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Coagglomeration of coal and limestone to reduce sulphur emissions during combustion†

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The beneficiation of fine coal using the oil agglomeration technique (spherical or liquid phase agglomeration) has been developed at the National Research Council of Canada over a number of years. In this present study the separation of pyrite and other non-carbonaceous materials from the coal particles by selective agglomeration with fuel oil and/or bitumen has been investigated. The oil agglomeration characteristics of freshly mined and agglomerated aged coals were also compared and found to depend on their relative hydrophobicity. An improved response for weakly hydrophobic coals was achieved by treatment with conditioning agents such as sodium oleate. Even after beneficiation, the Nova Scotia coal studied still contained up to 3 wt% of sulphur, and therefore sulphur emission control on combustion is likely to be necessary. It has been demonstrated that SO₂ adsorbents can be incorporated directly into coal or coke agglomerates during liquid phase agglomeration, using bitumen or heavy oil as the binder. Static combustion tests at 850°C were carried out in a muffle furnace and compared with results found for a bench scale fluidized bed unit at the same temperature. In both cases, sulphur capture of over 60% was obtained at a calcium to sulphur molar ratio of 1:1.

(Keywords: beneficiation; sulphur; coagglomeration)

Oily sludges and organic wastes are produced by a number of industries, particularly those related to the recovery and processing of petroleum. Traditional sludge disposal methods, involving concentration by impoundment followed by land filling or land farming, are meeting with increasingly stringent regulation. As a result of these pressures a new approach to oily waste disposal is required.

In some previous work by the present authors¹⁻⁴ it was demonstrated that petroleum coke could be used to remove bitumen from oil sands tailings sludge. The technique involved liquid phase agglomeration in which the oil in the tailings was adsorbed by ground coke particles, which then formed agglomerates up to 1 mm diameter when the coke to oil ratio was about 3:1. These agglomerates could be readily separated from the cleaned water stream by screening. Besides free oil removal, the treatment also resulted in a decrease of total dissolved carbon in the water from about 1000 ppm to less than 100 ppm.

The agglomerates produced during the sludge treatment process appeared to be useful as an ancillary fuel, owing to their higher volatiles content and calorific value compared with the original coke. However, the high sulphur content of the petroleum coke and heavy oil forming the agglomerates makes it imperative to include some form of desulphurization in any combustion system using this material as fuel.

Other workers^{5,6} have demonstrated superior sulphur emission reductions on burning coal co-agglomerated into pellets or briquets with ground limestone or other sulphur sorbents. Our earlier work¹⁻⁴ showed that

limestone could be easily coagglomerated with coke in a single step during sludge treatment, providing that the organic waste being removed was a heavy oil or bitumen. With light oils it was necessary to condition the sorbent with a collector such as sodium oleate to achieve satisfactory coagglomeration. It was also found that some beneficiation, with respect to ash content of the coke, also occurred during the process.

A large number of sites exist throughout the country in which powdered coal (200 mesh) is burnt either for steam generation or for direct heating, as in a cement kiln. In the maritime provinces the coal is usually high in sulphur. It is suggested that these coal burning facilities could also become centres for oily waste treatment. Before being burnt the powdered coal would be used to clean oily waste water shipped, perhaps, from a nearby oil refinery. The fuel value of the separated oil would be recovered in the combustion process and the cleaned water utilized on site as process water.

The presence of iron in coal, possibly as pyrite, has been identified as an inhibitor of sulphur capture, though the mechanism is not understood⁵. This observation implies that beneficiation of high pyrite coal is an appropriate pretreatment, before its combustion, to maximize sulphur capture by a coagglomerated sulphur sorbent. Also, the optimum combustion temperature for sulphur capture is reported^{5,7} to occur at less than 1000°C.

Fluidized bed combustion is normally carried out at 760–930°C to minimize the oxidation of atmospheric nitrogen. On the other hand, stoker furnaces and nozzle type burners usually operate at up to 1350°C. Consequently, the FBC process is most appropriate for the combustion of fuel in the presence of a sulphur

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sorbent. Another possibility would be to add the coagglomerated fuel to the charge of a cement kiln and burn it as it moves through the unit. In this way the very high temperatures in the main burner would be avoided.

Whatever mode of combustion is selected, finer sorbent particles are known to be more efficiently utilized for sulphur capture. However, a major problem with such fine particles is that they are easily entrained in exhaust gases and thereby removed from the system. Incorporation of the sorbent into a pellet overcomes the problem of elutriation loss and allows even finely ground particles to be used.

