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CENTRIFUGATION OF COAL-DERIVED LIQUIDS

MASTER

by

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June 1980

Pittsburgh Energy Technology Center
Pittsburgh, Pennsylvania

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ABSTRACT

The application of the continuous solid bowl centrifuge to the removal of solids from coal liquefaction products was investigated. The centrifuge removed from 23 to 88 pct. of the input ash and from 8 to 73 pct. of the input organic benzene insolubles while flow rates, viscosities, and dam heights were varied. Viscosity μ , effluent liquid rate Q_e , and Ambler's geometric parameter Σ were graphically correlated with attained separations. The separation was relatively insensitive to the variables, as a 50-fold increase in Q_e/Σ corresponded to a decrease in ash removal only from 84 pct. to 60 pct. and to a decrease in organic solids removal only from 77 pct. to 22 pct. Organic solids removal was poorer and more erratic than ash removal because of the lesser density differences and greater size variability of the organics. Ancillary studies demonstrated that coal liquefaction products may behave as a Bingham Plastic fluid, and that this results in an absolute limit on the attainable solids separation. Additional studies showed that little difference in density may exist between the organic solids and liquids, and that effects of aging may threaten the validity of viscosity measurements.

INTRODUCTION

All processes in which coal is liquefied to form liquid fuels require the removal of mineral matter and unconverted coal from the product. The thoroughness with which the solids must be removed varies with the requirements of the user and with the internal requirements of the process for a recycle fluid. However, even in the less stringent cases, separation of the solids is difficult because of the small particle size, low density difference between solid and liquid, a physico-chemical affinity between some of the particles and the liquid, and the high liquid viscosity. A panel of the National Research Council regarded the problem to be sufficiently important to recommend (1)^a that "Solid separation techniques should be vigorously investigated to identify the best approach".

A number of separation processes are available. Historically, (2) the German and British coal hydrogenation plants of the 1940's used centrifuges for their "heavy-oil" product. This was not entirely satisfactory. They also tried filtration and distillation, neither of which showed clear superiority. Solids separation was not critical for that period, however, because the "heavy-oil" was a minor product and the distillate was the major product. Centrifugation continues to be popular but inefficient, while

^aUnderlined numbers in parentheses refer to items in the list of references at the end of this report.

filtration is still under study (3) but continues to be plagued by high pressure drop and low capacities because of the properties of the particles and the liquid.

The coal liquefaction processes that have been under development at the Pittsburgh Energy Technology Center have used solid bowl batch centrifuges as the devices for solids removal. These centrifuges have proven adequate for the requirements of the process under study, which had considerable tolerance for solids in the recycle fluid. This report describes an investigation of the operation of continuous centrifuges in coal liquefaction processes that was undertaken for several reasons:

- (a) Batch centrifuges could clearly not be scaled up economically for use in the large-scale developments envisioned for the coal liquefaction industry. Solid bowl continuous centrifuges are the obvious continuation of the batch centrifuges which have proven feasible on the smaller scale.
- (b) Evaluation of the effect of operational and design parameters on capacity is necessary for application in a scaled-up process.
- (c) Elucidation of basic principles will permit optimization of the centrifuge for both present and future process

development and may suggest methods of improving the product quality to meet more stringent specifications.

EQUIPMENT AND PROCEDURE

The experimental separations were carried out with two sets of equipment, of similar principles but different construction. Both sets were constructed around centrifuges whose operating principle is illustrated in Figure 1. Slurry is pumped into the axis of the spinning cylindrical bowl where it impacts on surfaces which give it a rotary motion. The centrifugal force generated by the rotation throws the slurry radially against the wall of the rapidly rotating bowl. What happens next is most easily visualized by considering the lower half of the centrifuge in Figure 1 as if the operative field were gravity rather than centrifugal force. The slurry is retained in the pool whose side walls are the sloping cone at the right end of the bowl and the dam at the left. The height of the dam is less than that of the cone, so that the liquid traverses the pool and overflows the dam. During its traverse, whose length of time is set by the interaction of the height of the dam, the volume of the pool, and the rate of feed, the solids, which are heavier than the liquids, settle out so that only the clarified liquid overflows the dam.

Figure 1-Operating principles of continuous centrifuge

(Courtesy of Pennwalt Corp., Sharples-Stokes Div.)

The solids are pushed to the right by the conveyor screw, which is driven by the same pulley as the bowl but at a few revolutions per minute less than the bowl due to the interposition of a differential gear. Excess liquid drains from the solids as the latter are pushed up the slope to the discharge nozzles through which they are thrown into a solids receiver.

Horizontal Centrifuge Tests

Equipment

The first series of continuous centrifugal tests was carried out in a horizontal centrifuge, Sharples Super-D-Canter Model P660^b, whose construction is very closely portrayed by Figure 1. The differential gear could be continuously adjusted to provide any desired differential between bowl and conveyor from 0 to 64 rpm. The depth of the pond was adjustable by opening the horizontally split casing and rotating plates in the dam that carried eccentrically drilled holes. In this machine, the weight of the rotating bowl is carried on bearings at each end, with a seal at each bearing and an additional seal between the rotating bowl and the fixed feed tube. The ancillary equipment for this apparatus was a steam-jacketed stirred vessel for heating the feed and a pump. Both were mounted in close proximity to the centrifuge so that no insulation

^b Appearance of commercial or trade names and model numbers does not imply endorsement of the products by the U. S. Government or the Department of Energy. Such names are used only for identification.

or steam tracing was required to maintain a temperature difference of less than 15° F between the slurry in the feed tank and the centrifuge liquid discharge.

Procedure

In this series, centrifugation was performed on a gross liquid product (GLP), i.e., the unseparated mixture of liquids and solids of the reactor effluent from a coal hydroliquefaction process described by Akhtar, et al (4)^c. Two fifty-five gallon drums of GLP, an analysis of which is given in Table 1, were emptied into the feed tank and brought up to test temperature by adjustment of the steam flow to the feed tank jacket. The pump was started and the flow rate, as measured by weighing

TABLE 1.- Analyses of feed materials for horizontal continuous centrifuge experiments HC-1 thru HC-11

Sp. Gravity	
60°/60°	1.099
Viscosity	
SSF @ 140° F	32.3
SSU @ 203° F	76.3
Organic Benzene	
Insoluble, pct.	3.5
Organic Benzene	
Soluble, pct.	90.4
Ash, pct.	6.1

^cThe operating parameters that produced the material used in the various experiments are listed in the Appendix, Table A-1.

a timed discharge from the centrifuge, was adjusted to the desired rate. After discharge of about 5 gallons, steady state was reached. (This had been determined by preliminary experiments.) Samples of liquid and residue streams were collected for subsequent analysis. The flow to the centrifuge was then stopped. Changes were made in feed temperature and conveyor differential as specified, the pump was restarted, a new flow rate was established, and a new set of samples was taken. After all samples were taken, they were analyzed by the Pittsburgh Energy Technology Center Analytical Division. Experimental parameters and results are listed in the Appendix Table A-2 and are discussed in the "Results" section.

Vertical Centrifuge Tests

Equipment

A vertical centrifuge (Sharples Model P850) was installed with considerably more complex facilities than the horizontal unit. The vertical centrifuge is similar in principle and operation to the horizontal machine described in Figure 1, with the axis of rotation changed to the vertical. Since the centrifugal forces that drive the solids to the bowl wall are approximately 3,000 times gravity, the influence of gravity on the separation is negligible, but the vertical configuration imparts several practical advantages. The bowl and conveyor are suspended from the top like a plumb bob so that no lower bearing, other than a non-load carrying guide, is necessary. The upper bearing and seal can be isolated from the erosive slurry. This permits

a more effective seal, and the vertical centrifuge can be operated under pressure. For processing coal liquefaction products, this means that the centrifugation temperatures can be higher without loss of volatiles.

The solids discharge from the centrifuge fell into a receiver constructed from a 20-inch standard welding pipe T as shown in Figure 2. One end of the T was closed with a cap that had been provided with 1-inch and 1 1/2-inch pipe connections near the bottom of the T. The other end was fitted with a hinged 20-inch closure so that the receiver could be emptied manually by a shovel, if necessary.

