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COAL OXIDATION AND ITS EFFECT
ON OIL AGGLOMERATION

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ABSTRACT

Small particles of high volatile bituminous coal from two different sources were oxidized by air at 150°C for up to 72 hr. As the treatment progressed, samples of coal were recovered and characterized by measuring the heat of immersion of the particles in water and determining the agglomerability of the material with heptane while suspended in water. As oxidation proceeded, the heat of immersion increased and the agglomerability decreased, and a direct relationship between the two was observed.

INTRODUCTION

Previous studies [1-6] have shown that mild surface oxidation of coal by air at temperatures ranging from 100 to 320°C introduces oxygen functional groups which increase the hydrophilicity of the material. Consequently, the heat of immersion of the material in water is increased, and it becomes more difficult to recover the material by froth flotation. Furthermore, Sadowski *et al.* [6] showed that oxidation interfered with the recovery of Upper Freeport coal from Pennsylvania by the agglomeration of an aqueous suspension of coal particles with either heptane or hexadecane.

The present study was undertaken to extend the work of Sadowski *et al.* [6] to other types of coal and to relate changes in the oil agglomeration characteristics of the coal caused by oxidation to changes in the heat of immersion of coal in water. The study also provided an opportunity to relate changes in the turbidity of a suspension undergoing agglomeration to the quantity of agglomerates produced and subsequently recovered on a screen. Since the turbidity of a suspension undergoing agglomeration is determined largely by the concentration of unagglomerated fines and since the concentration of fines decreases during agglomeration, the process of agglomeration can be monitored by measuring the turbidity [7]. Samples of high volatile bituminous coal were oxidized in air at 150°C for

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up to 72 hr. At various intervals small portions were recovered and characterized by measuring both the heat of immersion in water at 25°C and the agglomeration characteristics with heptane.

EXPERIMENTAL

Handpicked samples of high volatile bituminous coal were used for this study. These included a sample from the No. 2 Gas Seam in Raleigh County, West Virginia, with an ash content of 4% and total sulfur content of 0.8% and a sample from the Colchester Seam in Schuyler County, Illinois, with an ash content of 4.5% and total sulfur content of 2.4%. The No. 2 Gas Seam coal was noticeably more hydrophobic than the Colchester coal. The materials were crushed, ground, and screened in the dry state. A high speed impact mill was used for grinding. The product which passed a 200 mesh screen was used for the oxidation experiments. To oxidize the surface of the coal, the material was placed in large aluminum pans and spread in a very thin layer. The pans were placed in a convection oven set at 150°C. Periodically the particles were mixed and sampled with a spinning riffle. The samples were subsequently characterized by measuring the heat of immersion in water and the response to agglomeration with heptane. Before measuring the heat of immersion, the samples were stored for 7-8 days in a desiccator containing a saturated solution of lithium chloride for moisture equilibration. The heat of immersion in deionized water at 25°C was determined by the isoperibol method (an essentially adiabatic method) using a Tronac Model 450 microcalorimeter.

To determine the oil agglomeration characteristics of a sample, 8 g No. 2 Gas Seam coal or 10 g Colchester Seam coal was suspended in 480 ml deionized water which completely filled the modified mixing unit. This unit had been adapted from a 14-speed blender by replacing the open container furnished with the blender with an inverted glass jar to form a closed system [7]. An external loop of tubing conveyed the suspension of particles being treated through an optical cell to measure the turbidity with a photometric dispersion analyzer. The suspension was first conditioned by stirring at 5500 rpm for 2-3 min. Next heptane was introduced in a series of measured increments while stirring at the same rate. After each increment, the turbidity was allowed to stabilize before adding the next increment. When a total of 1.28 ml heptane had been added in the case of No. 2 Gas Seam coal or 1.6 ml in the case of Colchester Seam coal, the agglomerates were recovered on a 100 mesh screen, dried in an oven at 100°C, and weighed to determine the percent recovery based on the initial weight of coal supplied.

RESULTS AND DISCUSSION

The measured heat of immersion in water is shown in Figure 1 for both No. 2 Gas Seam coal and Colchester coal. The heat of immersion of both coals increased as the oxidation treatment time was extended. The effect of the treatment was more pronounced during the first few hours than subsequently. Also the heat of immersion of Colchester coal was considerably

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greater than that of the more hydrophobic No. 2 Gas Seam coal. These results were similar to those observed previously with other types of coal [1-4].

When samples of Colchester coal which had been oxidized for various times were agglomerated with heptane, the results presented in Figure 2 were obtained. For any given sample, except for the one which had been oxidized for 72 hr, the turbidity decreased as more and more heptane was introduced. The decrease in turbidity was evidence of particle agglomeration, and the greater the change in turbidity the larger the degree of agglomeration. Therefore, as more heptane was introduced more of the fine particles were agglomerated. Furthermore, as the oxidation time was extended the samples experienced less agglomeration. Finally after 72 hr of oxidation the particles experienced little or no agglomeration. Although the experimental data are not shown here, similar results were observed with No. 2 Gas Seam coal.

To relate the change in turbidity experienced during agglomeration to the production of agglomerates, the suspension of agglomerates produced with 16 v/w% heptane (1.6 ml heptane/10 g coal) was screened in each case to determine the coal recovery. The results for Colchester coal are presented in Figure 3 as a function of the relative turbidity change produced by agglomeration with the given quantity of heptane. The relative turbidity change is defined as follows:

$$\text{Relative turbidity change (in \%)} = \tau_r' = \left[\frac{\tau_0 - \tau}{\tau_0} \right] 100 \quad (1)$$

where τ_0 represents the initial turbidity of the suspension and τ represents the turbidity after adding 16 v/w% heptane. Figure 3 shows that the agglomeration recovery and relative turbidity change are directly related. Application of linear regression analysis to the data produced an excellent fit with an R^2 of 0.989. The plotted regression line represents the equation,

$$Y = 4.29 + 1.23 \tau_r' \quad (2)$$

where Y is the percent recovery. This result shows that the relative turbidity change is a good indicator of the extent of agglomeration. It has the advantage of being determined more easily and more quickly than the agglomeration recovery.

When the relative turbidity change was plotted against the heat of immersion, the results shown in Figure 4 were obtained for Colchester coal. Although not shown, a similar diagram was obtained when the agglomeration recovery was plotted against the heat of immersion. For No. 2 Gas Seam coal the results differed somewhat from the above in that the agglomeration response of the material was affected only slightly by the first 8 hr of oxidation. However, upon further oxidation the agglomeration response declined in a manner similar to that noted for Colchester coal. Thus, for both types of coal the

agglomeration performance of the material was dependent on the surface properties of the material represented by the heat of immersion.

CONCLUSIONS

Mild surface oxidation of Colchester coal and No. 2 Gas Seam coal increased the heat of immersion of both materials in water and reduced their agglomerability since both the relative turbidity change during agglomeration and the recovery of agglomerates with a given amount of heptane were reduced. As the oxidizing treatment was extended over time, the effects of the treatment became more pronounced, and a good correlation was observed between the relative turbidity change and agglomeration recovery achieved with a given amount of heptane. Also these parameters were observed to correlate well with the heat of immersion.

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Fig. 1. Heat of immersion of oxidized coals in water at 25°C.

Fig. 2. Turbidity of agglomerated suspensions of Colchester coal oxidized for different times.

Fig. 3. Oil agglomeration recovery versus relative turbidity change for Colchester coal oxidized for different times.

Fig. 4. Relative turbidity change versus heat of immersion for Colchester coal oxidized for different times.

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