In the present investigation a high-sulphur Nova Scotia coal has been tested to determine its ability to collect heavy oil. The concomitant beneficiation of the coal during this process was also examined. Finely ground limestone was coagglomerated with the treated coal and combustion tests to determine sulphur capture were carried out in static and fluidized beds at 850°C.

EXPERIMENTAL

The coal used in this study was a bituminous thermal coal from the Prince Mine, Cape Breton Island, Nova Scotia. It was dry pulverized to approximately 80% minus 200 mesh prior to suspension in water for the beneficiation tests. Initial ash content of the coal was 19 wt% while the total sulphur was 3.8 wt% (pyritic sulphur 1.5 wt% and organic plus sulphate sulphur 2.3%). In addition, tests were also carried out on this same coal after it had been beneficiated, using about 2 wt% No. 4 fuel oil, and then stored in a drum for a period of about 2 months. The former coal is here referred to as the 'untreated' coal, while the latter is referred to as 'preagglomerated' or as the 'aged agglomerated coal'.

Coal beneficiation

Typically, 20–25 g of ground untreated coal were dispersed in 100 ml of tap water contained in a Waring Blendor. An appropriate amount of No. 4 fuel oil and/or bitumen was added to this suspension and the contents agitated at 15000 rpm for 30 s. At this stage a conditioning agent was added, if necessary, and the blending speed lowered to 7200 rpm. Blending was continued until discrete agglomerates formed (3–10 min). Coal oil agglomerates were then separated from the aqueous phase by screening. When both fuel oil and bitumen were used for agglomeration, the agglomerates produced by fuel oil were resuspended in water and the required amount of bitumen added in a second agglomeration step. A portion of the agglomerates was ashed to assess the degree of beneficiation while the remainder was used for coagglomeration with limestone.

Coagglomeration with limestone

Freshly beneficiated coal was coagglomerated with limestone using the liquid phase agglomeration procedure described elsewhere^{1–4}. However, the previously beneficiated, aged agglomerated coal could not be coagglomerated using this procedure. This coal was pressure compacted with limestone, using bitumen as a binder. The pellets were prepared in a 1.25 cm die fitted with dual pistons to allow uniform compaction. After loading, the die was placed in a hydraulic press and subjected to a pressure of 7 MPa; final pellet thickness was approximately 0.5 cm.

Combustion tests

Combustion tests were carried out in both a muffle furnace and a bench scale fluidized bed reactor⁸ at 850°C³. The SO₂ concentrations in the combustion gas from the fluidized bed reactor were measured with a Beckman model 865 SO₂ infrared analyser. Tests were also carried out with blank samples containing no sorbent. The percentage retention of SO₂ by the sorbent, called 'sulphur capture', was calculated by comparison of the results from the two tests. Corrections were made for the different sulphur contents of agglomerates used as blanks and those containing sorbent.

For the combustion experiments in a muffle furnace, the sulphur content in the ash residue was determined by X-ray fluorescence spectrometry. The sulphur fixed in the ash during combustion was then expressed as a percentage of the total sulphur in the original sample to give the 'sulphur capture'. In addition, 'calcium utilization' was determined as the amount of calcium fixed as CaSO₄ in the ash as a percentage of the total calcium present in the feed agglomerates.

RESULTS AND DISCUSSION

In our previous work^{1–4}, we have demonstrated that the desulphurization of petroleum cokes can be effectively achieved by coagglomeration with sulphur sorbents. The objective of the present investigation was to assess the effectiveness of this technique for coal samples from Cape Breton, Nova Scotia. Although simultaneous beneficiation and coagglomeration would be more desirable, it is important to optimize the conditions separately. For example, the Athabasca oil sands bitumen, which was found to be an excellent conditioner as well as a binder for sulphur sorbents³, is not very selective in the beneficiation of coals when the higher levels needed to form larger agglomerates are used⁹. By contrast, light refined fuel oils are quite selective in recovering the carbonaceous constituents but extremely poor conditioners and binders for sulphur sorbents^{3,9}. Hence, preliminary experiments involved the beneficiation of coal using both fuel oil and bitumen as an agglomerating aid.

Table 1 lists the results for the beneficiation of untreated coal. Whereas there is a significant reduction in the ash content of the coal, the reduction in total sulphur content is proportionately less. This is due to the high proportion of organic sulphur present in this coal and also due to the relatively hydrophobic nature of pyrite and its generally fine dissemination in the coal matrix¹⁰.