Unlike the horizontal centrifuge, the differential gear that drove the conveyor screw of the vertical centrifuge was operated with the pinion gear locked. This provided a fixed speed ratio. After initial tests, however, it was modified so that the pinion gear was belt-driven by an auxiliary motor. The speed of the pinion gear could be altered by changing the belt pulley diameters. The differential speed of the conveyor is given by the formula:

$$\text{Differential rpm} = \frac{\text{bowl rpm} - \text{pinion rpm}}{\text{gear ratio}}$$

To protect the centrifuge against damage if the conveyor is jammed or overloaded by excessive deposits, the pinion drive motor was wired into the main drive electrical system so that overloading of the auxiliary motor would shut off the power to the main drive as well as to the auxiliary. An optical

Figure 2.-Solids receiver for continuous centrifuge.

reflective tachometer was mounted on a movable shaft so that both bowl speed and pinion speed could be measured.

The dam of the vertical centrifuge was a simple orifice plate clamped within the rim of the bowl. Changing the dam height was accomplished by removing the pulleys, gear box, and bowl from the casing and replacing the orifice plate with one of a different inside diameter.

Centrifuge casing and solids receiver were vented to a common point so that no pressure difference could exist between the two sections that would prevent free-fall of the solids into the receiver. Figure 3, the diagram of ancillary equipment and piping, shows that the vent lines were equipped with drains emptying into light oil Pot 2, so that liquid that was condensed from the hot vapors in the system, or that was carried into the vent lines by entrainment, was removed from the vented streams.

The configuration shown in Figure 3 was designed for two functions: (1) to take maximum advantage of the temperature and pressure capabilities of the vertical centrifuge; and (2) to permit integration of the facility into the routine operations of the liquefaction process development units. The feed tank for the centrifuge, T1, and the CLP (Centrifuged Liquid Product) tank, T3, were sealed and capable of operating at 50 psig. They had "Panelcoil" jackets connected to a 20 kilowatt system that supplied heat transfer oil at temperatures up to 550° F. Both T1 and T3 were mounted on weigh scales and were equipped with propeller-type

Figure 3 - Continuous centrifuge flowsheet.

agitators. A progressing cavity slurry pump, with steel rotor in a steel casing (P1), recirculated the contents of T1 by pumping from a bottom outlet to a top inlet and also served as the transfer pump from T1 to the centrifuge. A similar pump (P3) recirculated the contents of T3 and could transfer these contents to a 55-gallon drum, to T1 for reprocessing, or to the liquefaction plant coal vehicle supply tank. Because of limited head room, it was not possible to position the centrifuge so that the CLP could flow by gravity to the product tank T3. A small accumulator tank (T2) received the discharge from the centrifuge. A slurry pump (P2) was automatically activated when T2 was filled and pumped the contents of T2 into T3.

A network of nitrogen supply lines and vent lines connected the centrifuge and vessels of the system so that the liquid could be transferred from the GLP tank to the centrifuge, thence to the CLP tank, either under uniform gas pressure so that the transfer was entirely under control of the pumps or in case of pump failure, under pressure differentials that could produce the desired flow rates. All gases passed through a heat exchanger that condensed any light oil vapors into the light oil trap, Pot 1, before the gases passed through the back pressure regulator, B.P.R., to the vent line. The line labeled "LED-1 metered vent" (upper left corner of Figure 3) permitted tank T1 to be used by the liquefaction engineering development unit (LED) as a regular

GLP receiver, since gases that were discharged along with the GLP could be returned to the LED unit.

All components of the system were insulated and steam traced, except for T1 and T3 which had hot oil jackets. The steam for the tracing was available either from the building supply at 90 psig or from a regulator which was adjustable between 5 and 45 psig.

Instrumentation

Temperatures were measured by iron-constantin thermocouples and recorded on a multi-point recorder. The points measured were the contents of tanks T1 and T3, the centrifuge inlet and outlet, the inlet and outlet of the T1-to-centrifuge transfer line, and the inlet and outlet of the jacket of T1. Temperature was regulated by on-off control of the power to the heat transfer oil electrical heater. This function was performed by an indicator-controller whose input signal during heat-up was the thermocouple in the recirculation loop of tank T1. When centrifugation was begun, the sensing of the controller was switched to the thermocouple at the centrifuge inlet. A second controller, which measured the temperature of the heat transfer oil directly, was set to override the main control if the heat transfer oil temperature exceeded that of T1 by 100° F.

Bourdon gauges measured the pressures in the vapor spaces of the centrifuge and each vessel. Liquid levels in T1, T2, and T3 were measured by nitrogen bubblers whose pressure difference

from their respective vapor spaces was transduced by differential pressure cells to pneumatic signals which were recorded on circular charts. The contents of T1 and T3 were also measured by the weigh scales on which these tanks were mounted, but because of all the piping connections to these tanks, the motion of the scales was somewhat hindered and the weights were not dependably free of drag forces.

Flow rates were measured by an all-steel rotameter with a magnetically-coupled indicator and by differentiating the liquid level changes measured by the weigh scales, the nitrogen bubblers, and a capacitance probe in accumulator tank T2. Although each method had drawbacks, the various methods were complementary, and the accuracy, as shown by material balances discussed in a later section, was good. The problems and solutions of the flow measurements have been discussed in another publication (5).

Procedure

Three of the parameters that defined the operating conditions for a given centrifugation were adjusted by physical alteration of the centrifuge. These were: (1) the bowl speed, which was changed by replacing the drive belt pulleys; (2) the conveyor screw differential, which was changed by either replacing the auxiliary drive pulleys or removing them and locking the differential pinion gear; and (3) the dam height, which was changed, as discussed in the preceding section, by replacing the dam. After these

parameters were fixed, the entire system was purged with nitrogen and pressure-tested. All instruments were activated. The steam tracings and the heat transfer oil system were activated, and the latter was brought up to 250° F. The feed material (preheated by drum heaters if this was necessary to insure fluidity) was transferred to feed tank T1, and the system was pressurized with additional nitrogen to 25 psig. Temperature control of the heater was placed under supervision of the thermocouple in the T1 recirculation loop, and the set point for the controller was adjusted to 50° F higher than the desired centrifugation temperature. The lubrication control panel for the centrifuge was activated and, when lubricating oil flows had stabilized within the safety-prescribed rates and temperatures, the conveyor auxiliary motor and then the main centrifuge drive motor were turned on.

When the temperature in the feed tank had reached the set point, the latter was readjusted to the desired centrifugation temperature, and supervision of the temperature controller was switched to the thermocouple at the centrifuge inlet. A portion of the flow recirculating through valve V6G (refer to Figure 3) was directed to the centrifuge at a rate measured by the rotameter and adjusted by valve V53G. Finer adjustment of the flow was accomplished while the desired amount of material was being centrifuged by calculating the flow from timed decreases

in the measured level of T1 or by the rate of rise in T2 while the accumulator pump P2 was inactive. Liquid samples were withdrawn at the feed pump through valve V25G and at the accumulator exit at V51G. Although it has since been shown that, during continuous production runs, solids can be continuously discharged through a one-inch line (not shown in Figure 3) in the bottom of the solids receiver, this was not attempted during the evaluation tests, as the representativeness of such samples would be in doubt. Instead, after a run was completed and the system purged and partially cooled, the contents of the receiver were removed through the door in the end of the receiver and properly sampled.

Sequence of Tests

After assembly of the equipment, four tests were run to check out its operation. Although the preliminary nature of the tests and some faulty instrumentation obviated an analytical treatment of these early results, the tests established that: (1) the machine was operable at the desired temperature and pressures; (2) it could be shut down without flushing and restarted several days later without difficulty, although a "light oil" flush prior to shutdown was advisable to reduce binding of the conveyor to the bowl; and (3) a minimum of five minutes on stream was necessary to establish a steady state of ash removal. Operating conditions and results for succeeding runs are tabulated in Appendix A-3 and A-4 and are discussed in the next section.

The feed materials were produced in the same equipment that provided feed for the horizontal centrifuge tests. The liquefaction experiment parameters varied, however, and the various feed batches thus had somewhat different properties as reflected in the analyses and viscosities reported in Table 2. Tests CC-8 to CC-22 were intended to be run as a factorially-designed series with uniform feed. For this purpose, 300 gallons of GLP were accumulated, thoroughly mixed and then stored in 55-gallon drums. However, the extended storage period and pre-heating of the drums caused measurable changes in the composition of the feed slurry, as indicated in Table 2. The operating variables, temperature and feed rate, were randomized in the sequence of runs, as shown in the later discussion.

