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MICRO-AGGLOMERATE FLOTATION FOR DEEP CLEANING OF COAL

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by

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OBJECTIVES

The goals of this research program are to demonstrate the technical and economic feasibility of a micro-agglomerate flotation process and to establish the essential criteria for reagent selection and system design and operation.

1. Introduction

The development of practical technologies for the deep cleaning of coal has been seriously hampered by the problems of carrying out efficient coal/mineral separations at the very fine sizes (often finer than 10 μm) needed to achieve adequate liberation of the mineral matter from the coal matrix. It is generally recognized that surface-based separation processes such as froth flotation or selective agglomeration offer considerable potential for such applications but there remain many problems in obtaining the required selectivity with acceptable recovery of combustible matter. In froth flotation, selectivity is substantially reduced at fine sizes due, primarily, to overloading of the froth phase which leads to excessive carryover of water and entrained mineral matter. Oil agglomeration, on the other hand, can provide good selectivity at low levels of oil addition but the agglomerates tend to be too fragile for separation by the screening methods normally used. The addition of larger amounts of oil can yield large, strong agglomerates which are easily separated but the selectivity is reduced and reagent costs can become excessive.

We are investigating the use of a hybrid process - Micro-agglomerate flotation - which is a combination of oil-agglomeration and froth flotation. The basic concept is to use small quantities of oil to promote the formation of dense micro-agglomerates with minimal entrapment of water and mineral particles, and to use froth flotation to

extract these micro-agglomerates from the water/dispersed-mineral phase. Since the floating units are agglomerates (about 30-50 μm in size) rather than individual coal particles (1-10 μm) the problems of froth overload and water/mineral carryover should be significantly alleviated.

Micro-agglomerate flotation has considerable potential for the practical deep cleaning of coal on a commercial scale. In principle, it should be possible to achieve both high selectivity and high yield at reasonable cost. The process requires only conventional, off-the-shelf equipment and reagent usage (oil, surfactants, etc.) should be small. There are, however, complications. The process involves at least five phases: two or more solids (coal and mineral), two liquids (oil and water) and one gas (air). It is necessary to maintain precise control over the chemistry of the liquid phases in order to promote the interfacial reactions and interactions between phases necessary to ensure selectivity. Kinetics as well as thermodynamic factors may be critical in determining overall system response.

The research program has been organized into several specific tasks as indicated below.

Task 1. Interfacial Studies

In order to provide a rational basis for reagent selection, fundamental studies of the various interfaces involved in Micro-Agglomerate Flotation are being conducted. In particular, data are being obtained on:

- liquid/air and liquid/liquid interfacial tensions for aqueous solution/hydrocarbon systems.
- solid/liquid/air and solid/liquid/liquid contact angles for coals and

important minerals (quartz, pyrite, etc.).

Task 2. Emulsification

The emulsification of oil in the presence of fine particles plays a critical role in the development of micro-agglomerate properties and in the rejection of pyritic sulfur and ash during agglomerate formation. The process is being investigated by measurement of emulsion droplet size distributions in agitated vessels of standard design. The effects of:

- coal type (especially hydrophobicity)
- surfactant type and concentration
- hydrodynamics

are of particular concern.

Task 3. Agglomerate Growth and Structure

In order to achieve the degree of selectivity required for effective deep cleaning of fine coal it is desirable to produce agglomerates which are large enough to be separated from the dispersed, refuse material and have sufficiently high density to minimize the inclusion of water and dispersed mineral particles. Studies of size/density relationships for oil-agglomerated fine coal are an important part of the research program. The role of hydrodynamics in agglomerate densification is of particular interest.

Task 4 - Agglomerate Flotation

The final separation of selectively aggregated fine coal particles from mineral matter is to be achieved using froth flotation. Standard procedures for flotation testing

are being used to evaluate the floatability of micro-agglomerates formed under various conditions. Specific studies being carried out include determination of the effects of micro-agglomerate size and structure on the kinetics of flotation and evaluation of the potential for further cleaning of the floated material in multi-stage flotation circuits.