The characteristics of the oil used as a bridging liquid are extremely important for both the beneficiation of coal and the coagglomeration of sulphur sorbents with coal^{3,10}. To be effective, the oil must not only wet both the coal and the sorbent but reject the ash originally associated with coal. Although Athabasca oil sands bitumen is a good wetting agent for both the coal and the sulphur sorbents¹, it is not very effective in ash rejection from coal when compared with the less dense, more refined products such as fuel oils¹⁰. As may be seen from the results in Table 1, the ash content of the coal was reduced to less than 11% with fuel oil compared to 15% with bitumen, using a 10% oil dosage in each case. An initial agglomeration with fuel oil followed by

Table 1 Beneficiation of untreated Prince coal^a

Exp. No.	Agglomerating agent and quantity (g/g coal)	Ash (wt%)	Analysis of agglomerated coal ^b	
			Sulphur	
			Total	Pyritic
1	Feed, blank	19	3.8	1.5
2	No. 4 fuel oil; 0.1	10.8	3.1	0.9
3	No. 4 fuel oil; 0.2	7.4	3.4	1.1
4	Bitumen; 0.1	15.1	3.5	1.3
5	Bitumen; 0.27	8.2	3.3	1.5
6	No. 4 fuel oil, 0.05; bitumen 0.18	5.2	3.4	1.2
7	No. 4 fuel oil, 0.05; bitumen 0.22	5.2	3.4	1.3
8	No. 4 fuel oil, 0.1; bitumen 0.1	6.9	3.5	N.D.
9	No. 4 fuel oil, 0.1; bitumen 0.1	6.3	3.7	N.D.
10	No. 4 fuel oil, 0.1; bitumen 0.1	3.2	3.5	1.2
11	No. 4 fuel oil, 0.1; bitumen 0.1	9.1	3.8	1.3
12	No. 4 fuel oil, 0.1; bitumen 0.15	7.8	3.6	0.7
13	No. 4 fuel oil, 0.1; bitumen 0.17	9.1	3.6	1.5
14	No. 4 fuel oil, 0.2; bitumen 0.15	7.3	4.1	1.5

^a Experiment No. 8 was carried out in the absence of a conditioning agent and No. 9 in the presence of 0.025 M-NaOH. All other experiments were carried out in the presence of 2-5 drops of Accoal 4433, a hydrocarbon surfactant mixture. Recovery of combustibles exceeded 99% in all experiments

^b Dry oil-free basis

additional treatment with bitumen in a second step appears to be more effective in the beneficiation of coal in terms of ash rejection.

Figure 1 is a plot of the ash content of the agglomerates versus total oil-to-coal ratio (No. 4 fuel oil + bitumen = oil). It demonstrates the effect of oil concentration on the quality of agglomerates in terms of ash content. It is obvious that the degree of ash rejection is a function of the amount of agglomerating liquid employed. Optimum ash rejection occurs at an oil level of around 20%. At larger oil dosages there is either no further improvement or the increase in ash rejection is only marginal.

The data in Table 1 and Figure 1 are typical for coal agglomeration using high levels of agglomerating oil, say 10 wt% or greater, and screen recovery of the agglomerates. Under these conditions the pyrite tends to be wetted by oil and becomes incorporated into the agglomerates; also agglomerates recovered by screening tend to trap impurities in the retained aqueous phase unless very high oil levels of agglomerating oil are used. More recent work at NRC has tended to emphasize oil levels in the range 1 to 5 wt% with agglomerate recovery by flotation¹¹. Under these conditions pyrite rejection with this Prince coal was in excess of 80% compared with about 40 or 50% in the tests cited here. It appears that when very small amounts of oil are used it is preferentially adsorbed by the coal rather than the less hydrophobic pyrite which is then rejected from the coal floc-agglomerates when they are recovered by flotation.

The effect of conditioning agents on the quality of coal-oil agglomerates

The possibility of complete separation of ash from

carbonaceous particles depends on the preferential wetting of the mineral matter with water. The surface characteristics of the mineral matter can be modified using conditioning agents. A number of conditioning agents were tested in an attempt either to increase the wettability of mineral matter towards water, or to increase the wettability of coal towards oil. The results are listed in Table 2. It is evident from these results, using relatively high levels of agglomerating oil that the ash content of agglomerates obtained in the presence of various conditioning agents is essentially the same as that of the agglomerates prepared in the absence of these agents. This result is consistent with those published previously¹⁰ for processes using high oil levels to yield capillary-state agglomerates.