RESULTS

Horizontal Centrifuge Tests

Primary measurements and product analyses from the horizontal continuous centrifuge experiments are tabulated in the Appendix, Table A-2. From an ash balance:

$$A_f \times F = (A_c \times C) + (A_r \times R) \dots \dots \dots (1)$$

where A_f , A_c , A_r = fraction ash in feed, centrifugate, and residue, respectively

F = Pct. feed = 100

C = Centrifugate, pct. of feed

R = Residue, pct. of feed = 100-C

TABLE 2.- Analyses of feed materials for vertical
continuous centrifuge experiments

Expt. No.	Sp. Gravity 60°/60°	Viscosity SSF @ 180° F	OBI pct	Asphaltenes pct	Oil pct	Ash pct
CC-2	-	211.0	5.1	30.1	59.1	5.7
3	-	284.0	4.1	28.7	63.4	3.8
4	-	290.0	4.1	28.4	63.6	3.9
5	1.139	47.6	4.4	27.2	63.6	4.8
6	1.143	79.8	4.3	29.5	61.4	4.8
7	1.141	94.4	4.3	24.5	66.4	4.8
8	1.153	112.0	4.6	28.5	61.1	5.8
9	1.164	168.0	5.0	28.2	61.1	5.7
10	1.166	167.0	4.5	31.7	57.9	5.9
11	1.151	160.0	4.8	28.8	60.2	6.2
12	1.167	257.0	4.9	27.3	61.7	6.1
13	1.168	232.0	4.9	29.4	59.7	6.0
14	1.132	271.0	4.8	37.4	51.9	5.9
15	1.157	136.0	4.5	27.7	61.9	5.9
16	1.159	184.0	4.9	37.1	52.1	5.9
17	1.173	212.0	4.8	29.1	60.3	5.8
18	1.163	220.0	4.7	39.1	50.2	6.0
19	1.169	183.0	5.0	37.1	51.9	6.0
20	1.161	195.0	4.2	28.6	61.1	6.1
21	1.168	194.0	4.9	26.5	62.3	6.3
22	1.169	199.0	4.9	28.4	60.7	6.0
23-1	1.143	68.0	3.8	21.9	67.2	7.1
23-2	1.143	63.0	3.9	23.6	65.4	7.1
23-3	1.145	67.2	3.5	24.1	65.5	6.9
23-4	1.138	67.0	3.8	25.1	63.8	7.3
23-5	1.138	65.5	3.3	24.8	65.3	6.6
23-6	1.137	64.0	3.8	22.4	67.3	6.5
23-7	1.142	60.0	3.8	21.9	67.0	7.3
24-1	1.137	91.0	4.0	25.2	63.5	6.5
24-2	1.144	107.0	3.5	24.7	64.8	7.0
24-3	1.133	95.0	3.7	23.9	65.4	7.0
24-4	1.130	90.0	3.9	22.2	66.7	7.2
25-1	1.121	46.0	3.4	20.9	69.5	6.2
25-2	1.130	49.0	3.4	23.7	66.7	6.2
25-3	1.141	53.0	-	20.5	70.2	-
25-4	1.132	58.0	4.0	21.4	68.6	6.0
25-5	1.140	62.0	3.3	20.6	69.9	6.2
26-1	1.143	62.0	3.5	21.7	68.2	6.6
26-2	1.141	59.0	3.7	20.2	69.6	6.6
26-3	1.141	59.0	3.4	21.6	68.4	6.6
26-4	1.141	56.0	3.6	22.8	67.1	6.5
27-1	1.173	143.0	5.8	35.6	53.5	5.1
27-3	1.162	151.0	6.0	29.5	59.4	5.1

whence yield of centrifugate is:

$$C = (A_r - A_f) / (A_r - A_c) \dots \dots \dots (2)$$

Percent of the ash removed from the part of the feed that became centrifugate (not considering the liquid that went to the residue stream) is:

$$S_A = 100 \times (A_f - A_c) / A_f \dots \dots \dots (3)$$

Similarly, percent of organic solids removed is:

$$S_{OBI} = 100 \times (OBI_f - OBI_c) / OBI_f \dots \dots \dots (4)$$

Applying these equations to the data of Table A-2 yields the graphs of Figures 4, 5, and 6. Inspection of Figure 4 shows that for the seven measurements at 203° F, the ash removal attained was strictly monotonic with the flow rate; i.e., each increase in flow rate caused a corresponding decrease in the amount of ash removed. A similar relation can be noted for the measurements at 140°; but comparing the separations attained at 140° with those at 203° for equivalent flow rates, it is apparent that the increase in temperature produced an improved separation that could be attained at the lower temperature only by considerably reducing the flow rate. For example, the ash separation that occurred at 203° F and 2.5 gpm could be attained at 140° F only when the flow was reduced to 0.4 gpm.

This data will be further analyzed in the "Discussion" section of this paper, but two other important facts are immediately noticeable in Figures 5 and 6. The separation of the organic solids is poorer and more erratic than that of the inorganic

Figure 4 - Ash removal in the horizontal centrifuge.

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Figure 5 - Organic solids removal in horizontal centrifuge .

10-20-79 L-16326

Figure 6 - Solids removed as a function of yield for horizontal centrifuge experiments.

10-20-79 L-16327

solids; and the relation between the yield and the ash separation is monotonic -- as the ash removed decreased from 77.0 pct. to 23.0 pct., the yield increased from 89.0 to 96.4 pct. of the feed.

Vertical Centrifuge Tests

Operating parameters and proximate analyses of the centrifugate product of the vertical centrifugation experiments are tabulated in Appendix Tables A-3 and A-4. Elemental analyses are given in Table A-5. Analyses of the residue streams are provided in Table A-6. Residue analyses were not obtained in each run because in a number of the experiments it was expeditious to pool the solids from several experiments. This was done to minimize the number of times the system had to be purged and cooled prior to opening the solids receiver.

An estimate of the precision of the measurements may be made by material balances. These balances were made by pooling the data from several runs in order to conform with the measurements of the residues which had been pooled for reasons given above. The balances are shown in Table 3. The early runs had 8.8 pct. unaccounted for, but all balances made after experiment CC-7 closed within 94.7 to 99.4 pct.

TABLE 3.- Material balances during centrifugation

Pooled Experiments Expt. Nos.	Feed Processed, lb.	Product, pct. of feed	Residue, pct. of feed	Condensates, pct. of feed	Unaccounted, pct. of feed
CC-5,-6,-7	311	76.5	10.8	3.9	8.8
CC-8,-9,-10	401	84.0	9.7	1.0	5.3
C-11,-12, -13,-14	388	84.5	11.3	3.2	1.0
CC-15,-16	430	85.6	9.3	0.5	4.7
CC-17,-18,-19	377	88.6	9.2	0.8	1.5
CC-20,-21,-22	342	87.1	7.9	0.9	4.1
CC-23	351	86.0	11.2	2.2	0.6
CC-24	261	83.8	13.8	1.0	1.4
CC-25	330	85.1	11.2	1.8	1.8
CC-26	245	80.9	15.1	2.1	0.8
CC-27	453	86.5	9.7	0.8	2.9

DISCUSSION

Theoretical Considerations

When a suspension is subjected to a centrifugal field in a solid bowl centrifuge, as in Figure 1, the particles are acted upon by a centrifugal force which tends to drive the particles radially outward, and by a retarding force due to the drag between particle and fluid. For the particle sizes with which we are concerned, it can be shown (6) that when the forces are in equilibrium, corresponding to a constant radial velocity, v_s , for a particle of equivalent spherical diameter d :

$$v_s = \frac{\Delta \rho d^2 \omega^2 r}{18 \mu} \dots \dots \dots (5)$$

By assuming plug flow through the centrifuge Ambler (7) developed equation 5 into an equation for d_c , the "cut-off" diameter, representing the feed particle size above which most particles were removed

from the product stream and below which size most particles went out with the "clarified" product:

$$d_c = \sqrt{(18\mu/g\Delta\rho)(Q_e/2\Sigma)} \dots\dots\dots (6)$$

The first term in parentheses includes only the variables pertaining to the fluid properties; Q_e is the volumetric flow rate of the clarified liquid effluent, and:

$$\Sigma = \pi L \omega^2 (3/4 r_c^2 + 1/4 r_d^2) \dots\dots\dots (7)$$

an expression which includes only the variables pertaining to centrifuge speed and dimensions. A number of investigators have challenged Ambler's assumption of plug flow and have developed correlations based on alternate flow patterns including parabolic flow (8), layered flow (9, 10), turbulent flow (11), and slip between bowl and fluid (12). Hilditch and Rushton (13) compared their own experimental results (for which they used aqueous slurries of sand and of zirconium silicate) with those predicted by some of the correlations referred to. They found that their data best fitted an equation suggested by Reuter (10), but even this correlation gave ratios of observed to calculated separation diameters of 3:1 to 1:3, depending on the diameters. It must be recognized that each of the flow pattern assumptions is probably valid in a specific combination of particle size distribution, fluid properties, flow rates, and machine configuration, and no a priori selection can be made. Coal liquefaction products differ from most fluids that have been used in centrifugation research in having higher values of viscosity, non-Newtonian characteristics, and possible variability in particle size distribution. Because of

the lack of more closely relevant correlating parameters, however, centrifuge operating parameters will be generally characterized by Ambler's Σ (equation 7), or d_c , (equation 6), although the latter assumes that the cut-off diameter is proportional to the square root of Q_e , a proportionality which is valid only for plug flow.