PROJECT STATUS

Task 1. Interfacial studies

Contact angle measurements were continued to determine the effect of the block co-polymeric surfactants that we plan to use later in agglomeration and flotation studies. In the previous quarterly report, contact angle was found to be a function of concentration and surfactant type. Similar measurements were made with other reagents and the maximum in contact angle is plotted as a function of HLB in Figure 1. For the homologous series of block co-polymers that were tested, the contact angle first increased and then decreased with increase in HLB. The data points at an HLB of 15 correspond to reagents of different molecular weights. The effect of HLB is statistically significant because the change in contact angle is greater than the standard deviation, which is plotted as a bar for various data. The results are currently being interpreted. Additional tests are being performed to determine the effect of molecular weight of the surfactant on contact angle.

Task 2. Emulsification

To determine the effect of salt concentration on the kinetics of emulsification, droplet size was measured as a function of time and the results are presented in

Figure 2. The results are shown for 0.1 % and 1.0 % dodecane. The kinetic phenomenon of emulsification is seen more clearly when the results are plotted on logarithmic scales as shown in Figure 3. A straight line plot for 0.1 % dodecane suggested that the droplet size is determined mainly by the dispersion sub-process. In comparison, both the dispersion and coalescence sub-processes are important at higher dodecane concentration. The effect of sodium chloride concentration on kinetics of emulsification is presented in Figures 4 and 5 for 0.1 % and 1.0 % dodecane, respectively. The effect of salt concentration was much more for the higher concentration of dodecane. On the basis of these results we conclude that the effect of salt is primarily on the coalescence sub-process. Since most of the data correspond to a mixed control where both the dispersion and coalescence sub-processes occur, we need to develop suitable models to analyze the results. This aspect of the work is currently in progress.

Task 3. Agglomerate Growth and Structure

The change in agglomerate size distribution with time following oil addition was described in a previous report (Quarterly Progress Report, July 1 - September 30, 1993). The size distributions for different times were generally found to be roughly parallel indicating that the median agglomerate size can be used as an indicator of the extent of agglomeration.

The variation of mean size with time, for different oil additions, is illustrated in Figure 6. Each of the curves has a similar characteristic shape, showing three regions. First is the region of emulsification and adhesion. During this period the oil is being emulsified and the droplets coat the particles. There is little or no

agglomerate growth during this time. The second region is a period of rapid agglomerate growth. During this period, test reproducibility is not good as can be seen from Figure 7 which shows the results of replicate measurements of the median sizes for a fixed oil addition. The greatest scatter in the test data is found during this period. Agglomeration is occurring quickly enough that small time differences in sampling and measurement are crucial. The resulting agglomerates in this region are loosely packed and may be unstable. The growth period is followed by the third region which corresponds to agglomerate compaction and is marked by a decrease in the median sizes which may signify a more dense agglomerate. It is believed that this area is where the entrainment of gangue material occurs. Therefore, flotation will first be tried during the second region, with special care taken so as not to break the agglomerates.

An interesting point about the growth region is illustrated in Figure 8. The Coefficient of Variation (standard deviation of the measured median sizes divided by the mean value) was calculated for each time and percent oil, and was plotted versus time. Each plot goes through a maximum. This maximum signifies the point at which the data scatter was greatest relative to the mean. This maximum shifts to the right for decreasing oil additions. It can be speculated that the period of maximum agglomerate growth occurs at later times for decreasing oil additions. This means that additional time is needed for the smaller amounts of oil to emulsify and be distributed throughout the system.

Further study is underway in which the oil is pre-emulsified before addition into the system so as to eliminate one of the processes during the first region. One objective is to improve the reproducibility of the tests so as to provide more reliable

information on agglomerate growth. More importantly, pre-emulsification of the oil should reduce the conditioning time needed before agglomerate growth begins, especially for the smaller oil additions.

Figure 1. Contact angle of a Pittsburgh seam coal as a function of HLB for two series of block co-polymers. ● - Pluronic P60 series; ○ - Pluronic 100.

Figure 2. The mean size of dodecane droplets (d_{50}) as a function of time.

Figure 3. Kinetics of emulsification of dodecane.

Figure 4. The mean size of dodecane droplets (d_{50}) as a function of time for different concentrations of sodium chloride. Dodecane concentration: 0.1%.

Figure 5. The mean size of dodecane droplets (d_{50}) as a function of time for different concentrations of sodium chloride. Dodecane concentration 1.0%.

Figure 6. Effect of oil addition on the kinetics of agglomerate growth.

Figure 7. Replicate measurements of agglomerate growth kinetics in the presence of 5% oil.

Figure 8. Variability of agglomerate growth with different oil additions.

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