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ABSTRACT

Novel chemicals that can be used for increasing the efficiency of fine coal dewatering was developed at Virginia Tech. During the past quarter, Reagent A was tested on three different coal samples in laboratory vacuum filtration tests. These included flotation products from Middle Fork plant, Elkview Mining Company, and CONSOL, Inc.

The tests conducted with the Middle Fork coal sample (100 mesh x 0) showed that cake moisture can be reduced by more than 10% beyond what can be achieved without using dewatering aid. This improvement was achieved at 1 lb/ton of Reagent A and 0.1 inch cake thickness. At 0.5 inches of cake thickness, the improvement was limited to 8% at the same reagent dosage.

The results obtained with the Elkview coal (28 mesh x 0) showed similar advantages in using the novel dewatering aid. Depending on the reagent dosage, cake thickness, drying cycle time and temperature, it was possible to reduce the cake moisture to 12 to 14% range. In addition to achieving lower cake moisture, the use of Reagent A substantially decreased the cake formation time, indicating that the reagent improves the kinetics of dewatering. The test results obtained with CONSOL coal were not as good as with the other coals tested in the present work, which may be attributed to possible oxidation and/or contamination.

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UNIVERSITY OF KENTUCKY'S REPORT

EXECUTIVE SUMMARY

Fine coal constitutes a relatively small portion of a product stream in a coal cleaning plant. However, its processing cost is approximately three times higher than the cost of processing coarse coals. Therefore, many coal companies chose to discard the fines to refuse ponds, causing a loss of profit and creating environmental concerns. This problem can be solved by developing a more efficient fine coal dewatering process, since bulk of the cost associated with processing fine coal is due to dewatering. For this reason, Virginia Tech has developed new chemicals that can increase the efficiency of mechanically dewatering coal fines.

It is the purpose of the present investigation to test the novel dewatering chemicals in laboratory vacuum filtration tests. During the current reporting period, three fine coal products obtained from operating coal preparation plants were tested. These include flotation products from Middle Fork plant, Elkview Mining Company, and CONSOL, Inc. Tests conducted with Reagent A as dewatering aid showed promising results. The cake moisture was reduced by 8 to 13% depending on the reagent dosage, cake thickness, drying cycle time, temperature.

INTRODUCTION

At present, the cost of cleaning and dewatering fine coal (finer than 0.5 mm) is approximately 3 times of those for cleaning coarser coals. For this reason, it is often more economical to discard the fines, if the fine coal constitutes only a small fraction of the product stream. This is typically the case with many coal producers around the world. In the U.S. alone, it is estimated that approximately 2 billion tons of fine coal has been discarded in abandoned ponds, and approximately 500 to 800 million tons of fine coal in active ponds. On a yearly basis, the U.S. coal producers discard approximately 30 to 50 million tons of fine coals to ponds. These problems cause not only losses of profit to coal companies but also environmental concerns.

The main component for the high cost of processing fine coal is the cost of dewatering the clean coal products rather than the costs of cleaning itself. The only process that can satisfactorily remove the surface moisture from fine coal is thermal drying, which is costly. Furthermore, thermal drying is difficult to get permits for new installations as it is perceived to emit pollutants. Mechanical dewatering processes, on the other hand, cannot remove the moisture below 20-35% by weight depending on the coal type and particle size. One way of increasing the extent of moisture reduction is to use surfactants, which will aid mechanical dewatering processes by lowering the surface tension of the water. However, most of the dewatering aids available today can remove the moisture by 1 to 2% beyond what can be achieved without them.

In an effort to solve this long-standing problem, Virginia Tech has recently developed novel dewatering processes. One of the processes entails use of chemicals that

can be added to coal slurry before mechanical dewatering. It is the purpose of this project to test the dewatering chemicals in laboratory filtration tests. The new dewatering chemicals are proprietary in nature at this point; therefore, the authors of this report are not at liberty to disclose their identities.