Separation Effectiveness

Although the "cut-off" diameter, d_c , as defined by equation 6 is a simple function of $Q_e \mu / \Delta \rho \Sigma$, its relation to the separation attained is complex. Several factors contribute to this complexity:

- (1) The presence of $\Delta \rho$ in the equation indicates that there is a separate d_c for each solid component of different density. In terms of coal liquefaction products, this means that different values of d_c exist for the ash and for the organic solids, with possibly more than one value of d_c for the latter.
- (2) Since most particle size distributions are asymptotic to some maximum and minimum size, their cumulative weight curves are "S"-shaped. The change in the amount of solids separated at a given cut-off size, d_c , when d_c is varied, will depend on how close d_c is to the "knee" in the curve. If d_c is just above the lower knee of the curve, a small decrease in d_c will cause a large increase in the amount of solids removed, whereas a small increase in d_c would cause only a small decrease in the separation.
- (3) In equation 6, the linearity of μ with d_c is predicated on Newtonian behavior of the fluid. Viscosity measurements.

have shown that liquefied coals are, in general, non-Newtonian and are usually Bingham plastic. The latter term signifies that a finite force has to be applied to cause any continuous motion in the fluid (as compared with a Newtonian fluid where an infinitesimal force is sufficient to produce motion), and hence there will exist an absolute limit of particle size for any given relative centrifugal force below which a particle will not be separated even with infinite residence time; i.e., for Q_e equal to zero.

(4) The properties of both liquids and solids in the feed materials can differ significantly even though the materials are from similar sources. Different liquefaction conditions can produce liquids with considerably different viscosities and with different content of organic insolubles. Storage conditions can also alter the liquid properties. These differences are documented by variation in the fluid viscosity values noted in the data in the Appendix tables. Changes in particle size distribution also occur due to particle agglomeration with time. Size changes are more difficult to measure than viscosity changes. Size distributions of coal liquefaction products have been reported (14), but the results are quite speculative because of the poor state-of-the-art of measurement techniques. As research at the University of Michigan (14) has demonstrated, measured values of size distributions of particles in coal liquefaction products can vary by a factor of 50, depending upon the type of instrument used.

(5) The linearity of Q_e with d_c is predicated on plug flow. The numerous deviations from this assumption have been discussed in the previous section on "theoretical considerations".

In order to accommodate the data analysis to the multiplicity of d_c values, factor (1) above, separate correlations are presented for ash and for organic solids. This distinction also allows for the fact that ash and organic solids probably have different size distributions because the particles are formed through different mechanisms. Ash particles are generated by physical disintegration of the mineral matter in the feed coal, whereas the organic solids are products of complex chemical alterations, polymerizations, and depolymerizations and probably have a diameter distribution extending down into colloidal sizes.

Some indication of size distribution (factor 2, above) can be seen by inspection of Figure 7. Direct measurement of size is extremely difficult, as noted in the discussion of factor (4) above; the size distribution curve of Figure 7 was constructed from centrifuge test operations on the assumption that the value of d_c calculated according to equation 6 was a good approximation to the minimum diameter of the particles actually removed. Figure 7 reveals three important facts: (a) the weight-diameter size distribution was not statistically normal, as shown by the asymmetry of the curve about the 50 weight pct. value; (b) operation near the limiting values of d_c that were obtained in the centrifuges that were tested, means that operations were close to the

Figure 7 - Correlation of ash separation with theoretical particle diameter for two centrifugation series.

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"knee" of the distribution curve which, for the reasons discussed earlier, signifies that small changes in operation can create large changes in separation; (c) the size distribution of the feed for the horizontal centrifugation was materially different from that for the vertical centrifuge tests. Those variations in the separations that are attributable to the "S"-shape and to the variability of the size distribution curves will be accommodated in the following analysis by correlating the results with graphs in which the separations are scaled with probability coordinates. No special consideration will be applied for factors (3) and (5), other than the recognition that they will contribute to the observed experimental error.

Analysis of Observations

The centrifuge operator has six variables under his control after the feed material is selected. These are dam height, bowl speed, pressure, temperature, flow, and conveyor/bowl speed differential. In the present investigation, the bowl speed was not varied. The pressure was maintained between 18 and 28 psig, but the sole purpose of pressure control is to reduce loss of light components from the oil being centrifuged, and pressure has no effect upon the solids separation process other than to prevent change in the properties of the oil by loss of components. It performs this function adequately, as shown by the material balances discussed in an earlier section. Of the remaining variables, dam height, temperature (as embodied in the viscosity of the fluid) and flow are all incorporated

in the Ambler correlation; the last variable, conveyor speed, has no direct effect on the radial motion of the solids and hence on the solids separation, but does have a small indirect effect because it controls the draining of the solids on the exit cone and hence adds a small amount to the liquid stream. The major effect of the conveyor speed is on the dryness of the residue.

Effect of temperature and flow

It has already been shown by Figure 7 that one form of the Ambler analysis, d_c , provides a good correlation for the centrifuge. A close check of this correlation is, however, precluded by the variability of the feed between experiments. A constant feed analysis can be maintained if a short experiment is run with a single charge in the feed tank. A moderate range of temperatures and flow rates was obtained in some of the runs over a period of time. Figure 8 shows the changes of temperature, flow, and product ash analyses with time during experiment CC-23.

The lowest curve in Figure 8 is for the centrifuge inlet temperature and the middle curve is for the flow rate. These two values are combined by Ambler's equation to produce the upper curve, which is for the theoretical cut-off diameter, d_c . The measured ash content of the product is superimposed on the d_c curve. It is apparent that the ash removal follows d_c quite closely, except for the single displacement at 180 minutes.

Figure 8 - Feed temperature, effluent rate, cut-off diameter and product ash content changes with time.
Vertical centrifuge experiment 23.

Effect of dam height

Because average operating conditions for experiments 8 to 14 as a group were similar to those for experiments 15 to 22, except for dam height, it is possible to make some simple comparisons. Table 4 shows the close equivalence of temperatures and pressures for the two groups. The dam heights are in the ratio 2:1, but when these heights are converted to radii, as called for in equation 7, and Σ is calculated, the sigma values

TABLE 4.- Effect of dam height

Experiment No.	8-14	15-22
Dam No.	4	2
Dam height, inch	0.6250	0.3125
Avg. flow, gpm	0.49	0.50
Avg. temp, ° F	306.2	303.4
Ash in product, pct.	2.36	2.42
Liquid in residue, pct.	51.6	49.1

differ by only five percent. As would be predicted, Table 4 shows that the decrease in dam height produced a small increase in the ash content of the product due to a decrease in liquid residence time, and a small decrease in the liquid content of the residue due to a longer drying section. The changes are so small, however, that they are within the range of experimental error and these observations should not be taken as definitive.

Data correlation

In summary of the previous discussion, for any given feed material, the factors having the greatest effect on solids

separation are: (1) temperature or, more precisely, viscosity; (2) flow rate; (3) centrifuge speed and geometry; and (4) the solids properties of size distribution and buoyant density (difference in density between solid and liquid). Factors (1), (2), and (3) may be combined in a separation parameter according to Ambler's equation (6) as the product $\mu Q_e / \Sigma$.

