DOE/PC/92543--T4-3

doeqtr.993/sc#9

DOE/PC: DE-FG22-92PC92543

MICRO-AGGLOMERATE FLOTATION FOR DEEP CLEANING OF COAL

Quarterly Progress Report: July 1 - September 30, 1993

by

S. Chander
R. Hogg

October 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

93 OCT 12 100 55

SB

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	5

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

In this reporting period, the captive bubble method was used to measure contact angle of coal in the presence of those surfactants which we plan to use in the agglomeration and flotation studies. This method was chosen over the 'interface-partitioning' method, described in the previous report, due to some instrument problems. The results, presented in Figure 1, show that the block co-polymers that we have selected initially could make the surface of coal more hydrophobic. The contact angle was a strong function of concentration and the surfactant type. One of the block co-polymers, L63, seems to be much more effective than the ethoxylated alkyl phenol. In comparison, the reagent L65, was less effective. The HLB is 11.0 for L63, 17.0 for P65, and 13.5 for X-100. These limited data showed an increase in contact angle with decrease in HLB. Further studies are needed to determine the role of HLB on contact angle. Other reagents with varying HLB values are being selected.

Task 2. Emulsification

Studies of the effect of salt type and concentration on the drop size were continued. To confirm the previous findings, additional measurements were made with several other electrolytes and the results are presented in Figure 2 for monovalent ions, and in Figure 3 for divalent ions. These results basically confirm the previous result that mean drop size decreases at low salt concentrations. Even though these results might be explained in terms of the Jones-Ray effect, that is, a decrease in surface tension at low salt concentrations, some controversies exist in the literature with regard to validity of the Jones-Ray effect. In 1938 Langmuir explained the effect by introducing a correction term to the radius of the capillary to account for the thickness of a thin film of water along the capillary wall. Langmuir's explanation seems to be generally accepted (Hunter, 1989. Foundations of Colloid Science, Vol. I, p. 423). To explain these results, it would be necessary to separate the effect of salt on the dispersion and coalescence sub-processes. To separate the effect of these sub-processes we intend to conduct studies of kinetics of emulsification. In addition, interfacial tension (oil-water) measurements will be made at various salt concentrations.

Task 3. Agglomerate Growth and Structure

A systematic investigation of the kinetics of agglomerate growth has been initiated. Experiments are being conducted in a 1.5 liter standard mixing tank as illustrated in Figure 4. In the initial test series, 1200 ml of the coal slurry (1% solids by weight) were agitated at 1500 rpm for 30 minutes to ensure complete

wetting and dispersion. 0.65 ml of oil (dodecane) corresponding to 5 weight percent of the coal present was added rapidly (over about a 2 second period) to initiate agglomeration. Samples were taken using a 6 mm diameter, open glass tube immediately prior to oil addition and at appropriate time intervals thereafter. Particle/agglomerate size distributions were measured using a Microtrac, Standard Range Analyzer (SRA). To minimize changes in agglomerate size distribution during analysis, the normal recirculation system of the Microtrac was not used. Measurements were made, instead, during a single pass of the sample through the instrument.

Some preliminary results from these agglomeration tests are given in Figures 5 and 6. The evolution of the agglomerate size distribution is illustrated in Figure 5. It appears that particles smaller than about 10 μm are eliminated within the first 2.5 minutes of conditioning. The distribution assumes a more-or-less constant form after about 5-7 minutes. The growth kinetics can be seen more clearly in Figure 6 which is a plot of the volume median size versus time. For the conditions used in these tests, the limiting (median) size is about 100 μm . Further investigations on the effects of oil and coal concentration, agitation level, etc., are in progress.

Figure 1. Contact angle of Pittsburgh Seam coal as a function of surfactant concentration. Surfactants: ○ - Pluronic L63; ● - Pluronic P65; ▽ - Triton X-100.

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

Figure 3. The mean size of dodecane droplets (d_{50}) as a function of ionic strength for calcium and magnesium chlorides.

Figure 4. Configuration of the 1.5 liter standard mixing tank (11 cm diameter) used in the agglomerate growth studies.

Figure 5. Change in agglomerate size distribution with time for a 1% by weight fine (Pittsburgh Seam) coal suspension agitated at 1500 rpm in a 1.5 liter tank with oil (dodecane) addition of 5% (based on the coal weight).

Figure 6. Median agglomerate size following oil addition (conditions as in Figure 5).

**DATE
FILMED**

3 / 10 / 94

END