Beneficiation of preagglomerated aged coal

Samples from a drum of coal preagglomerated with No. 4 fuel oil and stored under laboratory conditions for 2 months was dispersed in water in the Waring Blender and agitated with bitumen to assess the effect of ageing

Figure 1 Effect of oil dosage on ash rejection (data from Table 1)

Table 2 The effect of conditioning agents on ash rejection from untreated Prince coal^a

Exp. no.	Conditioning agent and concentration	Wt% ash rejection
9	—	71
10	—	73
11	—	63
12	Accoal, 1 drop per 20 g coal	67
13	Accoal, 1 drop per 20 g coal	68
14	Accoal, 2 drop per 20 g coal	67
15	Oleic acid, 1 drop per 20 g coal	70
16	Oleic acid, 1 drop per 20 g coal	69
17	Oleic acid, 1 drop per 20 g coal	65
18	Sodium oleate; 0.05%	70
19	Sodium silicate; 0.02%	68
20	Witco TRS/10-80; 0.05%	65
21	NaOH; 0.025 M	67

^a Fuel oil No. 4 and bitumen (10% each) were used to agglomerate the coal

Table 3 Beneficiation of preagglomerated aged Prince coal^a

Exp. no.	Amount of bitumen (g/g coal)	Analysis of beneficiated coal ^b		
		Ash (wt%)	Sulphur	
			Total	Pyritic
1	Feed, blank	10.9	3.4	0.9
2	0.1	6.6	2.1	0.30
3	0.1	4.1	1.8	0.20
4	0.14	3.9	1.9	0.20

^a The coal was first agglomerated with 10% No. 4 fuel oil and left sitting in the laboratory for about 2 months before it was treated with bitumen. Recovery of combustibles exceeded 99% in all experiments. Experiments No. 2 and 3 were carried out in the presence of 2-3 drops of Accoal and experiment No. 4 was carried out in the presence of 0.05 M-NaOH

^b Dry oil-free basis

on further beneficiation. The results are listed in Table 3. It is obvious from the results that there is a significant improvement in the quality of agglomerates in terms of both the ash and sulphur content. Total sulphur in the agglomerates was about half the amount present in the original feed. This reduction in sulphur content was mostly due to the rejection of pyritic sulphur which was reduced to about 10% of that present in the feed coal. Ash rejection was also somewhat better than that obtained with the freshly agglomerated feed. It therefore appears that the ageing of the preagglomerated coal was beneficial, particularly in the rejection of pyritic sulphur.

Successful removal of pyrite from coal has previously been reported, using a combination of bacterial treatment followed by oil phase agglomeration⁹. Bacteria oxidize the pyrite surface, rendering the particles more hydrophilic. It is possible that the ageing of the preagglomerated coal also selectively oxidizes the pyrite surface thus facilitating its removal because of the hydrophilic nature of the oxidized particles.

Coagglomeration of Prince coal with limestone

Recently, we have demonstrated that the coagglomeration of SO₂ sorbent with coke results in its greater utilization, compared with the systems where the sorbent was added separately². This results from two factors: first, the agglomeration step allows use of much finer sorbent particles in a fluid bed system (it is well known that finer particles will give increased SO₂ adsorption¹²); second, agglomeration provides intimate contact between the sorbent and fuel particles. During burning, sulphur dioxide formed within the agglomerates is not subject to the flushing action of the fluidizing gas and consequently has a greater period of contact with the sorbent compared to the case for physical mixtures. When the agglomerates break down, this advantage is lost.

The earlier results were based on the coagglomeration of petroleum cokes whereas the present study deals with the coagglomeration of Prince coal. Untreated Prince coal was first agglomerated into microagglomerates using No. 4 fuel oil. The microagglomerates were then successfully coagglomerated with the preconditioned limestone using Athabasca oil sands bitumen, which is a good wetting agent for both the hydrophobic coal and the hydrophilic sorbent. The combustion tests on coal-sorbent agglomerates were carried out in both a bench scale fluidized bed apparatus and a muffle furnace

at 850°C. The results are graphically represented in Figure 2.

