Development of a Gas-Promoted Oil Agglomeration Process

**Quarterly Report
January 1 - March 31, 1997**

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Work Performed Under Contract No.: DE-FG22-93PC93209

For
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Office of Fossil Energy
Federal Energy Technology Center
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DEVELOPMENT OF A GAS-PROMOTED OIL AGGLOMERATION PROCESS

Quarterly Technical Progress Report

January 1, 1997 - March 31, 1997

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ABSTRACT

Two series of agglomeration tests were conducted as part of an effort to find a suitable basis for size scale-up of the mixing system used for a gas-promoted oil agglomeration process. In the first series of tests the agitator impeller diameter and speed were varied among runs so as to vary impeller tip speed and agitator power independently while keeping other conditions constant. In the second series of tests the mixing tank size and agitator speed were varied while the ratio of tank diameter to impeller diameter were held constant. All tests were conducted with finely ground Pittsburgh No. 8 coal and with i-octane as the agglomerant.

The results of these tests showed that the minimum time t_e required to produce spherical agglomerates was predominantly a function of the agitator power input per unit volume. In addition, the size of the agglomerates produced in a given time was also strongly dependent on power input. At lower power input levels, the mean size rose as power input increased until a point was reached where agglomerate breakage became important and the mean size decreased. The results also showed that the ash content of the agglomerates produced in a given time tended to decrease with increasing power input. On the other hand, the recovery of clean coal on a dry, ash-free basis was not greatly affected by power input.

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EXECUTIVE SUMMARY

The overall purpose of this research 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.

Recent work has focussed on determining a suitable basis for size scale-up of the mixing system. Two series of agglomeration experiments are described in this report. In the first series, the effects of the ratio of mixing tank diameter to agitator impeller diameter and of impeller speed were investigated. In the second series, the effect of increasing the mixing tank size was investigated as well as the effect of changing agitator speed and power input. System performance was characterized by measuring and recording changes in agitator torque, by collecting numerous samples and examining them with a microscope, and by recovering and analyzing the agglomerated product. All of the agglomeration tests were conducted with finely ground Pittsburgh No. 8 coal using i-octane as an agglomerant.

The results of the agglomeration tests showed that the minimum time t_e required to produce spherical agglomerates was primarily a function of the agitator power input per unit volume (P/V). Consequently, t_e decreased rapidly as P/V increased. The effects of other parameters on t_e were largely masked by the overriding influence of P/V .

The properties of the agglomerates produced in a given time were also greatly influenced by P/V . The mean diameter of the agglomerates increased as P/V increased until a level of agitation was reached where breakage of the agglomerates became important. While

the ash content of the agglomerates tended to decrease as P/V increased, the recovery of clean coal was not greatly affected.

INTRODUCTION

Numerous agglomeration tests have been conducted with concentrated suspensions of Pittsburgh No. 8 coal to study the effects of various parameters on system performance. The agglomerant used in all of these tests has been i-octane, and agglomeration has been promoted by introducing small amounts of air into the agitated system. One series of tests was conducted to study the effects of different parameters on the time required to produce compact spherical agglomerates (refs. 1,2). These results indicated that the time depends mainly on the agitator power input per unit volume. Another series of tests was carried out to study the effects of oil concentration, air concentration, agitator speed, and treatment time on agglomerate size, coal recovery, and ash rejection (refs. 2-5). The results showed that agglomerate size increased and coal recovery and ash rejection improved when any one of the following variables was increased: oil concentration, air concentration, or treatment time. When agitator speed increased, the results tended to improve up to a point and then remain constant.

Two additional series of agglomeration tests have been conducted and the results are reported below. The two series were designed to test two of the more widely used rules for scaling up the size of mixing systems. According to one rule, the size should be scaled up by maintaining constant agitator power input per unit volume. According to another rule, the size should be scaled up by maintaining constant impeller tip speed. For the first series of tests, a 15.24 cm diameter mixing tank was employed and the impeller diameter and speed

were varied so that either the tip speed could be held constant while the power input was varied or the power input could be held constant while the tip speed was varied. Although the tip speed was readily controlled, the power input proved difficult to control because it not only varied during the course of an agglomeration test but also depended on the characteristics of the agglomerates produced which could not be predicted with any degree of accuracy.