The measured separation attained may be plotted against $\mu Q_e / \Sigma$ as in Figures 9 and 10. In consideration of the expected normal distribution curve, the separation is shown on a probability scale and since the organic solids and the ash have different distributions and different densities, separate curves are presented for the ash separation (S_A) and the organic solids (S_{OBI}) measured as organic benzene insolubles. Figures 9 and 10 show that this selection of coordinates gives a generally linear representation over a more than ten-fold range of $\mu Q_c / \Sigma$. The fact that the data for the horizontal and vertical centrifuges are not colinear may be ascribed to several sources, including geometric differences and the flaws in the Ambler assumptions described in an earlier section, but the most probable explanation is that the feed materials differed substantially in particle size distribution and in the degree of non-Newtonian behavior of the liquids. The fact that the differences are more pronounced for the OBI than for the ash is consistent with the concept that, whereas the ash particle sizes are the result of the natural coal structure and coal grinding, the OBI are

Figure 9 - Ash removed as a function of operating parameter $\mu Q_e / \Sigma$

Figure 10 - Organic solids removed as a function of operating parameter $\mu Q_e / \Sigma$

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produced by complex dissolution and polymerization processes that are very sensitive to the coal liquefaction conditions, and hence OBI particles are more variable than are ash particles.

Effects of conveyor/bowl differential

Comparisons of three pairs of experiments in the horizontal centrifuge are shown in Table 5. The members of each pair have approximately the same temperature and flow rates, but one member had a conveyor speed 10 rpm less than the bowl and the other member 30 rpm less. The configuration of the conveyor is such that a smaller differential causes the solids to have a longer retention in the drying zone than does a larger differential. In two of the comparisons the ash content of the product was slightly better for the larger differential, although the differences were small enough to be within experimental error; in the third comparison, the product was poorer for the larger differential. The differences could be accounted for by the slight variation in the flow rates. In all three comparisons, however, the liquid content of the residue was significantly less for the smaller conveyor differential.

TABLE 5. -Effects of conveyor/bowl differential

Expt. No.	Diff. Speed rpm	Horizontal Centrifuge			Liquids in Residue pct.
		Temp ° F	Flow gpm	Ash in Prod. pct.	
9	10	204	1.30	2.9	40.4
8	30	203	1.22	2.8	41.5
10	10	205	2.50	3.5	39.8
5	30	200	2.42	3.3	43.3
11	10	205	0.54	1.4	40.5
7	30	202	0.57	2.0	42.8

Expt. No.	Diff. Speed rpm	Vertical Centrifuge			Liquids in Residue pct.
		Temp ° F	Flow gpm	Ash in Prod. pct.	
27	6.3	325	0.42	1.7	45.7
15,16	64.0	325	.98	2.5	51.3
26	6.3	301	.22	1.6	42.7
20,21,22	64.0	274	.34	2.5	46.0
24	6.3	320	.22	1.9	42.3
17,18,19	64.0	318	.35	2.3	50.1

Comparisons of the data from the vertical centrifuge are less well defined than those from the horizontal centrifuge because the operating parameters for the experiments made with the low conveyor differential (Expts. 23 to 27) could not be matched with those for the higher differential (Expts. 8 to 22), and because the residues removed from these runs were pooled from several experiments and hence could not be exactly associated with any single set of conditions. The ordinate in Figure 11 differs somewhat from that used in Figure 9. Figure 9 correlates ash removed from the material that became product, and hence is descriptive of product quality, but Figure 11 correlates ash

Figure 11 - Effect of conveyor differential on ash separation in the vertical centrifuge

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removed from the total feed, thus incorporates the effect of variation in yield, and is more descriptive of the operation of the centrifuge. It is clear from Figure 11 that, as a group, the ash removed at the lower conveyor differential followed the same function of $\mu Q_e / \Sigma$ as the other experiments. Hence, the conveyor speed had no observable effect on product quality. The residues, however, were consistently dryer at the lower conveyor speeds, as can be seen in all comparisons made in Table 5.

Ancillary Studies

Operational adequacy

The previous discussion has developed quantitative correlations predicting the separation attainable with the specified operating parameters. The correlation is incomplete because some of the parameters, chiefly those describing the feed material, remain unquantified. The question of the adequacy of the attained separation in terms of the process requirements can be addressed in two ways. In the first, measurable properties of the products can be compared with specifications established by the consumer of the product. This approach is difficult when the requirements of the user may be continually changing, as they are in an evolving industry such as the utilization of liquefied coal.

The second approach is to compare the results obtained in the new device with those obtained in a prior device, results known to be acceptable if not optimum. Such a comparison

is made in Table 6, where experiments in the continuous centrifuge are compared with runs in the batch centrifuge with equivalent feed. The batch centrifuge (a Sharples No. 16 Super Centrifuge) has been in use at the Pittsburgh Energy Technology Center since the initiation of the coal liquefaction program. It has proved adequate for producing products whose solids content was sufficiently low that they could be continually recycled, an operation necessary for the development of a coal liquefaction process. The separations shown for the batch (PDU) centrifugations of both FB-61A and FB-62 were the results of two successive centrifugations of each batch.

TABLE 6. -Comparison of products from continuous centrifuge and from batch centrifuge

A. ASH REMOVAL

Cfge. Run No.	CC-8 to -22	PDU: FB-57	CC-23,-24	PDU: FB-61A
Cfge. feed source:				
Liquefaction run	FB-57	FB-57	FB-61A	FB-61A
Batch nos.	81,87,91,107 109,133,150 to 162	92 to 132	110 to 118	109
Avg. cfge. temp, ° F	305	(200)	280,320	(200)
Ash in product, pct.	2.4	3.1	1.6,1.6	2.2
Sulfur in product, pct.	0.76	0.83	0.5,0.4	0.7

B. VISCOSITY CHANGE

Cfge. Run No.	CC-27	PDU: FB-62
Cfge. feed source:		
Liquefaction run no.	FB-62	FB-62
Batch nos.	76,78,84,90,96	74,79,85,91,97

TABLE 6. -Comparison of products from continuous centrifuge and from batch centrifuge (continued)

Avg. viscosity input, SSF @ 180° F	147	128
Viscosity of mixed product	125	171 ^a

^aCalculated as arithmetic average of separate batches

In all cases, the separations attained in the comparable continuous centrifuge run were better than those of the batch centrifuge. Section A of table 6 shows that the ash removal was more thorough in the continuous centrifuge. Section B shows another aspect of the operation. Continuous exposure to heat and air causes an increase in the oil viscosity, sometimes referred to as aging; removal of solids causes a viscosity decrease. Thus, it is not possible to predict, a priori, the effect of centrifugation on product viscosity. In both continuous and batch centrifuges, access of air is reduced by the use of a blanket of nitrogen. The sealing and the reduced handling of the oil in the continuous centrifuge was, however, so much better than that of the batch centrifuge that a substantial reduction of the product viscosity was attained in the continuous device, whereas a net increase was observed in the batch machine. Table 6 does not imply that the batch machine is inherently inferior to the continuous centrifuge, as the operating parameters for the former had not been optimized, but only that the continuous centrifuge produced a product that was better than other products that had been found adequate for the process development research to date.

Density studies

The importance of the density difference between the solid particles and the liquid is clearly stated in equations 5 and 6. The correlations used in earlier sections of this report circumvented the effect of this variable by treating ash and organic solids with separate correlations and assuming that the density of the liquid and of the two solid components did not vary from experiment to experiment. An estimate of the component densities may be obtained by expanding the assumption of constant component density to include all process streams, feed, product and residue, and applying to each sample the material balance equations:

$$\frac{1}{\rho_m} = \frac{\chi_a}{\rho_a} + \frac{\chi_o}{\rho_o} + \frac{\chi_s}{\rho_s} \dots \dots \dots (8)$$

where ρ is density, χ is the weight fraction, and the subscripts m, a, o, and s refer respectively to mixture (i.e., the actual sample), ash, organic solids and solvent (i.e., the solid-free liquid).

As $\chi_a + \chi_o + \chi_s = 1.0$, equation 8 becomes:

$$\frac{1}{\rho_m} = \left(\frac{1}{\rho_a} - \frac{1}{\rho_s}\right) \chi_a + \left(\frac{1}{\rho_o} - \frac{1}{\rho_s}\right) \chi_o + \frac{1}{\rho_s} \dots \dots \dots (9)$$

in which ρ_m , χ_a , and χ_o are measured values for each sample.

Sixty-four samples from two feed materials were analyzed. The results, in the form of equation 9, were subjected to multiple linear regression to determine the values of ρ_s , ρ_a , and ρ_o , as shown in Table 7.

