DOE/PC/92543--T3

doeqtr.793/sc#9

DOE/PC: DE-FG22-92PC92543

MICRO-AGGLOMERATE FLOTATION FOR DEEP CLEANING OF COAL

Quarterly Progress Report: April 1 - June 30, 1993

by

S. Chander
R. Hogg

July 1993

Work Performed Under Contract No.
DE-FG22-92PC92543

For

U.S. Department of Energy
Pittsburgh Energy Technology Center
Pittsburgh, PA

by

Penn State University
University Park, PA 16802

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED *ps*

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

TABLE OF CONTENTS

	<u>Page</u>
OBJECTIVES	1
Introduction	1
Task 1. Interfacial Studies	2
Task 2. Emulsification	3
Task 3. Agglomerate Growth and Structure	3
Task 4. Agglomerate Flotation	4
PROJECT STATUS	4
Task 1. Interfacial Studies	4
Task 2. Emulsification	5
Task 3. Agglomerate Growth and Structure	6

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

Investigations of contact angles on solids at oil-water interfaces are continuing. In order to correlate contact angles with variations in floatability and agglomeration behavior, the interface-partitioning procedure (Wei, Chander, and Hogg, Coal Preparation, 10, 37-45, (1992)) is being used to determine contact angle distributions. An example of the distribution of contact angle for the Pittsburgh Seam coal at the air-water interface is shown in Figure 1. The relatively broad range of angles ($30-55^\circ$) indicates the significant variability associated with a nominally uniform sample of "clean" coal.

The interface-partitioning procedure involves direct observation of a particle at an interface (air-water, oil-water, etc.). Measurements are made of the relative cross-sectional areas immersed in each phase which requires a sharp image of the particle in each phase. In practice, this is made difficult by the presence of a meniscus at the two-phase contact with the cell wall.

For air-water measurements, the meniscus problem can be largely eliminated through the use of plastic cell walls with an air-water contact angle close to 90° . However, this solution becomes impractical for oil-water systems and for any case involving the use of surfactants to modify surface tensions. As a more general solution, we have designed and fabricated a special glass cell in which a line inscribed on the inside of the wall is used to "trap" the interface. The apparent contact angle at the wall can then be adjusted to about 90° simply by the addition or removal of liquid from the lower (normally aqueous) phase.

Task 2. Emulsification

In this part of the project, emulsion droplet size is being measured as a function of ionic strength and type and concentration of surfactant. The effect of ionic strength on the mean droplet size, d_{50} is shown in Figure 2. The measurements were made in the presence of sodium and calcium chloride to determine the effect of the type of salt. The droplet size first decreased and then increased with a gradual increase in ionic strength for both the salts. At low concentrations, $\sim 10^{-4}\text{M}$, the droplet size was the smallest. Sodium chloride produced smaller droplets but the reasons are not clear. Since the mean droplet size is determined by the relative rates of the dispersion and coalescence sub-processes, the salt could effect either of them. We consider that the salt is likely to have a greater influence on the coalescence rate but this hypothesis needs confirmation. Additional studies are being performed to determine the effect of salt and surfactant type.

Task 3. Agglomerate Growth and Structure

A 10 kg batch of the Pittsburgh Seam coal has been ground to a nominal -40 μm using the following procedure:

1. The entire batch was passed through a Holmes pulverizer.
2. The pulverized coal was classified at 40 μm using a Donaldson Acucut classifier.
3. The oversize coal was passed again through the pulverizer and re-classified.
4. This process was repeated until essentially all of the coal was reduced to -40 μm .

The fine coal was stored under argon in 500 g sealed bags. The particle size distribution is shown in Figure 3.

The dry grinding/air classification procedure is very convenient for sample preparation in the laboratory since it permits the use of air classification, the preparation of sufficient material for a complete series of tests and obviates the need for storage of wet coal. In practical (i.e., commercial) applications, however, the coal would normally be ground wet.

A drawback of the dry grinding and storage procedure is that dispersion of the coal in water becomes a critical first step in the evaluation of the agglomeration process. Because of the natural hydrophobicity of the coal, pre-wetting of the material is especially important and, in order to preserve the hydrophobic character, no surfactants or wetting agents can be used. An important consequence of the hydrophobic nature of coal is that the presence of even very small quantities of air can promote agglomeration of the particles. For

studies of the growth of agglomerates following oil addition, therefore, it is vital to ensure that the coal is thoroughly pre-wetted and dispersed.

A standardized procedure has been developed for wetting and dispersion of the coal. The sample of dry coal is first wetted by means of a water spray from a wash bottle. When wetting appears, visually, to be complete, more water is added until the desired solids concentration (normally 1% by weight) is achieved. The resulting suspension is then agitated for 20 minutes at 2000 rpm in the standard, one-liter tank discussed in a previous report. The effectiveness and reproducibility of the wetting-dispersion procedure are evaluated by particle size analysis (Microtrac) on samples taken directly from the tank and subjected only to the required dilution. The measured size distribution is compared to the standard result obtained using wetting agents and dispersants. An example of such a comparison is given in Figure 3. The close agreement among the curves indicates that the procedure is adequate and there is no significant agglomeration of the dispersed coal particles.

Figure 1. Contact angle distribution for Pittsburgh Seam coal.

Figure 2. The mean size of dodecane droplets (d_{50}) as a function of ionic strength for sodium and calcium chlorides.

Figure 3. Size distribution of fine coal sample showing comparison of results obtained using wetting and dispersing agents with those using the standard reagent-free procedure.

**DATE
FILMED**

10 / 25 / 93