To circumvent our inability to predict agitator power accurately, a second series of tests was conducted in which mixing tank size and agitator power were varied while the ratio of tank diameter to impeller diameter and initial properties of the coal-oil-water-air mixture were held constant. For each tank size, agitator power was varied by changing agitator speed which also caused impeller tip speed to vary. Therefore, tip speed and power input were not independent.

RESULTS AND DISCUSSION

Agglomeration tests were conducted with Pittsburgh No. 8 Seam coal from Belmont County, Ohio. The coal was prepared by a somewhat different procedure than that used previously. Whereas previously a double roll crusher had been used to grind 5 mm size coal particles, a high-speed impact mill was used to grind the material for the present series of tests. In both cases, the material was ground further in a stirred ball mill, and the resulting suspension was partially dewatered to form a thick paste which was stored in a refrigerator at 5°C until used. Measurement of the particle size distributions by automated image analysis showed that the wet milled material used for the present tests had a projected area mean particle diameter of 3.86 μm with individual particles ranging from 1 to 16.6 μm in diameter.

The corresponding mean particle diameter for the material prepared by the previous grinding procedure was 4.82 μm with individual particles ranging from 1 to 31 μm in diameter.

The equipment and procedure described previously (ref. 1) were used for conducting agglomeration tests. Pure i-octane was used as the agglomerant or “oil” in each test. For the first series of tests, the particle suspensions were degassed first and then dosed with i-octane. After a suspension had been conditioned for 5 min. by running the agitator at the desired test speed, a measured volume of air was introduced to initiate agglomeration. As the test continued, agitator speed was held constant while agitator torque was allowed to vary. During the test the torque was measured and recorded. The mixing tank was cooled to maintain the temperature of the suspension close to room temperature. At frequent intervals, small samples of the suspension were collected and examined with a microscope to observe changes in particle aggregation. At the end of the run a sample of the suspension was collected for determining the size distribution of the agglomerates by image analysis. The remaining suspension was diluted with an equal volume of water and separated with a 250 μm screen. The agglomerated product and tailings were recovered separately and then dried and weighed. The ash content of the product and tailings was determined subsequently.

For the second series of tests, a similar procedure was used except that the suspensions were not degassed by applying a partial vacuum. Therefore, the suspensions contained dissolved air at the outset. After introducing i-octane, the suspensions were conditioned for 5 min. as had been done previously before introducing a measured amount of air. The tests were then conducted as before until the agglomerates were recovered by screening. For this operation the agglomerates were washed into a deep vessel fitted with a screen bottom which was placed in a pail of water. The agglomerated coal tended to float

while the fine unagglomerated mineral particles settled through the 250 μm mesh screen. The separation was enhanced by gently stirring the mixture. Eventually the screen was raised out of the water and the agglomerates were allowed to drain. In previous tests, the agglomerates were washed onto a dry screen which quickly became plugged so that a considerable quantity of mineral particles became trapped.

The conditions employed and the results obtained in the two series of agglomeration tests are presented in Table 1. In the first series of tests, impeller diameter and speed were varied among runs so as to vary impeller tip speed and agitator power independently. All of the runs were made with a 15.24 cm diameter tank using a solids concentration of 30 w/w%, oil concentration of 30 v/w%, and an air concentration of 9 v/w%. The oil concentration and air concentration were based on the weight of coal, i.e., an oil concentration of 30 v/w% corresponded to 30 cm^3 oil/100 g coal. Each run was continued for 25 min. following the introduction of air except for run 95 which was terminated after 15 min. because the agitator motor overheated.

