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DEVELOPMENT OF A GAS-PROMOTED OIL AGGLOMERATION PROCESS

Technical Progress Report

January 1, 1995 - March 31, 1995

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PURPOSE

The overall purpose of this research project is to carry out the preliminary laboratory-scale development of a gas-promoted, oil agglomeration process for cleaning coal using model mixing systems.

INTRODUCTION

A semi-empirical mathematical model was proposed last quarter for representing and analyzing the kinetics of oil agglomeration of coal particles suspended in water (ref. 1). The model was used to analyze the results of a number of oil agglomeration experiments which explored the effects of different initial particle concentrations and different amounts of air on the rate of agglomeration. It was found that in most cases the mathematical model fit the data for an individual run quite well. Furthermore, it was observed that the rate of agglomeration was approximately a first order function of particle concentration for the conditions employed and that an increase in the volume of undissolved air from 0.7 to 1.8% based on the total volume of the system produced a noticeable increase in the rate of agglomeration.

During the most recent quarter a number of additional experiments were conducted to study the effects of agitator speed and oil dosage on the rate of agglomeration. Again the data were analyzed by fitting the model to the data and the results are discussed below. In

addition, further consideration was given to the study of oil agglomeration of concentrated particle suspensions. A promising technique reported previously (ref. 2) involves monitoring changes in agitator torque during oil agglomeration. To lay the groundwork for the application of this technique, a number of measurements of agitator torque were made to determine the effects of particle size on agitator torque at different speeds.

WORK PERFORMED

Agglomeration Kinetics of Dilute Suspensions

The apparatus, experimental technique, and materials described previously (ref. 1) were used for measuring the rate of agglomeration of coal particles suspended in water. A mixing tank having a diameter of 15.24 cm (6.00 in.) and holding 2870 cm³ (0.101 ft³) was used for conducting agglomeration. The contents of the tank were agitated by a 5.08 cm (2.00 in.) diameter Rushton-type turbine driven by a variable speed motor equipped with an instrument which indicated both motor speed and torque. In addition, the system was equipped to provide a continuous indication of the turbidity of the coal particle suspension undergoing agglomeration.

Coal from the Pittsburgh No. 8 Seam in Belmont County, Ohio, was utilized for agglomeration tests. The coal had an ash content of 27-28% and sulfur content of 5%. The coal was prepared by crushing and then grinding in a stirred ball mill as a slurry. The particle size distribution of the material was measured by automatic image analysis, and the weight mean particle size was 14.38 μm . The ground material was partially dewatered and stored as a wet paste until needed.

To prepare for an agglomeration test, a small amount of the paste was weighed out and mixed with deionized water. The resulting slurry was transferred to the mixing tank which was then filled completely by adding more water. The agitator was operated to eliminate any remaining pockets of air. Some tests were conducted without any visible air present while other tests were conducted with a measured volume of air present. After conditioning the slurry, a measured amount of either heptane or hexadecane was introduced with a syringe. The progress of agglomeration was monitored subsequently by observing changes in the turbidity of the suspension.

The results of each test were analyzed to determine the rate of agglomeration throughout the test. This required determining the rate of change of turbidity with respect to time ($d\tau/dt$) and using the value in the following expression to calculate the rate of change of particle concentration (dN/dt).

$$\frac{dN}{dt} = \frac{3 N_o}{\tau_o^3} \tau^2 \frac{d\tau}{dt} \quad (1)$$

The initial measured turbidity of the suspension (τ_o) and the initial particle number concentration (N_o) were also required. The value of N_o was estimated by employing the relation,

$$N_o = \frac{6 m}{\rho \pi d^3 V} \quad (2)$$

where m is the total mass of coal, d the mean particle diameter, ρ the density of a single particle, and V the total volume of the suspension. This relation is based on the assumption that the particles are uniform spheres of the same size.

In order to determine the rate of change of turbidity, a tenth order polynomial equation was fitted to the turbidity data for each test. The equation was then differentiated with respect to time to obtain an expression which related the rate of change of turbidity to time throughout the test.

