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DEVELOPMENT OF A GAS-PROMOTED OIL AGGLOMERATION PROCESS

Quarterly Technical Progress Report

October 1, 1995 - December 31, 1995

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PURPOSE AND OBJECTIVES

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. Specific objectives include determining the nature of the gas promotion mechanism, the effects of hydrodynamic factors and key parameters on process performance, and a suitable basis for size scale-up of the mixing system.

INTRODUCTION

The results of a series of oil agglomeration tests conducted with concentrated particle suspensions of Pittsburgh No. 8 coal were reported previously (ref. 1). In that series the progress of agglomeration during a batch test was observed by monitoring agitator torque and by examining samples of the coal suspension at different stages of agglomeration. This examination was conducted with an optical microscope. The general procedure for conducting an agglomeration test involved adding i-octane to a degassed suspension of coal in water, and after several minutes of conditioning, injecting a small amount of air near the rotating impeller. The agitator torque was observed to undergo various changes after air was introduced and coal particles became incorporated into flocs and then into agglomerates. No flocculation or agglomeration appeared to take place before air was introduced.

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This line of investigation was extended during the past quarter by conducting further experiments with concentrated suspensions of Pittsburgh No. 8 coal. The suspensions were degassed and then conditioned with i-octane. Air was introduced by a different procedure which involved withdrawing a measured volume of the suspension that was replaced by a corresponding volume of air. The progress of agglomeration was monitored even more closely than before by observing changes in agitator torque and by frequent sampling and examination of the suspension with a microscope. A number of experiments were carried out to study the effects of mixing tank size, impeller diameter, and impeller speed on the time required to achieve various stages of flocculation and agglomeration.

WORK PERFORMED

Experimental Methods

Coal from the Pittsburgh No. 8 Seam in Belmont County, Ohio, was used for the agglomeration tests. The coal contained 5.0% sulfur and 28% ash on a dry basis. A large batch of coal was reduced with a double-roll crusher to approximately 2 mm size particles and then divided into smaller portions by riffing. These portions were stored under argon until needed. Sometime before an agglomeration test or series of tests, one or more of the smaller portions was recrushed with the double-roll crusher to pass a 1.4 mm screen. The material was then ground in a stirred ball mill to produce particles in the 1 to 10 μm size range. The slurry from the ball mill was partially dewatered to produce a paste containing 56% water. The coal paste was stored in a refrigerator set at 5°C until used.

For an agglomeration test, the ground coal was suspended in deionized water having a resistivity of 17 megohm-cm, and i-octane having a boiling point of 99-100°C and obtained from Burdick and Jackson Laboratories was used as an agglomerant.

The process of agglomeration was carried out in enclosed, flat-bottom, cylindrical tanks which were fitted with four vertical baffles and an agitator. Three interchangeable tanks were used which differed in size while preserving geometric similitude. The height of each tank was equal to the diameter of the tank, and the width of an individual baffle was equal to 1/12 of the tank diameter. The largest tank had an inside diameter of 15.24 cm (6.00 in.) and a net volume of 2870 cm³ (175 in.³) when fitted with baffles and an agitator. The middle tank had a diameter of 11.43 cm (4.50 in.) and a net volume of 1185 cm³ (72.3 in.³), and the smallest tank a diameter of 7.62 cm (3.00 in.) and a net volume of 346 cm³ (21.1 in.³). The tanks were constructed largely of Plexiglas except for the top and bottom of the largest tank which were made of stainless steel. The top and bottom of each tank was slightly concave to facilitate draining and/or venting of the material.

While in use, an individual tank was fitted with a variable speed agitator that was controlled by a calibrated unit which indicated both shaft speed and torque. A Rushton-type turbine impeller which had six, vertical flat blades mounted on a horizontal disk was attached to the vertical agitator shaft. The center of the impeller was located at the center of the tank. Three impellers which differed in size were used interchangeably. The following impeller diameters were utilized: 6.35 cm (2.50 in.), 5.08 cm (2.00 in.), and 3.65 cm (1.44 in.).

In preparing for an agglomeration test, one of the mixing tanks was partly filled with deionized water, and a measured amount of coal paste was dispersed in the water by stirring

