	21
145	240.6	721.8	3:1	750	5	865.2	10
147	242.6	727.7	3:1	750	70	719.6	26

^aExperiment numbers were determined by the pages in the log book in which the information was recorded for the coal conversion and subsequent filtration. Thus, for experiment 89-104, the coal conversion data are recorded on p. 89, and the subsequent filtration data are recorded on p. 104.

Table 5. Slurry compositions in coal conversion studies

Experiment No.	Pyridine insolubles (%)	Conversion ^a (%)
89-104	7.0	85.3
91-101	3.2	82.0
103-106	19.5	-0.24
144-150	0.4	72.0
119-122	6.6	88.0
126-124	9.6	74.6
130-132	9.7	80.3
134-136	10.3	76.9
138-139	11.4	69.6
141-142	7.6	82.0
145	11.6	47.5

^aCalculated on a maf basis.

Figure 4 shows the relationship between percent conversion and reaction time at a reaction temperature of 750°F. The maximum conversion occurs at a reaction time of 25 min, after which time repolymerization apparently begins. The high degree of conversion obtained in such a short reaction time (~25 min) is an indication that substantial mixing occurred in the rocking autoclave.² The repolymerization was expected, and the constant conversion values at extended reaction times indicate that repolymerization does not occur to such an extent that conversion is totally reversed.

A particle size distribution was determined for each of the slurries. The resulting curves are shown in Appendix B. The large particle size of the solids made it necessary to use an optical microscope to produce the particle size distribution curve. However, particle size determination by optical microscope is limited because this technique cannot detect the presence of submicron-sized particles (fines). This invalidates a comparison of the number of particles in a given size range for the various distributions and suggests a comparison based on volume of particles. The presence of fines would have very little effect on the overall volume of particles. Accordingly, Fig. 5 shows a comparison of the particle size distributions as a function of conversion at a reaction temperature of 750°F. Specifically, the curves show the volume percent of particles with diameters $<10\ \mu$ as a function of conversion. It should be noted that the values for percent conversion were obtained from Fig. 4. It is immediately apparent that the curve is exponential in shape, with the emergence of small particles increasing dramatically between conversions of 85 and 90%.

Fig. 4. Coal conversion at 750°F as a function of reaction time.

Fig. 5. Comparison between volume of particles <10 μ diam and degree of conversion.

Table 6, showing a comparison between the percent of conversion and the volume percent of particles with diameters $<10 \mu$, illustrates this point quite clearly. The percentage of particles with diameters $<10 \mu$ increases by a factor of ~ 3 (from about 20 to 60%) between 85 and 90% conversion. The data do not contradict the "Swiss-cheese" model of coal dissolution, although the emergence of small particles is not a step function with respect to conversion. Apparently, as conversion progresses, the percentage of smaller particles increases proportionately as individual particles are reacted to such an extent that their lattice-like structure collapses. The proportional relationship continues until a threshold conversion limit is reached between 75 and 85%, at which time the particle structures disintegrate in bulk; this results in the near-step function relationship between percent of small particles and conversion at that point.

The remaining large particles present at all conversions (compared to slurries produced at Wilsonville³) suggest the slurry might be subject to some degree of flocculation. As discussed by Grace,⁴ filtration of slurries subject to flocculation initially produces filter cakes with a high void fraction. As filtration continues, the increasing shear forces cause the flocs to disintegrate into distinct particles. Thus, assuming flocculation is increased by the increasing percentage of large particles, filtration at these lower degrees of conversion would be expected to be characterized by a lower average specific cake resistance and a higher compressibility factor due to their degree of flocculation.

Table 6. Comparison between percent conversion and particle size at a reaction temperature of 750°F

Reaction time (min)	Conversion ^a (%)	Particles <10 μ diam ^b (vol %)
5	47.5	4
10	80.0	16
15	85.0	20
20	87.5	30
25	88.5	64

^aValues obtained from Fig. 4.

^bValues obtained from Fig. 5.