The rate of agglomeration was assumed to be equal to the rate of change of particle concentration given by equation 1. This rate was used to test the mathematical model shown below which had been proposed to represent the agglomeration kinetics.

$$-\frac{dN}{dt} = K N^a \quad (3)$$

For testing this model, the particle concentration N also had to be known through time. For a suspension of uniform particles which agglomerate equally well, the value of N at any time is related to the corresponding turbidity τ by the following equation:

$$N = N_o (\tau/\tau_o)^3 \quad (4)$$

Since the Pittsburgh coal contained a relatively large concentration of ash-forming mineral particles which were not likely to agglomerate, the preceding relation was modified to reflect the weight concentration of ash-forming material X_a present initially. The resulting expression is given below

$$N = (1-X_a) N_o [(\tau-X_a\tau_o) / (1-X_a)\tau_o]^3 \quad (5)$$

Values of dN/dt given by equation 1 were used together with values of N given by equation 5 to test equation 3 expressed in the following form:

$$\ln\left(-\frac{dN}{dt}\right) = a \ln N + \ln K \quad (6)$$

Linear regression analysis was used to determine values of "a" and K for each test or run. The results for a series of runs made with different experimental conditions are shown in Table 1.

In the first set of runs (No. 24 to 33), 20 ml of air was introduced before each run to promote agglomeration, and 20 v/w% heptane was used as an agglomerant. Agitator speed was varied among runs to determine its effect on the rate of agglomeration. For agitator speeds between 1500 and 2100 rpm, the kinetic order was nearly 1.0 in each case, and the average value was 1.02 for the four runs in this speed range. For the two runs made at 1000 and 1200 rpm, respectively, the kinetic order was much higher. Figure 1 shows the results of fitting equation 1 to the data collected in selected runs. It can be seen that for any given particle concentration the rate of agglomeration increased with increasing agitator speed. If lines representing the runs made with intermediate agitator speeds had been plotted, they would have fallen between the lines shown in the same order as the speed.

In the second set of runs (No. 42 to 48), no air was introduced to promote agglomeration, and 20 v/w% hexadecane was used as an agglomerant. Again agitator speed was varied to determine its effect on the rate of agglomeration. The results were somewhat inconsistent in as much as the kinetic order was approximately 1 at the two lower agitator speeds (1500 and 1700 rpm) and 1.6 at the two higher agitator speeds (1900 and 2100 rpm). The results for the runs made at the three highest agitator speeds are indicated by Figure 2. At the highest particle concentration corresponding to the start of any given run, the initial rate of agglomeration varied among runs in direct proportion to agitator speed. However, at the higher agitator speeds the rate of agglomeration decreased more rapidly with declining

Table 1. Agglomeration runs made with finely ground Pittsburgh No. 8 coal.

Run No.	Coal wt., g	Oil			Air, ml	Speed, rpm	N_o $10^{-6}/\text{cm}^3$	τ_o ml	Order "a"	ln K
		Type	ml	v/w%						
24	2.68	C_7H_{16}	0.55	20	20	1500	0.462	0.260	1.086	1.699
29	2.68	C_7H_{16}	0.55	20	20	1000	0.462	0.259	9.792	-110.2
30	2.68	C_7H_{16}	0.55	20	20	1200	0.462	0.268	3.337	-28.33
31	2.68	C_7H_{16}	0.55	20	20	1700	0.462	0.268	0.982	3.564
32	2.68	C_7H_{16}	0.55	20	20	1900	0.462	0.271	1.063	3.298
33	2.68	C_7H_{16}	0.55	20	20	2100	0.462	0.276	0.942	5.073
42	2.68	$C_{16}H_{34}$	0.55	20	0	1500	0.462	0.279	0.894	3.074
46	2.68	$C_{16}H_{34}$	0.55	20	0	1700	0.462	0.275	1.009	1.921
47	2.68	$C_{16}H_{34}$	0.55	20	0	1900	0.462	0.277	1.604	-4.926
48	2.68	$C_{16}H_{34}$	0.55	20	0	2100	0.462	0.279	1.638	-4.898
50	2.68	$C_{16}H_{34}$	1.00	37	0	1500	0.462	0.277	1.857	-8.292
51	2.68	$C_{16}H_{34}$	1.50	56	0	1500	0.462	0.276	1.676	-5.561
52	2.68	$C_{16}H_{34}$	2.00	75	0	1500	0.462	0.277	1.504	-3.177
67	1.41	C_7H_{16}	0.28	20	0	1400	0.243	0.133	1.031	1.613
68	1.41	C_7H_{16}	0.28	20	0	1500	0.243	0.136	1.016	1.892
70	1.41	C_7H_{16}	0.28	20	0	1700	0.243	0.140	0.951	2.971
72	1.41	C_7H_{16}	0.28	20	0	1900	0.243	0.139	0.848	4.098
73	1.41	C_7H_{16}	0.28	20	0	2000	0.243	0.178	0.886	4.402
74	1.41	C_7H_{16}	0.28	20	0	2100	0.243	0.141	0.956	3.866

Figure 1. Effect of agitator speed on agglomeration rate for a system containing 0.093 w/w% coal, 0.70 v/v% added air, and 20 v/w% heptane.