manually. The quantity of slurry was designed to occupy approximately 80% of the tank volume. The tank was sealed and connected to a vacuum pump. A vacuum corresponding to -95 kPa was applied for 20 min. in order to degas the suspension. During this time the tank was alternately shaken and tapped against the laboratory bench to release air bubbles from the slurry. The tank was opened subsequently and a measured amount of i-octane was introduced. The agitator was inserted, and the tank was closed. Next the tank was filled completely with deionized water which had been degassed by the above procedure. Great care was taken to insure that no gas bubbles were present in the system. With the temperature of the system close to room temperature (19°C), the agitator was turned on and operated at a preselected speed. After 5 min. of stirring and conditioning a measured volume of slurry was withdrawn from the tank and a corresponding volume of air was admitted. This operation required about 30 s. For agglomeration tests involving agitator speeds greater than 1800 rpm, conditioning was carried out with an agitator speed of 1800 rpm, and after 4.5 min. of conditioning the speed was increased to the ultimate value. Air was introduced as indicated above and the test was continued. During a test the agitator was operated at a constant speed, and the indicated torque was recorded. The temperature of the system was kept between 19 and 21°C by circulating cooling water through a coil attached to the bottom of the large tank or by surrounding the other tanks with an ice bath. Small samples (1-2 cm³) of the coal suspension were withdrawn from the system at various intervals by using a syringe equipped with a needle having a large bore (1.5 mm). As each sample was removed, a corresponding amount of water was added so that the volume of air in the system remained constant. After a test was completed, the samples

were examined with an optical microscope to determine the general shape and size of particle aggregations present at various stages of agglomeration.

Experimental Conditions and Results

A series of 35 agglomeration tests was carried out to determine the effects of various parameters on the characteristics of a gas-promoted oil agglomeration process. Three sizes of mixing tanks were employed as well as three sizes of impellers. Various agitator speeds ranging from 1100 to 2450 rpm were also used. Different amounts of air ranging from 0 to 18 v/w% and i-octane concentrations of either 20 v/w% or 30 v/w% were utilized. Both the air and oil concentrations were based on the weight of solids supplied to the mixer. Therefore, an oil concentration of 20 v/w% corresponded to 20 ml oil/100 g solids. The solids concentration employed in each test was 30 w/w% based on the weight of water. The specific combination of conditions used for each test is shown in Table 1.

A preliminary analysis of the results revealed a great deal about the nature and basic characteristics of the agglomeration process. Air had to be present in the mixing system for agglomeration to take place. This can be seen from the results of run 35 conducted without air. Figure 1 indicates that the agitator torque decreased more or less steadily throughout the run. Past results showed that particle agglomeration is always accompanied by a marked increase in agitator torque (ref. 1). Furthermore, when the suspension of coal particles and i-octane was examined after 90 min. of agitation, only free particles and microflocs were observed. In sharp contrast to these results were the results of runs made with air present in the system which included all other runs. In every run where air was present, there was evidence of agglomeration. Even in run 32 where the concentration of air was only 2.25%, agglomeration was

Table 1. Experimental conditions used for a series of batch agglomeration tests of Pittsburgh No. 8 coal with i-octane.

Run No.	Tank		Impeller		Solids, w/w%	Oil, v/w%	Air, v/w%
	Dia., cm	Vol., cm ³	Dia., cm	rpm			
1	15.24	2870	6.35	1300	30	20	9
2	15.24	2870	5.08	2400	30	20	9
3	15.24	2870	5.08	2000	30	20	9
4	15.24	2870	5.08	1600	30	20	9
5	15.24	2870	5.08	1300	30	20	9
6	15.24	2870	5.08	1100	30	20	9
7	11.43	1185	3.65	1600	30	20	9
8	7.62	346	5.08	1600	30	20	9
9	7.62	346	3.65	1600	30	20	9
10	15.24	2870	3.65	2400	30	20	9
11	15.24	2870	3.65	2000	30	20	9
12	15.24	2870	3.65	1600	30	20	9
13	7.62	346	3.65	1750	30	20	9
14	11.43	1185	5.08	1080	30	20	9
15	7.62	346	3.65	1675	30	20	9
16	7.62	346	5.08	1080	30	20	9
17	7.62	346	5.08	1150	30	20	9
18	7.62	346	5.08	1300	30	20	9
19	7.62	346	3.65	2000	30	20	9
20	11.43	1185	5.08	2450	30	20	9
21	7.62	346	3.65	1750	30	30	9
22	7.62	346	3.65	1550	30	30	9
23	7.62	346	3.65	2200	30	30	9
24	11.43	1185	5.08	2000	30	30	9
25	11.43	1185	3.65	1850	30	30	9
26	7.62	346	3.65	1750	30	20	18
27	7.62	346	3.65	1750	30	20	4.5
28	7.62	346	3.65	2200	30	20	4.5
29	7.62	346	5.08	1400	30	20	4.5
30	11.43	1185	5.08	2000	30	20	4.5
31	11.43	1185	3.65	2400	30	20	4.5
32	7.62	346	3.65	2200	30	20	2.25
33	7.62	346	3.65	1750	30	20	2.25
34	7.62	346	3.65	2200	30	20	18
35	7.62	346	3.65	1750	30	30	0

Figure 1. Changes in agitator torque in run 35 made without air.