Filtration may be represented by Poiseuille's equation:⁵

$$\frac{dV}{Ad\theta} = \frac{\Delta P}{\mu[\alpha(W/A) + r]} \quad (2)$$

where

dV = change in volume of filtrate collected for change in time $d\theta$,

A = filter cake surface area,

ΔP = pressure drop across cake and medium,

μ = viscosity of filtrate,

W = mass of accumulated solids, dry cake basis,

α = average specific cake resistance, and

r = resistance per unit area of medium.

In some instances, filtration may be represented by the Kozeny-Carmen equation:⁵

$$\frac{dP}{dL} = \frac{k\mu u (1-\epsilon)^2 (S_p/V_p)^2}{g_c \epsilon^3} \quad (3)$$

where

dP = pressure drop through layer of thickness dL ,

k = constant,

u = linear velocity of filtrate based on filter area,

ϵ = porosity of cake,

S_p = surface area of single particle,

V_p = volume of single particle, and

g_c = Newton's law conversion factor.

A comparison between the two equations yields the following mathematical relationship for specifying cake resistance:

$$\alpha = \frac{k(S_p/V_p)^2(1-\epsilon)}{\epsilon^3 \rho_p} \quad (4)$$

where ρ_p = density of the particles.

It is apparent that the variables S_p , V_p , and ϵ are dependent on particle size. Thus the fact that particle size is a function of conversion indicates that changing conversion will change the specific cake resistance which will, in turn, alter the filtration rate.

3.2 Filtration

In an attempt to verify the conclusions of the previous section, each of the slurries was subsequently filtered. A successful filtration was defined as one which reduces the sulfur and ash content of the filtrate to below standards set by the Environmental Protection Agency⁶ for a product with a heating value of 16,000 Btu/lb (i.e., 0.96 and 0.15 wt % respectively). The sulfur content of the filtrate was determined by a mass balance around the filter.

$$\% W_{FS} = 100 \frac{(M_S W_{SS} - M_{FC} W_{FCS})}{M_S - M_{FC}}, \quad (5)$$

where

$\% W_{FS}$ = weight percent of sulfur in filtrate,

W_{SS} = weight fraction of sulfur in slurry,

M_{FC} = mass of filter cake, g, and

W_{FCS} = weight fraction of sulfur in filter cake.

In calculating the ash content of the filtrate, it was assumed that the ash introduced by the coal remains in the system; thus

$$\% W_{FA} = \frac{100(M_C W_{CA} - M_{FC} W_{FCA})}{M_S - M_{FC}}, \quad (6)$$

where

$\% W_{FA}$ = weight percent of ash in filtrate, and

W_{FCA} = weight fraction of ash in filter cake.

The results of these calculations are shown in Table 7. As indicated by the data, the filtration successfully removes the contaminants of interest.

The experimental technique of collecting the filtrate in an open beaker results in some loss of filtrate due to evaporation. This evaporation causes the collected filtrate vs time curve to show less filtrate than has actually passed through the filter. With this in mind, the filtrate curves were corrected to include the evaporated material and a constant evaporation rate was assumed. As indicated in Table 8, the evaporation loss was substantial (~33%). The corrected filtrate curves are shown in Fig. 6. A typical filter cake produced by each filtration is shown in Fig. 7. The short filtration times were such that the sensitivity of the filtrate recorder was exceeded. This caused the linear segments of the filtrate curves rather than the expected parabolic shape.