For each run, a record was obtained of the variation in agitator torque with time, as well as an indication of coal recovery and ash rejection. The minimum time (t_e) required to produce spherical agglomerates after air was introduced was determined, and the measured torque at this point was used for estimating the reported agitator power per unit volume (P/V). The final size range of the agglomerates was determined by optical microscopy, and the projected area mean diameter of the agglomerates at the end of each run was determined by automated image analysis. An interesting correlation was observed between the mean

Table 1. Experimental conditions and results of oil agglomeration runs made with Pittsburgh No. 8 coal and i-octane.

Run No.	T, cm.	D, cm.	N, rpm	S, m/s	Solids, w/w %	Oil, v/w%	Air, v/w%	t _e min.	t _t min.	P/V, ^a W/L	Agglom. Size, μ m range	Size, μ m mean	Ash, ^b w/w%	Ash ^c Rej., %	Recov., ^d %
<u>First Series</u>															
90	15.24	5.08	1800	4.79	30	30	9	19	25	12.33	100-200	149	17.16	48.5	88.3
91	15.24	7.62	1063	4.24	30	30	9	6	25	25.33	100-200	195	12.28	63.1	96.8
92	15.24	6.35	1440	4.79	30	30	9	6	25	23.97	100-250	212	13.34	62.9	97.0
93	15.24	5.08	2089	5.55	30	30	9	6	25	18.72	100-200	204	16.93	48.5	94.9
94	15.24	6.35	1440	4.79	30	30	9	6	25	23.60	100-250	212	15.96	50.6	97.8
95	15.24	7.62	1200	4.79	30	30	9	3	15	37.03	100-200	153	13.60	58.5	97.2
<u>Second Series</u>															
100	11.43	3.65	1700	3.25	20	30	9	37	55	6.10	50-170	126	11.69	64.7	95.6
101	15.24	5.08	1300	3.46	20	30	9	51	55	4.60	40-180	123	14.51	54.0	96.6
102	11.43	3.65	2020	3.86	20	30	9	13	55	9.86	100-200	166	8.99	72.4	97.1
103	15.24	5.08	1900	5.05	20	30	9	11	55	13.49	120-220	175	9.33	71.8	96.9
104	15.24	5.08	1550	4.12	20	30	9	18	55	7.14	100-200	156	10.11	67.8	97.1
105	11.43	3.65	2400	4.59	20	30	9	9	55	13.90	110-220	176	8.50	74.2	96.8
106	24.13	7.62	1000	3.99	20	30	9	31	40	5.39	100-200	146	14.26	55.9	96.8
107	24.13	7.62	1150	4.59	20	30	9	15	40	8.05	80-200	147	12.04	63.5	97.0

^aPower input at t_e

^bAsh content of agglomerates

^cAsh rejected to tailings

^dCoal recovery on a dry, ash-free basis

agglomerate diameter and the power input per unit volume (Figure 1). It can be seen that the largest agglomerates were produced with a power input of 25 W/L. Increasing the power input much above this level resulted in a smaller mean size probably because of agglomerate breakage.

The effects of power input on coal recovery, ash rejection, and agglomerate ash content are indicated by Figure 2. It can be seen that coal recovery on a dry, ash-free basis increased gradually with power input until it reached 97% at a power input of 25 W/L. At this point coal recovery leveled off while ash rejection increased sharply, partly due to a drop in product ash content.

The minimum time t_e required to produce spherical agglomerates also correlated strongly with agitator power input per unit volume (Figure 3). Linear regression analysis of the data produced the following relation with a correlation coefficient r of 0.953:

$$t_e = 816(P/V)^{-1.56} \quad (1)$$

This correlation is similar to one reported previously (ref. 2) except for values of the coefficients.

It had been our intention to conduct runs 91 to 94 in such a way that the power input per unit volume would have been the same in all of these runs so that the effect of impeller tip speed could be seen clearly and unambiguously. Although a constant power input was not achieved in this set of runs, it became apparent that any variation in performance caused by changes in tip speed was relatively small.