The significant conclusion to be drawn from Table 7 is the negligible density difference between organic solids and liquid at room temperature. The fact that separation between these two phases does occur in the centrifuge is evidence that the decrease in density that occurs when the temperature is raised is substantially more for the liquid than for the solid. However, it is also quite possible that ash and organic solids do not exist as separate entities but that the actual particles are variable mixtures of the two components. Thus the density of individual particles could fall anywhere between the "ash" and "organic solids" densities of Table 7. The variability in particle density creates a degree of indeterminacy in the calculation of d_c and may also account for the variance in the measured separations, as represented by the scatter in Figures 8, 9 and 10.

TABLE 7.- Densities of coal liquid (ρ_s), ash (ρ_a) and organic particles (ρ_o) computed from the density (ρ_m) (measured at 80° F), ash and organic fractions (χ_a , χ_o) of suspensions by the multiple linear regression equation (9)

Source of data	FB-61A	FB-57
Type of coal	Kentucky	Kentucky
No. of pts.	49	15
Range of χ_m , g/cc	1.074-1.637	1.129-1.534
Range of χ_a	0.008-0.443	0.024-0.370
Range of χ_o	0.009-0.176	0.023-0.170
Std. error of est	0.0086	0.0038
Density, liquid, g/cc	1.08	1.11
Density, ash, g/cc	4.21	4.47
Density, organic solids, g/cc	1.07	1.11

Additional correlations that incorporate this density difference will require accurate measurements made at centrifuge operating temperatures.

Non-Newtonian viscosity

The solids separation correlations and the data analyses that we have used were based on the assumption that the liquid was a Newtonian fluid, i.e., viscosity is a single value for any given fluid at a specified temperature. The viscosity of coal liquefaction products is, however, a much more complex property. In a Newtonian fluid, the viscosity is independent of the shear rate (and, therefore, of the rate of flow), and a graph of shear stress vs. shear rate would be a straight line passing through the origin. A graph of shear stress vs. shear rate for a typical high viscosity coal liquefaction product, Figure 12, is a straight line not passing through the origin. This behavior is known as Bingham plastic. Measurements of shear stress as a function of shear rate were taken for either feed or product for each of the vertical centrifuge runs from CC-9 to CC-22 at several temperatures. Each set of measurements was subjected to a linear regression treatment. The coefficients of the regressions are tabulated in Appendix Table A-7. The high values of the coefficients of determination shown in Table A-7 are evidence that each of the samples is, in fact, a Bingham plastic.

On a shear stress vs. shear rate curve, the effective viscosity is the slope of the line from the origin to the curve at the point representing the shear rate to which the fluid is being subjected. It is apparent that as the shear rate increases (equivalent to an increase in flow rate of the fluid

Figure 12 - Viscous behavior of a heavy coal liquefaction product .

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with respect to a solid particle or a confining wall), the effective viscosity will decrease asymptotically to the slope of the curve, which is known as the plastic viscosity. Conversely, as the shear rate decreases, the effective viscosity will increase until the shear rate is zero, at which point the viscosity is infinite. The finite value of shear stress at zero shear rate (the yield stress, τ_0) signifies that a finite force is required before a relative motion between planes of a liquid or between particle and liquid can be attained.

This finite force, which is equal to the product of yield stress and particle surface, is developed by the centrifugal field acting on the particle. Analytically, this relationship can be expressed as:

$$\tau_0 \pi d^2 = \Delta \rho \frac{\pi d^3}{6} r \omega^2 \dots \dots \dots (10)$$

Rearranging equation 10:

$$d = (6 \tau_0) / (\Delta \rho r \omega^2) \dots \dots \dots (11)$$

"d" is the diameter of the particle that will satisfy the equality of yield stress and centrifugal force. Figure 13 shows that the yield stress for the fluid used in a specific test (experiment CC-11) was about 1.0 dyne/cm². Substituting in equation 11 this value for τ_0 as well as the other operative values ($\Delta \rho = 3.3 \text{ gm/cm}^3$, $r = 7.6 \text{ cm}$, $\omega = 628 \text{ radians/sec}$), we find that ash particles of diameter .006 μm or less will not move through the fluid and hence represent an absolute limit which cannot be separated, even at infinite residence time (i.e., $Q_e = 0$).

Figure 13-Shear stress as a function of shear rate for product CC-II.

Whether this limit has any real significance or is merely of academic interest will depend upon whether the particle size distribution is such that a significant fraction of the solids exist at or below this diameter and whether or not the Ambler cut-off diameter (equation 6) approaches this size.

Aging effects on viscosity

Another problem associated with coal-derived fluids is the change in viscosity with time, temperature, and atmospheric exposure. Such changes can bring into question the validity of the viscosity measurements, as changes may occur in the course of making the measurements with a heated viscometer. A brief investigation was made of the viscosity of samples from a single source. The samples were held for various times at different temperatures and in either air or carbon dioxide atmospheres, before the measurements were made. The time-temperature history of the samples tabulated in Appendix Table A-8 is shown in Figure 14. The results of the study are given in Figure 15, in which the values of the viscosity that were measured at 220°, 240°, and 340° F, are shown as a function of the "Equivalent Age" of the sample at time of measurement; the "Equivalent Age" is the integrated product of time and temperature (the area under the curves of Figure 14). It is apparent from Figure 15 that the viscosity of a sample changes significantly with the length of time required to make the measurement and with the amount of air in contact with the sample. However, high-temperature

Figure 14-Temperature history of viscometer samples.

Figure 15-Effect of time of testing on viscosity measurement.

viscosity is affected considerably less than low-temperature viscosity. This fact leads to several important inferences: (a) values measured at the usual continuous centrifuge operating temperature from which high solids separations are predicted are reasonably accurate; (b) change in residence time in the centrifuge should not change the effective viscosity; (c) the accuracy of viscosity measurements made at the usual temperature of the laboratory Saybolt viscometer may be seriously affected by the operator's technique which is reflected in the time required to make a measurement; (d) the accuracy of extrapolation of the standardized Saybolt measurement to operating conditions is in doubt. Observed differences in the temperature coefficients of the slopes and of the intercepts of correlations such as Figure 12 suggest that the phenomenon is probably due to the viscosity being the sum of two components, a high viscosity component and a low viscosity component with different aging sensitivities.

CONCLUSIONS

A continuous discharge solid bowl centrifuge proved capable of removing more solids from a coal liquefaction product than the batch type of solid bowl centrifuge that had been in use in the same process. The continuous centrifuge removed from 23 to 88 pct. of the ash in the gross liquid product from a coal liquefaction process and from 8 to 73 pct. of the organic benzene insolubles. Material balances with closures better

than 95 pct. indicated that the experimental measurements were dependable.

The variations over the ranges indicated were correlated with temperature, flow, fluid viscosity, and centrifuge geometry by Ambler's parameters (equations 6 and 7 in the text), with the effect of particle size distribution being accommodated by the use of probability coordinates in the ordinate. A major difference between results with the horizontal and the vertical centrifuge was noted, but this was attributed to the observed, but not precisely quantified, differences in the feed to the two machines, rather than to any inherent difference in the equipment.

An increase in temperature, and hence a decrease in viscosity, improved the solids separation at constant flow rate, and decrease in flow rate improved the separation at constant temperature. The relationship was, however, relatively insensitive -- the ash removal decreased from 84 pct. to about 60 pct. and the organic solids from 77 to about 20 pct. when the operating parameter $\mu Q_e / \Sigma$ was increased 50 fold. The separation of the organic solids was poorer and more erratic than that of the mineral matter because of lesser differences between particle density and liquid density and because the size distribution of the organics was inherently wider in any given material and more variable between materials. In general, correlations such as those represented in Figures 9 and 10 will be adequate for scale-up if the feed

materials used in the testing are identical to those used in the scaled-up process.

Changes in the bowl rotation speed were not tried. Changes in the solids conveyor speed had negligible effect on the quality of the liquid product but did have some effect on the dryness of the residue and hence on the yield.

Ancillary phases of this investigation added several interesting conclusions. The coal liquefaction products are non-Newtonian and this sets an absolute limit on attainable solids separation. Density differences between organic solids and the liquid phase may be extremely small, and this may make the degree of organic solids removal much more temperature sensitive than is the mineral matter separation. Time, temperature, and ambient atmosphere alter the viscosity of coal liquefaction products; this affects the validity of moderate-temperature viscosity measurements, but does not appear to affect high-temperature viscosities as much.