The resulting overall filtration rates shown in Fig. 8 were calculated by

$$R = \frac{W}{A\theta}, \quad (7)$$

Table 7. Determination of filtrate ash and sulfur content

Experiment No.	Sulfur content of slurry (wt %)	Composition of filter cake		Calculated filtrate composition	
		Sulfur (wt %)	Ash (wt %)	Sulfur (wt %)	Ash (wt %)
89-104	1.1	2.3	22.2	0.9	0.06
91-101	0.9	2.9	25.3	0.6	<0.01
103-106	0.96	3.0	11.2	<0.01	<0.01
108-110	0.77	2.2	45.8	0.6	0.15
119-122	0.98	2.3	28.4	0.8	0.10
126-124	1.1	2.7	21.9	0.7	<0.01
130-132	1.1	2.6	24.2	0.8	0.06
134-136	1.04	2.4	21.5	0.8	0.09

Table 8. Determination of filtrate evaporation rates

Experiment No.	Slurry (g)	Filter cake (g)	Actual filtrate collected (g)	Filtration time (min)	Calculated evaporation rate (g/min)
89-104	522.3	81.8	284	1.5	105.7
91-101	559.4	72.6	276	2.2	95.8
103-106	848.2	275.4	374	3.8	52.3
108-110	759.6	63.4	526	5.5	31.0
119-122	518.0	63.5	280	2.0	87.3
126-124	649.3	96.7	324	2.8	81.6
130-132	608.5	88.9	260	1.8	144.2
134-136	633.9	100.7	316	3.4	63.9
138-139	668.8	135.1	318	4.1	52.6
141-142	734.8	119.7	388	3.5	64.9

Fig. 6. Comparison of cumulative filtrate curves.

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Fig. 7. Typical filter cake produced during filtration.

Fig. 8. Comparison of filtration rates as a function of total filtrate collected.

where

R = overall filtration rate, $\text{lb hr}^{-1} \text{ft}^{-2}$.

W = total filtrate collected, lb,

A = effective filter area, 0.0873 ft^2 , and

Θ = collection time, hr.

It should be noted that Table 5 indicates that conversion is negligible at 400°F and at a reaction time of 90 min. It was thus assumed that reheating the slurries to the filtration temperature of 350°F does not alter the degree of conversion that was determined in the preceding section. The data indicate a substantially constant filtration rate during the initial portions of the run for all filtrations. However, this period of constant filtration rate is probably unrealistic, and the rate actually decreases during this period. The recording equipment simply lacked the sensitivity to determine this slight decrease.

Figure 9 shows a comparison between the overall filtration rates for the various slurries after filtering 1 lb of filtrate. Although the data scatter is substantial, the resulting curve indicates that conversion does, indeed, affect the filtration rate. Significantly, the filtration rate decreases with increased conversion prior to repolymerization (reaction time ~ 25 min). After repolymerization begins (conversion decreased) the filtration rate again appears to increase. The data scatter here prohibits definitive conclusions. It is not clear whether the increased particle size due to repolymerization produces filtration rates similar to those prior to repolymerization at identical conversions or whether repolymerization actually gives higher filtration

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Fig. 9. Comparison of filtration rates at a total throughput of 1 lb of filtrate.

rates due to the formation of a more rigid solid structure. If the latter is true then repolymerization could actually be an aide to filtration.

Classically, constant-pressure cake filtration data are analyzed by the Poiseuille equation.⁵ This analysis determines not only the filtration rate but the average specific cake resistance as well. Ruth⁷ has shown that for the Poiseuille equation to be applicable, the filtrate curve must be in the shape of a parabola with a nonnegative intercept at time equal to zero. This is not the case for these data and, consequently, the Poiseuille equation was not used in this analysis. It is assumed that this deviation from existing theory was due to instrument insensitivity. It should be noted, however, that another possibility⁸ also exists. In our experiments, the end of filtration was assumed to be that point at which the total filtrate collected became constant with respect to time; however, the true end of filtration occurs when the argon overhead gas breaks through the filter cake. Hence, the constant filtrate value may not be the end of filtration but simply an indication that the rate of additional filtrate being collected is equal to the evaporation rate. If gas breakthrough does not occur simultaneously with the leveling of the filtrate curves, the subsequent data analysis will show an erroneously high evaporation and filtration rate.

