AIM

Association for Information and Image Management

1100 Wayne Avenue, Suite 1100
Silver Spring, Maryland 20910
301/587-8202

Centimeter

Inches

MANUFACTURED TO AIM STANDARDS
BY APPLIED IMAGE, INC.

DOE/PC/92543--T6

qrdoe 494/sc#9

DOE/PC: DE-FG22-92PC92543

MICRO-AGGLOMERATE FLOTATION FOR DEEP CLEANING OF COAL

Quarterly Progress Report: January 1 - March 31, 1994

by

S. Chander
R. Hogg

April 1994

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

RECEIVED
DOE/PC/92543
94 APR 23 PM 2:46
ACQUISITION & INFORMATION DIV.

MASTER

870

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

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	3
 PROJECT STATUS	 4
Task 1. Interfacial Studies	4
Task 2. Emulsification	4
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

Contact angle measurements were continued to determine the effect of surfactant concentration and molecular weight for two homologous series of block co-polymers. The contact angle for selected reagents in Pluronic the 60 and 100 series of surfactants are presented in Figure 1. The effect of hydrophobic and hydrophilic groups on contact angle seems to be complex. For the Pluronic 60 reagents, a gradual increase in concentration first reduced the contact angle. It then increased and eventually decreased again as the concentration approached the CMC. The behavior was somewhat different for the Pluronic 100 series. With these reagents the decrease in contact angle at low concentrations was not observed. Since the contact angle depends on the adsorption of the reagent at solid-liquid, solid-gas and liquid-vapor interfaces, adsorption data are needed to interpret the results. Selected studies of adsorption phenomenon are contemplated.

Task 2. Emulsification

A significantly smaller drop size was obtained when the emulsion is produced in a blender, as can be seen from the results in Figure 2. These tests were

performed to produce an emulsion of known characteristics for use in subsequent agglomeration and flotation studies. The effect of pre-emulsification on agglomeration is discussed in the next section. Smaller droplets were also obtained with the surfactants used in contact angle measurements as can be seen from the results in Figure 3. These and the contact angle results will be used in the design of additional agglomeration and flotation experiments. A theoretical model is also being developed to describe the kinetics of emulsification.

Task 3. Agglomerate Growth and Structure

The results of some experimental studies of the kinetics of agglomerate growth were described in our October 1 - December 31, 1993 Quarterly Progress Report. Based on this work, it was concluded that agglomeration occurs in several stages, the first of which involved emulsification of the oil (in the presence of coal particles) and coal-oil adhesion processes. It was postulated that control over the agglomeration process could be improved through the use of pre-emulsified oil.

A series of tests has been carried out in which a Waring blender was used to emulsify a 15% dodecane/distilled water mixture which was then added, in appropriate amounts, to the coal-water slurry. Studies of this emulsification process indicated that the median droplet size decreased progressively with blending time even after 40 minutes of blending (see Figure 2). In order to ensure a consistent droplet size in the emulsion added to the coal slurry, a procedure was adopted in which each agglomeration test was carried out with freshly prepared emulsion. For the 30 ml blending vessel used in these tests, a blending time of 30 sec, giving a median droplet size of about 27 μm , was adopted as a standard pre-emulsification procedure.

The effects of pre-emulsification of the oil are shown in Figure 4. At low oil concentration (0.1%), pre-emulsification leads to an increase in the initial rate of agglomerate growth. At higher concentration (0.5%), the initial rate is essentially unchanged but subsequent growth becomes less erratic with the pre-emulsified oil. Long-time compaction of the agglomerates does not appear to be affected by pre-emulsification.

The effects of agitation speed during agglomeration are illustrated in Figure 5. As would be expected, increasing speed increases both the initial growth rate and the extent of agglomerate compaction after prolonged agitation. Further analysis, of the actual agglomerate size distributions, is in progress.

Figure 1. Contact angle of a hvA bituminous coal as a function of reagent concentration for various surfactants.

Figure 2. Kinetics of emulsification of dodecane under various conditions.

Figure 3. Kinetics of emulsification of dodecane in the presence of various surfactants.

Figure 4. Effect of oil pre-emulsification on agglomerate development.

Figure 5. Effect of agitation speed on agglomerate formation and growth with pre-emulsified oil.

Figure 5. Effect of agitation speed on agglomerate formation and growth with pre-emulsified oil.

DATE

FILMED

7/13/94

END