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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 2. Emulsification

An emulsification study was done for Pluronic L-64 in the Waring blender. 25.5 ml of surfactant of varying concentrations was added to 4.50 ml of dodecane and blended for 30 seconds. A sample was taken and analyzed using the Microtrac SRA. Figure 1 shows that the median droplet size decreases with increasing surfactant concentration. These droplets are generally smaller than those found in our earlier studies in the standard mixing tank (see Figure 3 of the January 1 - March 31, 1994 Quarterly Progress Report). Presumably, the difference is due to the very high shear in the blender. It is interesting to note that the rapid fall-off in droplet size occurs close to the CMC for this surfactant (about 10^{-5} M).

Task 3. Agglomerate Growth and Structure

Three different sets of tests were run to show the effect of Pluronic L-64 concentration on agglomerate growth. All tests were run at 1500 RPM and at 0.1% dodecane with respect to the coal.

In the first set of tests, surfactant was added to the standard mixing tank 30 seconds prior to the addition of emulsified oil. Enough surfactant was added to a one weight percent coal slurry to achieve the desired final concentration. 30 seconds later, 0.1 ml of a 15.0% by volume dodecane/distilled water emulsion was added. Samples were taken and measured in the Microtrac SRA. The effects of surfactant concentration on agglomerate growth in this system are shown in Figure 2. Agglomerate size reaches a maximum at 10^{-6} M surfactant. It also should be noticed that at the extreme concentrations, agglomerate size is less than with no surfactant at all. At these concentrations the surfactant may be acting as a wetting agent. These results correspond well with the contact angle study done previously (Figure 1, January

1-March 31, 1994, Quarterly Progress Report).

The second set of tests was similar to the first set except that the oil was not pre-emulsified. Pure oil was added to the coal-water slurry 30 seconds after surfactant addition.

Figure 3 shows that the surfactant increases agglomeration of the coal for concentrations between 10^{-12} and 10^{-6} M. At these concentrations there is little difference in the maximum agglomerate size and all show enhanced agglomeration relative to tests done with no surfactant. Once again, at 10^{-5} M, the surfactant may be acting as a wetting agent.

In the third set, surfactant and oil were emulsified together and then added to the coal slurry in the tank. This was accomplished by first taking 25.5 ml of surfactant at the appropriate concentration to provide the desired surfactant concentration in the tank. This surfactant solution was combined with 4.5 ml dodecane (15.0% dodecane by volume) and blended for 30 seconds. A 0.1 ml sample was taken and transferred to the mixing tank. Agglomerate samples were taken and measured as before. The results of these tests are shown in Figure 4. The maximum agglomerate size peaks at 10^{-8} M. This does not correspond well with previous results which showed maxima in both contact angle and agglomerate size at 10^{-6} M. However, because of the very high concentrations of surfactant needed for the emulsification step, foaming in the emulsifying vessel became a problem and the results obtained at 10^{-6} M may be questionable.

Task 4. Agglomerate Flotation

Based on results obtained to date under Tasks 1-3 of the project, a systematic investigation of the flotation of micro-agglomerates has been initiated.

Figure 1. Effect of surfactant concentration (Pluronic L-64) on the emulsification of dodecane in a Waring Blender.

Figure 2. Effect of surfactant concentration (Pluronic L-64) on agglomerate formation and growth with pre-emulsified oil. (Surfactant added prior to pre-emulsified oil).

Figure 3. Effect of surfactant concentration (Pluronic L-64) on agglomerate formation and growth with straight oil. (Surfactant added prior to direct addition of pure oil).

Figure 4. Effect of surfactant concentration (Pluronic L-64) on agglomerate formation and growth with pre-emulsified oil. (Surfactant added to oil/water mixture prior to pre-emulsification).

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