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NOMENCLATURE

CLP	Centrifuged liquid product
d	Particle diameter
d_c	Cut-off particle diameter calculated by equation 6
GLP	Gross liquid product (i.e., uncentrifuged product of liquefaction reactor)
L	Centrifuge bowl length
OBI	Organic benzene insolubles
Q_e	Volumetric flow rate of clarified liquid effluent
r	Radial position of particle
r_c	Centrifuge bowl radius
r_d	Centrifuge dam radius
S_A	Ash Separation
S_{OBI}	Organic solids separation
v_s	Radial velocity of solid particle
x_i	Weight fraction of component i
$\Delta\rho$	Density difference between particle and liquid
μ	Viscosity of fluid
ρ	Density of fluid or solid
Σ	Geometric centrifuge parameter
τ_o	Yield stress of fluid
ω	Angular velocity of centrifuge

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APPENDIX

TABLE A-1.- Sources of feed materials for centrifugation studies

Cfge. Expt.	Source Liquefaction Experiment				Coal
	Expt. No.	Reactor Dimension	Pressure psig	Temp ° C	
HC-1 to HC-11	FB-42 R3	1" x 28'	4,000	450	W. Ky., hvAb
CC-2	FB-57	1" x 28'	4,000	450	do.
CC-3,-4	Centrifuge from CC-2	(None)			
CC-5 to -7	Combined centrifuge from CC-2, -3,-4	(None)			
CC-8 to -22	FB-57	1" x 28'	4,000	450	do.
CC-23,-24	FB-61	1" x 28'	4,000	420- 450 ^a	do.
CC-25,-26	FB-61A	1" x 28'	4,000	450	do.
CC-27	FB-62	1" x 28'	4,000	450	W. Va., hvAb

^aRaised from initial 420° C to 450° C after 360 hours on stream

TABLE A-2.- Primary measurements, horizontal centrifuge experiments

Test No.	HC-1	-2	-3	-4	-5	-6	-7	-8	-9	-10	-11
Effluent rate, lb/min	4.13	7.00	15.0	26.6	21.4	16.8	5.00	10.8	11.5	22.1	4.75
rate, lb/hr	250	420	900	1600	1284	1008	300	650	690	1325	285
Temp, ° F	140	139	140	141	200	201	202	203	204	205	205
Bowl RPM	6000	←-----→ 6000									
Differential RPM	30	←-----→ 30							10	10	10
Dam No.	3	←-----→ 3									
Feed: Li-											
quids, %	90.4	-----→									
Ash	6.1	-----→									
Organic											
Insols.	3.5	-----→									
Total											
Insolo.	9.6	-----→									
Sulfur	0.95	-----→									
Specific Gravity	1.099	60° F/60° F -----→									
Viscos-											
ity	32.3	-----→							76.3	-----→	
	SSF								SSU		
	140° F								204° F		
Liquid											
Products:											
Liquids,											
%	93.5	93.4	92.7	92.2	93.5	94.6	95.9	94.9	95.1	93.8	96.2
Ash	3.67	3.59	4.3	4.7	3.3	3.10	2.05	2.8	2.9	3.5	1.4
Organic											
Insols.	2.8	3.0	3.0	3.1	3.2	2.3	2.0	2.3	2.0	2.7	2.4
Total											
Insols.	6.47	6.6	7.3	7.8	6.5	5.4	4.1	5.1	4.9	6.2	3.8
Sulfur	0.79	0.76	0.85	0.81	0.73	0.60	0.48	0.56	0.57	0.69	0.42
Specific Gravity	**	**	1.078	**	**	**	1.061	**	**	**	**
Viscosity	**	**	31.6	**	**	**	76.1	**	**	**	**
			SSF				SSU				
			140° F				204° F				
Residues:		←-----→									
Liquids,											
%	49.3		48.3	42.7	43.3	41.7	42.8	41.5	40.4	39.8	40.5
Ash	37.8		36.3	43.2	43.0	43.7	44.1	44.4	43.1	45.0	44.1
Organic											
Insols.	12.9		15.4	14.5	13.7	12.6	13.1	14.1	16.5	15.2	15.4
Total											
Insols.	50.7		51.7	57.7	56.7	58.3	57.2	58.5	59.6	60.2	59.5
Sulfur	3.15		3.2	3.6	3.9	4.5	4.6	4.8	4.5	4.6	5.0

TABLE A-3.- Operating parameters of the vertical centrifuge experiments

<u>Expt.</u> <u>No.</u>	<u>Dam</u> <u>No.</u>	<u>Bowl speed,</u> <u>rpm</u>	<u>Diff. conveyor</u> <u>speed, rpm</u>	<u>Press.</u> <u>psig</u>	<u>Temp</u> <u>° F</u>	<u>Flow</u> <u>gpm</u>
CC-						
2	2	6,000	64	18	-	0.70
3	do.	do.	do.	15	-	0.06
4	do.	do.	do.	17	-	0.25
5	4	do.	do.	24	340	6.00
6	do.	do.	do.	24	325	1.50
7	do.	do.	do.	24	300	1.50
8	do.	do.	do.	25	260	0.06
9	do.	do.	do.	25	305	0.48
10	do.	do.	do.	25	290	0.27
11	do.	do.	do.	18	315	0.31
12	do.	do.	do.	18	330	0.71
13	do.	do.	do.	28	330	0.42
14	do.	do.	do.	28	320	0.18
15	2	do.	do.	20	320	0.77
16	do.	do.	do.	20	330	1.18
17	do.	do.	do.	25	300	0.13
18	do.	do.	do.	25	340	0.39
19	do.	do.	do.	25	315	0.52
20	do.	do.	do.	25	290	0.54
21	do.	do.	do.	26	263	0.33
22	do.	do.	do.	28	269	0.16
23-1	do.	do.	6.3	28	300	0.15
23-2	do.	do.	do.	27	299	0.10
23-3	do.	do.	do.	27	299	0.09
23-4	do.	do.	do.	27	309	0.14
23-5	do.	do.	do.	25	300	0.15
23-6	do.	do.	do.	25	316	0.16
23-7	do.	do.	do.	25	289	0.19
24-1	do.	do.	do.	27	321	0.37
24-2	do.	do.	do.	27	309	0.15
24-3	do.	do.	do.	27	321	0.21
24-4	do.	do.	do.	27	329	0.16
25-1	do.	do.	do.	23	269	0.05
25-2	do.	do.	do.	24	282	0.17
25-3	do.	do.	do.	24	290	0.24
25-4	do.	do.	do.	23	291	0.24
25-5	do.	do.	do.	24	290	0.30
26-1	do.	do.	do.	21	280	-
26-2	do.	do.	do.	21	294	0.24
26-3	do.	do.	do.	21	299	0.29
26-4	do.	do.	do.	21	310	0.14
27-1	do.	do.	do.	a	320	0.53
27-2	do.	do.	do.	a	319	0.54
27-3	do.	do.	do.	a	330	0.37
27-4	do.	do.	do.	a	330	0.23