Figure 2. Changes in agitator torque in run 32 made with 2.25% v/w% air.

indicated by a sharp increase in agitator torque which occurred 20 min. after air was introduced (see Figure 2). Agglomeration was confirmed by the presence of agglomerates in a sample taken at the time when the torque was a maximum. The agglomerates ranged from 0.03 to 0.08 mm in diameter.

A curve representing changes in agitator torque as a function of time for a batch agglomeration test usually included several maxima and minima, and it indicated that the process was relatively complex and consisted of several stages. Examples of such curves are presented in Figures 3 to 5. By examining samples of suspensions taken at various stages of agglomeration it was possible to relate the changes in agitator torque to physical changes in the suspensions. For the purpose of discussion, key turning points in agitator torque have been identified by various letters in Figures 3 to 5. The initial conditioning period when oil is dispersed is identified by letter A. During this period and depending on the intensity of agitation, oil droplets usually become covered and stabilized by hydrophobic coal particles and some microflocs are produced. However, the majority of the particles generally remain freely suspended. After air is introduced, there is a drop in agitator torque as the air is dispersed into small bubbles which apparently reduces the viscosity of the suspension, and the torque decreases to a minimum values at point B. As the process continues from point B to point C, aggregates which contain oil and hydrophobic coal particles are drawn to and accumulate on the surface of bubbles because the bubbles are highly hydrophobic. The accumulation of particles at the air/water interface produces larger flocs and flakes which may become detached from the bubbles after reaching a certain size. This process ultimately reduces the concentration of free coal particles and microflocs to a level where there is a marked change in the color of the

Figure 3. Changes in agitator torque in run 1 made with $P/V = 15.4$ W/L.

Figure 4. Changes in agitator torque in run 7 made with $P/V = 5.93$ W/L.

Figure 5. Changes in agitator torque in run 21 made with $P/V = 21.4$ W/L.

suspension from black to grey. The color change becomes very noticeable at point C. Previous investigators have referred to the color change as the "inversion point."

Between point C and point D the agitator torque drops which may be the result of several factors. One possible factor is the release of gas bubbles by the detachment of flocs and flakes. Another possible factor is the formation of a gel-like structure which takes place between C and D. The structure has the appearance of flocks interconnected by gas bubbles. This structure tends to limit circulation of the suspension to a region near the impeller. At point D most of the hydrophobic particles appear to be incorporated into relatively large flocs or loose agglomerates which are converted subsequently into spherical agglomerates between D and E.

Subjecting the agglomerates to further agitation beyond point E increases the sphericity of individual agglomerates and produces a narrower size distribution of agglomerates. Also the

agglomerates seem to become more hydrophobic which could be the result of oil being forced to the surface of the agglomerates. Consequently, under some conditions the agglomerates combine to form larger agglomerates with a framboidal structure. The consolidation of individual agglomerates has little effect on agitator torque as can be seen by the results indicated between E and F in Figure 3. However, as agglomerates combine to form larger agglomerates there is a decrease in agitator torque. This decrease appears beyond point F in Figure 3.

Figures 3 to 5 are illustrative of the results obtained with three mixing systems which differed in size and power input per unit volume. Figure 3 represents the results of a run made with the largest mixing tank and a moderately large power input per unit volume, while Figure 4 represents the results of a run made with the intermediate size tank and a much lower power input per unit volume. Finally, Figure 5 represents the results of a run made with the smallest tank and a larger power input per unit volume. The torque versus time curves are similar up to the point (E) where spherical agglomeration was achieved in each of the three runs. However, it is apparent that the time required to reach the various stages of agglomeration tended to decrease as the power input per unit volume increased. This phenomenon is shown more clearly in Figure 6 where the time intervals between points C, D, or E and the time of air introduction are presented as a function of the initial power input per unit volume based on measured torque and speed.

Up to this time the experimental results have only been partially analyzed. A complete analysis of the results will be undertaken during the coming months.

Figure 6. Correlation of the time intervals between the time of air introduction and agglomeration stages C, D, or E with power input per unit volume.

CONCLUSIONS

An investigation of the phenomena which occur during the oil agglomeration of coal particle suspensions showed that the process of agglomeration involves several steps which can be identified by changes in agitator torque and by application of optical microscopy. During one of these steps, aggregation of hydrophobic particles and microflocs takes place on the surface of gas bubbles dispersed in the suspension with the result that large flocs or flakes are produced which subsequently evolve into agglomerates. The time required to produce spherical agglomerates appears to be a function of the power input per unit volume with the time decreasing as the power input increases.

FUTURE WORK

The experimental results described above will be subjected to a thorough analysis in the near future to relate the time required to produce spherical agglomerates to various system parameters including tank diameter, impeller diameter, and agitator speed. This analysis should lead to an engineering method for size scale up of a gas-promoted agglomeration system.

REFERENCE

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