4. SUMMARY

Particle size distribution data have been collected from slurries exhibiting various degrees of coal conversion. The data show a correlation between the percent (by volume) of particles with diameters $<10 \mu$

and the degree of conversion. The percentage of particles of this size increases steadily to a conversion of ~85%. At higher conversions, the increase in percentage of small particles is almost a step function from about 20 to 60%. These data suggest that by varying the degree of coal conversion the filtration rate of the resulting slurry can be altered due to the variation in particle size (filtration rate is a function of particle size); at reduced degrees of conversion the resulting slurry may also be subject to flocculation.

Subsequent filtration of these slurries indicates that there is indeed a correlation between conversion and overall filtration rate, although the data scatter is substantial. The lowest filtration rates were obtained with slurries having the highest degree of conversion, indicating that the filtration rate is increased with lower conversions. It should be noted that these experiments were exploratory in nature and are therefore insufficient to determine the magnitude of the increase in filtration rate with low conversions. However, implicit in the results obtained is the fact that a degree of conversion exists at which the operational cost of filtration of the resulting slurry is a minimum.

5. RECOMMENDATIONS FOR FUTURE WORK

1. The experiments have shown a relationship between particle size and conversion at 750°F. Particle size distributions on slurries obtained at other reaction temperatures should be obtained to determine if this relationship is primarily a function of conversion or the conditions of conversion.

2. Filtrations of a wider variety of slurries should be made to define the relationship between filtration rate and conversion.

3. Important filter characteristics such as specific cake resistance and compressibility should be determined for the slurries obtained in step 2.

4. The effect of propeller speed in the filter preheater on slurry filterability should be determined.

5. The experimental equipment should be modified to minimize operational and analytical problems. These modifications should include:

a. making the slurry addition a 1-in. line to allow filtration studies on more viscous slurries;

b. installing a rotameter in the gas addition line to determine the point of gas breakthrough (i.e., to determine the end of filtration more accurately); and

c. installing an enclosed filtrate collection vessel to minimize (if not eliminate) the effects of filtrate evaporation.

6. A preliminary economic analysis should be performed using the filtration data obtained to indicate the relationship between conversion and filtration operation costs.

6. ACKNOWLEDGMENTS

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8. APPENDIXES

8.1 Appendix A. Experimental Apparatus

8.1.1 Equipment for coal conversion

The coal conversion experiments were performed in an autoclave that had been hydrostatically tested at 4500 psig. The dimensions of the autoclave are 4-3/8 in. OD, 3-5/16 in. ID, and an inside depth of 10 in.; the approximate volume is 0.4 gal. Figure A.1 shows the various parts of the autoclave. It contains a thermocouple well for two Chromel-Alumel thermocouples and two ports in the top, one of which was used to vent, pressurize, and monitor the pressure of the autoclave. The other port contains a pressure relief valve set at 4000 psig. The autoclave is made of 316 stainless steel. The maximum working pressures for the autoclave are at 39°C, 5050 psig; 260°C, 4600 psig; 343°C, 4550 psig; and 436°C, 4500 psig. The autoclave fits snugly into an electric heater, rated at 1600 W, and the whole assembly can be rocked through an angle of 30° at 36 cycles/min (see Fig. A.2). The pressure in the autoclave is monitored by a 0- to 5000-psi Baldwin gauge and is continuously recorded. The electric heater contains a Variac and was controlled by a thermocouple that rests between the autoclave and the electric heater (inserted through a hole in the bottom of the heating unit) to hold the temperature $\pm 5^{\circ}\text{C}$. The temperature inside the autoclave is continuously recorded. The autoclave can be water-cooled by copper cooling coils that are wrapped around the outside of the heating unit. A sketch of the experimental equipment is shown in Fig. A.3.

8.1.2 Equipment for filtration

After cooling, the material from the rocking autoclave was filtered in the apparatus shown in Fig. A.4. Operations in this area are limited

Fig. A.1. Exploded view of autoclave.

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Fig. A.2. Heating jacket and shaking mechanism used in coal conversion studies.