EXPERIMENTAL

Apparatus

Figure 1 shows the apparatus used for laboratory vacuum filtration tests. A 94-mm diameter buchner funnel with a medium porosity glass frit was used as the filter. Use of a buchner funnel for filtration test is advantageous over the standard vacuum leaf filter test in that the cake thickness can be controlled. The buchner funnel was mounted on a vacuum flask, which in turn was connected to a larger vacuum flask. The larger flask helped stabilize the vacuum pressure during filtration. A coal slurry placed in the buchner funnel was subjected to a vacuum pressure when the valve between the two vacuum flasks was opened. Most of the filtration tests were conducted at 25 inches Hg of initial vacuum pressure (the pressure measured before the valve was open), which decreased to 22-23 inches Hg at the end of a test.

Procedure

In most cases, coal samples were received in 5-gallon buckets as slurry. A coal sample contained in a bucket was agitated by means of a dynamic mixer to homogenize the slurry. A known volume of the slurry was removed from the bucket while the slurry was being agitated. The slurry was poured into a 250-ml flask, to which a known volume of solution

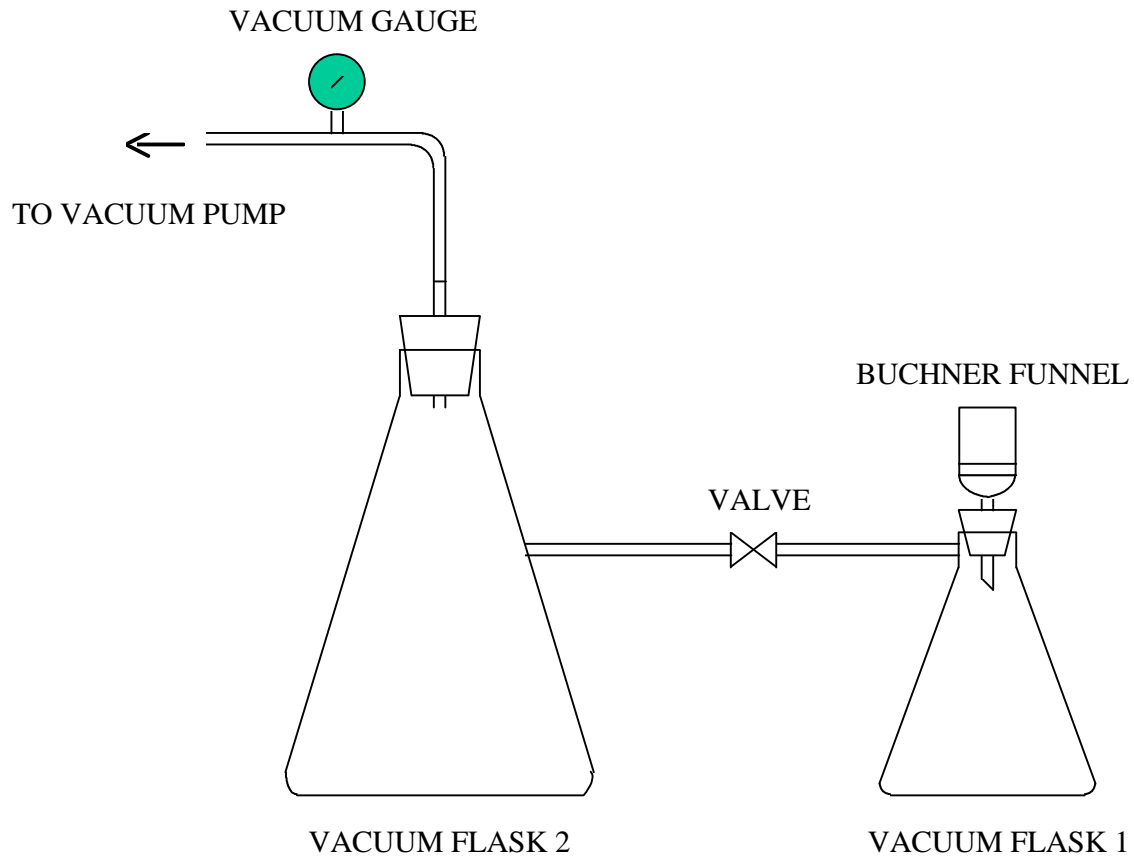


Figure 1. Experimental setup for laboratory vacuum filtration tests

containing dewatering aid was added by means of a Microliter syringe. The flask was subjected to hand shaking for 1 minute to allow for the reagent to adsorb on the surface of coal particles. The coal slurry conditioned in this manner was then transferred to the buchner funnel. Filtration test was commenced when the slurry was subjected to a vacuum pressure by opening the valve between the two flasks. Bulk of the water was quickly removed, and a cake was formed on the glass frit of the buchner funnel. After the cake was formed, the vacuum pressure was kept on for a desired period of time to further drain the water from the cake. After the drying cycle time, approximately 10 grams of the filter cake was removed from the funnel, weighed, and dried for 12 hours at

115 °F. The coal sample was weighed again after the drying, and the moisture content was calculated from the difference between the dry and wet weights.