It is obvious from Figure 2 that the efficiency of sulphur capture is a function of the calcium to sulphur molar ratio in the agglomerates. The fluidized bed combustion results indicate that a linear relationship existed between these two parameters up to a Ca:S molar ratio of at least 1, whereas for the muffle furnace tests the sulphur capture increased regularly up to a Ca:S ratio of 0.7 and then tailed off to a plateau at a ratio beyond 1.

Figure 2 also illustrates the effect of Ca:S molar ratio on the per cent calcium utilization for the muffle furnace tests. Calcium utilization progressively increased with increase in Ca:S molar ratio until it reached a plateau at a Ca:S ratio of about 0.7, when it started to decrease. A similar decline in utilization was not observed for the fluid bed results. This observation is not consistent with our previous findings for cokes³.

Sulphur capture efficiency of limestone in coal-limestone pellets

As has been discussed above, the preagglomerated aged coal was successfully beneficiated with respect to pyrite rejection. However, even after beneficiation this coal contained up to 2 wt% of sulphur and therefore may well require sulphur emission control on combustion. Attempts to coagglomerate this coal with limestone were unsuccessful. It is very likely that the weathering has rendered the surface of the coal more hydrophilic. The presence of hydrophilic limestone will further reduce the oleophilicity of the material which in turn will have a deleterious effect on the oil agglomeration characteristics of the material. The relative oleophilic/hydrophilic nature of the coal surface has been reported to be extremely important to the oil agglomeration process^{10,13,14}. Because of their hydrophobic nature, unoxidized or mildly oxidized coals are readily wetted by the bridging oil and are thus easy to agglomerate with considerably smaller quantities of bridging liquid compared with oxidized and low-rank hydrophilic coals.

Figure 2 Effect of Ca:S ratio on sulphur capture and on Ca utilization. Sulphur capture: Δ , fluidized bed; $\square$, muffle furnace. Ca utilization: $\circ$, muffle furnace

Figure 3 Effect of Ca:S ratio on: ●, sulphur capture; and □, Ca utilization for coal/limestone pellets made from preagglomerated coal during combustion in muffle furnace

As the preagglomerated, weathered coal could not be coagglomerated with limestone, it was compacted instead with varying proportions of limestone using a pellet press. Combustion tests on these pellets were carried out in a muffle furnace at 850°C. The results are shown graphically in *Figure 3*, which is a plot of the Ca:S molar ratio against per cent sulphur capture, as well as per cent Ca utilization. Although there is considerable scatter in the data, there appears to be a linear correlation between SO₂ capture and Ca:S molar ratio. This is contrary to the results for coal-limestone agglomerates where a linear correlation was only observed up to a Ca:S ratio of approximately 0.7; beyond a Ca:S ratio of 1, sulphur capture was essentially constant (see *Figure 2*). This difference could have been a result of the greater strength of the pressure compacted pellets compared with the agglomerates. CO₂ pressure from the calcination of limestone will increase considerably with increased amounts of limestone in the agglomerates/pellets. The agglomerates being weaker than the pellets may not withstand this increased pressure and will break down. This will result in a reduction in contact time between SO₂ and the sorbent within the agglomerates, resulting in a lower Ca utilization at higher limestone loadings for the agglomerates compared with pellets.

CONCLUSIONS

Bituminous coal from Cape Breton, Nova Scotia was successfully beneficiated in terms of ash reduction by oil phase agglomeration. The ash content of the coal was

reduced to approximately 5%. Certain surfactants such as oleic acid, Accoal 4433 (a hydrocarbon surfactant mixture) were beneficial in the agglomeration of coal but had no effect on the extent of beneficiation.

About 40% to 50% pyrite reduction, on an energy basis, was obtained from the beneficiation of fresh coal. Over 80% pyrite was rejected during oil phase agglomeration of a portion of the same preagglomerated, weathered coal.

Microagglomerates of fresh coal were successfully coagglomerated with varying amounts of limestone as a means of reducing sulphur emissions during combustion. Sulphur capture of over 60% was achieved at a calcium to sulphur molar ratio of 1:1.

Surface active agents such as oleic acid and Accoal 4433 facilitated coagglomeration of the coal with sulphur sorbents, but had no effect on the extent of sulphur retention by the sorbent.

Weathered coal could not be coagglomerated with limestone and was dry compacted with varying amounts of limestone. At lower calcium to sulphur molar ratios, combustion behaviour of agglomerates was comparable with that of the pellets. However, at higher limestone loadings, the compacts were more efficient in terms of sulphur retention than the liquid phase agglomerated material.

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