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Fig. A.3. A simplified schematic of coal conversion equipment.

Fig. A.4. Filtration equipment as it appears in place.

to conditions already experienced in previous laboratory experiments (750°F and 300 psig). The bench-scale equipment precludes using quantities of material larger than 1 liter.

The equipment consists of a 1-liter mixing autoclave that can be emptied into a settling vessel or a filter vessel, as shown in Fig. A.5. The mixing vessel is a standard Autoclave Engineer's MagneDrive Packless Autoclave, rated at 5000 psig. The filter vessel was constructed by Mott Metalurgical Corp. and was hydrostatically tested by the Inspection Engineering Department at 450 psig. The 3-in. 304-stainless steel pipe has a pressure rating of 1300 psi at 750°F. The 300-lb flanges have a pressure rating of 340 psi at 750°F. The nonblow Flexitallic gaskets are rated at over 300 psi up to 1100°F. The settling vessel was constructed at ORNL, and tested at 800 psig. A pressure relief valve, set at 350 psig, is provided on the argon supply line to the autoclave.

Fig. A.5. Schematic of filtration equipment.

8.2 Appendix B. Particle Size Distribution of Slurries

Fig. B.1. Particle size distribution of slurry reacted at 400°F for 90 min.

Fig. B.2. Particle size distribution of slurry reacted at 750°F for 5 min.

Fig. B.3. Particle size distribution of slurry reacted at 750°F for 10 min.

Fig. B.4. Particle size distribution of slurry reacted at 750°F for 20 min.

Fig. B.5. Particle size distribution of slurry reacted at 750°F for 25 min.

Fig. B.6. Particle size distribution of slurry reacted at 750°F for 30 min.

Fig. B.7. Particle size distribution of slurry reacted at 750°F for 50 min.

Fig. B.8. Particle size distribution of slurry reacted at 750°F for 60 min.

Fig. B.9. Particle size distribution of slurry reacted at 750°F for 85 min.

Fig. B.10. Particle size distribution of slurry reacted at 750°F for 110 min.

Fig. E.11. Particle size distribution of slurry reacted at 750°F for 135 min.

Fig. B.12. Particle size distribution of slurry reacted at 750°F for 195 min.

Fig. B.13. Particle size distribution of slurry reacted at 750°F for 280 min.

ORNL-DWG 78-17838

148 X

0 50 100
MICRONS

148 X

0 50 100
MICRONS

Fig. B.14. Optical micrograph of solids in slurry reacted at (top) 400°F for 90 min, and (bottom) 750°F for 5 min.

ORNL-DWG 78-17839

425X

0 25 50
MICRONS

340X

0 40 80
MICRONS

Fig. B.15. Optical micrograph of solids in slurry reacted at (top) 750°F for 10 min, and (bottom) 750°F for 20 min.

ORNL-DWG 78-17840

240X

0 25 50
MICRONS

0 50 100
MICRONS

Fig. B.16. Optical micrograph of solids in slurry reacted at (top) 750°F for 25 min, and (bottom) 750°F for 30 min.

ORNL-DWG 78-17841

425X

0 25 50
MICRONS

425X

0 25 50
MICRONS

Fig. B.17. Optical micrograph of solids in slurry reacted at (top) 750°F for 50 min, and (bottom) 750°F for 60 min.

ORNL-DWG 78-17842

425X

0 25 50
MICRONS

148X

0 50 100
MICRONS

Fig. B.18. Optical micrograph of solids in slurry reacted at (top) 750°F for 85 min, and (bottom) 750°F for 110 min.

ORNL-DWG 78-17843

148 X

0 50 100
MICRONS

425 X

0 25 50
MICRONS

Fig. B.19. Optical micrograph of solids in slurry reacted at (top) 750°F for 135 min, and (bottom) 750°F for 195 min.

148X

ORNL-DWG 78-17837

0 50 100
MICRONS

Fig. B.20. Optical micrograph of solids in slurry reacted at 750°F for 280 min.

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