^a Pressure variable due to leaking back pressure regulator

TABLE A-4.- Proximate analyses of vertical centrifuge products

Experiment	S.G. 60°/60°	Viscosity SSF, 180° F	wt. pct.			
			OBI	Asphaltene	Oil	Ash
CC-2		263.0	3.4	29.4	65.1	2.1
3		238.0	3.6	29.8	64.0	2.6
4		283.0	3.8	37.0	56.5	2.7
5	1.120	209.0	2.7	28.6	66.5	2.2
6	1.122	80.6	3.0	31.3	63.7	2.0
7	1.119	84.9	3.0	29.9	64.9	2.2
8	1.097	43.1	2.9	26.0	69.1	2.0
9	1.130	132.0	3.3	33.5	60.7	2.5
10	1.133	159.0	3.7	30.0	63.5	2.8
11	1.119	165.0	2.9	30.6	64.0	2.5
12	1.140	254.0	3.5	33.1	61.2	2.2
13	1.126	215.0	3.3	30.7	63.8	2.2
14	1.134	217.0	3.1	33.7	60.9	2.3
15	1.126	141.0	3.8	32.0	61.5	2.7
16	1.129	173.0	3.1	30.0	64.7	2.2
17	1.129	177.0	3.5	41.6	52.3	2.6
18	1.135	204.0	3.2	43.8	50.9	2.1
19	1.129	188.0	3.6	34.4	59.8	2.2
20	1.135	170.0	3.5	28.7	65.2	2.6
21	1.135	183.0	3.0	31.0	63.5	2.5
22	1.131	160.0	3.5	32.0	62.0	2.5
23-1	1.127	65.3	1.1	24.7	72.2	2.0
23-2	1.101	70.4	1.2	32.3	64.7	1.8
23-3	1.101	66.5	1.1	25.3	72.0	1.6
23-4	1.102	74.0	1.7	28.2	68.0	2.1
23-5	1.099	71.5	1.6	25.8	70.4	2.2
23-6	1.101	69.0	1.6	24.4	72.4	1.6
23-7	1.106	70.5	1.5	25.8	70.4	2.3
24-1	1.098	96.3	1.7	27.2	69.2	1.9
24-2	1.101	102.0	1.6	26.2	70.6	1.6
24-3	1.104	101.0	1.9	26.4	69.5	2.2
24-4	1.102	103.0	1.4	35.9	60.9	1.8
25-1	1.074	19.0	0.9	23.2	75.1	0.8
25-2	1.089	42.0	1.1	22.9	74.9	1.1
25-3	1.095	54.0	1.3	24.4	73.2	1.1
25-4	1.100	60.0	1.5	23.8	73.4	1.3
25-5	1.090	61.0	1.3	24.3	73.1	1.3
26-1	1.103	59.0	1.7	23.3	73.6	1.4
26-2	1.105	71.0	1.5	22.4	74.6	1.5
26-3	1.106	70.0	1.7	27.7	68.9	1.7
26-4	1.110	67.0	1.7	22.9	73.8	1.6
27-1	1.135	197.0	4.4	29.2	64.9	1.5
27-2	1.136	108.0	4.5	35.5	58.3	1.7
27-3	1.144	178.0	4.6	32.1	61.4	1.9
27-4	1.136	133.0	4.3	31.1	62.9	1.7

TABLE A-5.- Elemental analyses of vertical
centrifuge products, wt. pct.

Experiment	C	H	N	S
CC-2	85.7	7.6	1.5	0.7
3	85.0	7.6	1.4	0.8
4	85.3	7.7	1.5	0.7
5	85.4	7.7	1.4	0.7
6	85.4	7.6	1.4	0.7
7	85.8	7.7	1.4	0.6
8	86.1	8.0	1.4	0.7
9	85.3	7.7	1.5	0.8
10	84.7	7.6	1.5	0.8
11	85.5	7.6	1.5	0.8
12	85.8	7.5	1.5	0.7
13	85.7	7.7	1.5	0.7
14	85.7	7.7	1.5	0.8
15	85.3	7.6	1.5	0.8
16	85.7	7.6	1.5	0.7
17	85.4	7.6	1.5	0.8
18	86.1	7.6	1.5	0.7
19	85.7	7.6	1.5	0.7
20	85.5	7.6	1.5	0.8
21	85.3	7.6	1.5	0.8
22	85.2	7.6	1.5	0.8
23-1	85.5	8.3	1.2	0.6
23-2	86.0	8.3	1.2	0.6
23-3	86.1	8.5	1.2	0.5
23-4	85.6	8.4	1.1	0.6
23-5	85.5	8.4	1.2	0.6
23-6	85.9	8.5	1.1	0.5
23-7	85.3	8.4	1.2	0.6
24-1	85.8	8.5	1.1	0.6
24-2	86.1	8.5	1.1	0.6
24-3	85.8	8.4	1.1	0.7
24-4	85.6	8.5	1.1	0.7
25-1	85.9	8.4	1.2	0.4
25-2	86.3	8.4	1.3	0.5
25-3	85.7	8.2	1.3	0.5
25-4	85.5	8.2	1.3	0.6
25-5	86.6	8.4	1.3	0.5
26-1	85.8	8.1	1.3	0.6
26-2	85.4	8.1	1.3	0.5
26-3	85.7	8.0	1.3	0.7
26-4	85.4	8.1	1.3	0.6
27-1	86.8	7.4	1.4	0.8
27-2	86.3	7.3	-	0.8
27-3	85.7	7.2	1.4	0.9
27-4	86.4	7.4	1.3	0.7

TABLE A-6.- Analyses of residue streams from vertical centrifugation

Expt. No.	S.G. 60°/60° F	Analyses, pct.							
		Solvent Analysis				Ultimate Analysis			
		OBI	Asphaltene	Oil	Ash	Carbon	Hydrogen	Nitrogen	Sulfur
CC-									
2	-	24.4	6.8	26.5	42.3	46.8	3.8	0.1	6.8
3,4 ^a	-	22.4	8.4	25.6	43.7	47.3	3.8	0.6	6.1
5,6,7 ^a	-	21.8	9.1	26.5	42.6	48.9	4.1	0.8	6.3
8,9,10 ^a	1.459	15.1	14.0	33.2	32.7	56.1	4.9	0.9	5.7
11,12,13,14 ^a	1.454	13.8	15.7	40.2	30.3	58.6	5.0	1.0	5.0
15,16 ^a	1.498	15.2	12.7	38.6	33.5	55.6	4.9	0.9	5.4
17,18,19 ^a	1.475	16.0	11.8	38.3	33.9	55.3	4.8	0.9	5.6
20,21,22 ^a	1.534	17.0	12.6	33.4	37.0	52.4	4.5	0.8	6.1
23	1.511	17.5	8.6	30.1	43.8	45.6	4.2	0.6	7.3
24	1.361	16.7	10.2	32.1	41.0	48.7	4.5	0.7	6.8
25	1.621	17.6	7.8	30.4	44.2	45.4	4.2	0.6	7.5
26	1.592	16.2	9.3	33.4	41.2	49.2	4.3	0.7	7.0
27	1.540	17.8	11.3	34.4	36.5	52.5	4.4	0.8	6.2

^a Residues pooled for indicated experiments.

TABLE A-7. Summary of linear regression coefficients τ_0 and μ_p ,
and coefficients of determination R^2 ,
for shear stress vs. shear rate measurements

Sample	Temperature ° F	μ_p , cP	τ_0 , dynes/cm ²	R^2
Feed CC-9	305	14.1	1.22	0.9977
Prod CC-10	290	16.4	3.40	.9986
Feed CC-11	295	13.1	1.77	.9989
do.	315	10.0	1.97	1.0000
do.	335	9.1	1.98	0.9992
Feed CC-13	310	12.4	1.81	.9998
do.	330	10.2	1.18	.9903
do.	350	8.0	1.39	.9988
Feed CC-14	320	17.0	1.58	-
Prod CC-15	320	11.3	1.11	.9994
Prod CC-16	330	8.8	2.75	.9992
Prod CC-17	260	20.6	2.13	.9999
do.	280	14.5	2.03	.9997
do.	300	11.4	1.87	.9997
Prod CC-18	320	11.9	1.15	.9965
do.	340	9.3	0.85	.9989
Prod CC-19	260	33.5	1.70	1.0000
do.	280	21.4	1.79	0.9999
do.	300	16.5	1.07	.9999
Prod CC-20	290	16.1	1.42	.9976
Feed CC-20	290	19.1	1.15	-
Prod CC-21	263	28.4	2.09	-
Feed CC-22	269	26.9	1.74	-

Defining equation:

$$\text{Shear stress} = \text{yield stress} + (\text{plastic viscosity} \times \text{shear rate}).$$

$$\tau = \tau_0 + (\mu_p \times \text{rate}).$$

TABLE A-8.- Effect of age, temperature, and ambient atmosphere
on viscosity measurements for identical samples of
centrifuged liquid product

<u>Sample no.</u>	<u>Age, min.</u>	<u>Measurement temp, ° F</u>	<u>Ambient Atmosphere</u>	<u>μ @ 118.6 sec⁻¹ cp</u>
1	30	220	Air	74.8
1	40	260	do.	28.8
1	55	280	do.	19.6
2	30	180	do.	381.3
2	60	320	do.	13.4
2	115	240	do.	77.5
3	15	300	do.	16.7
3	48	200	do.	125.7
3	118	340	do.	10.9
3	173	220	do.	146.1
4	30	240	do.	40.8
4	55	200	do.	212.3
4	105	340	do.	10.5
5	30	220	CO ₂	65.1
5	55	340	do.	9.9
5	135	220	do.	84.6
5	180	340	do.	10.1
5	245	220	do.	89.7