For the second series of runs, the mixing tank size and agitator speed were varied while the ratio of tank diameter T to impeller diameter D was held constant. Also held

Figure 1. Effect of agitator power input on mean agglomerate diameter in the first series of runs.

Figure 2. Effect of agitator power input on coal recovery and quality for the first series of runs.

Figure 3. Effect of agitator power input on t_e in the first series of runs.

Figure 4. Effect of agitator power input on t_e in the second series of runs.

constant were the solids concentration (20 w/w%), oil concentration (30 v/w%), and air concentration (9 v/w%). Most of the runs were continued for 55 min. after the introduction of air. Runs 106 and 107 were stopped after 40 min. because the agitator motor overheated. As for the previous series of runs, a record was obtained for each run of the variation of agitator torque with time. Again the minimum time t_e required to produce spherical agglomerates correlated strongly with the power input per unit volume (Figure 4). Linear regression analysis of the data produced the following relation with a correlation coefficient of 0.949:

$$t_e = 390(P/V)^{-1.45} \quad (2)$$

This relation is similar to the one produced by the first series of runs except for values of the coefficients. Interestingly, when the results of both series of runs were combined, the correlation represented by Figure 5 and by the following expression were obtained:

$$t_e = 253 (P/V)^{-1.21} \quad (3)$$

The correlation coefficient was 0.962 for the combined series of runs. Since both series of runs were well represented by the same expression, it is apparent that the power input per unit volume is an overriding parameter which tends to mask the effects of less important parameters such as tank size, ratio of tank diameter to impeller diameter, impeller tip speed, or solids concentration. Therefore, many more tests may be needed to discern the effects of secondary parameters accurately.

Figure 6 shows that coal recovery on a dry, ash-free basis was 97% for most of the runs in the second series. Coal recovery appeared to be independent of agitator power input. On the other hand, the ash content of the coal decreased as agitator power per unit volume

Figure 5. Effect of agitator power input on t_e in the combined series of runs.

Figure 6. Effect of agitator power input on coal recovery and quality for the second series of runs.

was raised from 5 to 10 W/L which produced a corresponding increase in the percent of ash rejected to the tailings. A further increase in agitator power seemed to have no effect on the ash content. A comparison these results with those obtained in the first series of tests (Figure 2) shows that the ash content of the product was appreciably lower in the second series. Consequently, more ash was rejected in the second series. This result was probably due to the improved screening procedure used in the second series of tests. However, some difficulty was encountered even with the improved procedure when large amounts of coal had to be recovered as in runs 106 and 107 made with the largest mixing tank. Consequently, the product from these runs was not as clean as the product from runs made under similar conditions in smaller tanks (see Figure 6). The higher ash content of the product produced in runs 106 and 107 could also have been due to the shorter mixing time used in these runs.

CONCLUSIONS

The two series of oil agglomeration tests which were conducted showed that the minimum time t_e required to produce spherical agglomerates was strongly dependent on the agitator power input per unit volume. Due to the overriding influence of the power input, the effects of other parameters such as tank size, ratio of tank diameter to impeller diameter, and impeller tip speed were largely masked.

The size of the agglomerates produced in a given time was also strongly dependent on agitator power input per unit volume. As the power input was raised, the mean agglomerate diameter was observed to rise, pass through a maximum value, and then decline. The decline appeared due to breakage caused by an excessive power input.

Coal recovery was generally large and not strongly dependent on agitator power input. However, the ash content of the agglomerated product decreased to some extent as the power input was raised which resulted in greater ash rejection. An improved screening method for recovering the agglomerates produced a cleaner product with a lower ash content.

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NOMENCLATURE

D	Impeller diameter
N	Agitator speed
P	Agitator power input
S	Impeller tip speed
t_e	Minimum agglomeration time after the introduction of air
t_t	Total treatment time after the introduction of air
T	Tank diameter