Figure 2. Effect of agitator speed on agglomeration rate for a system containing 0.093 w/w% coal, no added air, and 20 v/w% hexadecane.

particle concentration than at lower agitator speeds. Consequently, toward the end of each run, there appeared to be little actual difference in agglomeration rate among runs.

In the third set of runs (No. 50 to 52), no air was introduced to promote agglomeration, and the amount of hexadecane was varied among runs to determine its effect on the rate of agglomeration. An agitator speed of 1500 rpm was used in each run. The results were consistent in as much as the rate of agglomeration increased with increasing hexadecane concentration (see Figure 3). On the other hand, the kinetic order was not consistent with the value obtained previously in run 42 in which a hexadecane level of 20 v/w% was employed. The kinetic order of that run was found to be 0.894, whereas at the higher hexadecane levels employed in the present set, the kinetic order was much higher, and it decreased as the level of hexadecane was increased.

In the fourth set of runs (No. 67 to 74), no air was introduced to promote agglomeration, and 20 v/w% heptane was used as an agglomerant. Again agitator speed was varied among runs to determine its effect on the rate of agglomeration. The initial coal particle concentration was approximately one-half that used in the preceding runs. For a majority of the runs the kinetic order was approximately 1. The results of selected runs are presented in Figure 4. In general, the rate of agglomeration was found to increase with increasing agitator speed. In most cases for a given agitator speed, the rate of agglomeration found in this set with no added air was below that observed in the first set with added air. Consequently, the benefit of having some air present was confirmed again.

Figure 3. Effect of hexadecane concentration on agglomeration rate for a system containing 0.093 w/w% coal and no added air, using an agitator speed of 1500 rpm.

Figure 4. Effect of agitator speed on agglomeration rate for a system containing 0.049 w/w% coal, no added air, and 20 v/w% heptane.

Concentrated Suspensions

It was reported previously (ref. 2) that agitator torque increases noticeably during the agglomeration of a concentrated suspension of coal particles. Presumably the overall increase in particle size due to agglomeration accounts for the increase in agitator torque. In order to verify this theory, a series of measurements of agitator torque was made using concentrated suspensions of either silica sand or Pittsburgh No. 8 coal. The coal was prepared by crushing and then screening to provide three different size fractions for testing. The different size fractions were left in open containers for a week to oxidize and eliminate any tendency for the material to coagulate or agglomerate when suspended in water. Since different size fractions of sand were available, little preparation of this material was required.

The agitator torque measurements were made with the 11.43 cm (4.50 in.) diameter tank described previously (ref. 2). This tank has a height of 11.43 cm (4.50 in.) and a net volume of 1185 cm³ (0.042 ft.³). Agitation was provided by a 6.35 cm (2.50 in.) diameter Rushton-type turbine impeller driven by 93 watt (1/8 hp) controllable speed motor. The impeller was located 1.9 cm (0.75 in.) above the bottom of the tank.

For each series of measurements the tank was filled completely with a particle suspension which consisted of 355.5 g solids and sufficient deionized water to fill the tank. The solids concentration was 28 w/w% for coal and 25 w/w% for sand. Although the water was not degassed, there was no air visible inside of the tank. For each material of a given size the agitator torque was measured at five different speeds which ranged from 1000 to 1800 rpm. In each case the particles appeared to be suspended completely even at the lowest speed.

The results of this series of measurements are shown in Figures 5 and 6 for coal and sand, respectively. It can be seen that for both materials, indicated agitator torque increased greatly with speed, and for both materials the indicated torque increased to a limited extent with particle size at any given speed. For coal the increase in indicated torque between the smallest and largest size fractions was 17% at 1000 rpm and 13% at 1800 rpm. A change of this magnitude during an oil agglomeration test would probably be sufficient for monitoring the progress of agglomeration. However, it must be noted that the torque measurements were made with an uncalibrated instrument which is part of the agitator drive system. When this instrument is calibrated in the future, the more accurate values which are obtained may differ somewhat from those reported here.

PLANS FOR NEXT QUARTER

Further agglomeration tests will be conducted to see whether some of the inconsistencies noted above in determining the effect of various parameters on the agglomeration rate can be eliminated through better control of experimental conditions. Also the possible effect of emulsifying the heptane or hexadecane before it is introduced into the system will be investigated. In addition, further consideration will be given to the role of air in agglomeration.

Figure 5. Effect of coal particle size on uncalibrated agitator torque at different agitator speeds.

Figure 6. Effect of silica particle size on uncalibrated torque at different agitator speeds.

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