Coal Sample

Three different coal samples were used for the filtration tests. All of the samples were flotation products from operating coal companies:

- Elkview Mining Co., British Columbia, Canada,
- Consol Inc., Pennsylvania, USA,
- Pittston, Coal Management Co., Virginia.

The first two were run-of-mine coals (28 mesh x 0), while the last was a pond fine (100 mesh x 0) from the Middle Fork coal preparation plant. The coal sample received from Consol Inc. was a Pittsburgh No. 8 seam coal.

Experimental Results

Figure 2 shows the results of the filtration tests conducted on the Middle Fork coal sample (100 mesh x 0) as a function of drying cycle time. The tests were conducted with and without dewatering aid (Reagent A) for comparison. The cake thickness was approximately 0.1 inches in all tests. The tests conducted using 1 lb/ton of Reagent A produced filter cake with approximately 10% lower moisture than without dewatering aid. After 1 minute drying cycle time, the cake moisture was 25.4 and 15.5% in the absence and presence of dewatering aid, respectively. When the drying cycle time was increased to 5 minutes, the cake moisture was further reduced to 13.1% in the presence of dewatering aid. Increase in drying cycle time beyond 5 minutes did not make a significant difference

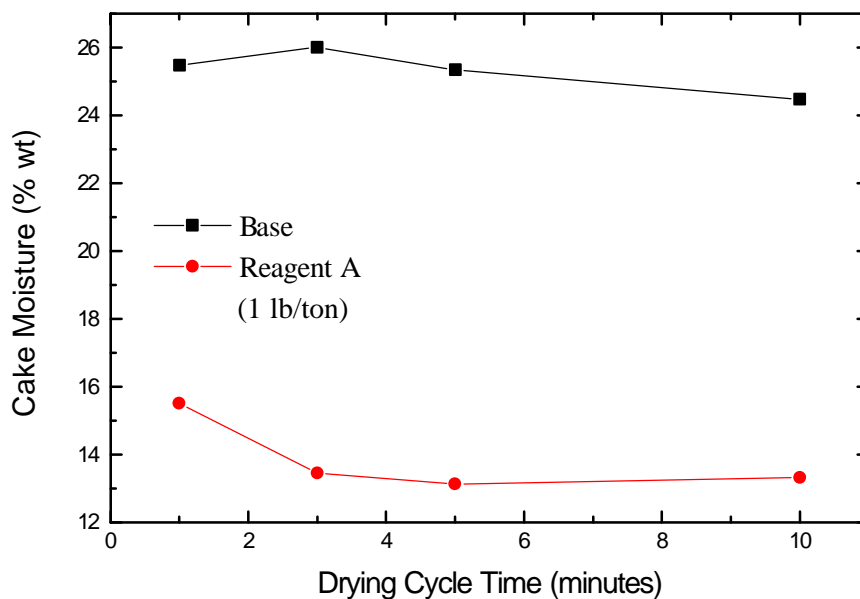


Figure 2. Effects of drying cycle time on the cake moisture. Filtration tests were conducted on a Microcel flotation product from the Middle Fork coal preparation plant with and without dewatering aid (1 lb/ton of Reagent A). The cake thickness was approximately 0.1 inches.

in cake moisture. In the absence of dewatering aid, however, drying cycle time did not show a significant difference.

Next series of filtration tests were conducted by changing the cake thickness. The tests were conducted on the flotation product (100 mesh x 0) from the Middle Fork coal preparation plant using 1 lb/ton of Reagent A. For the purpose of comparison, control tests were conducted without using dewatering aid. The drying cycle time was fixed at 5 minutes in all tests. The cake thickness was varied by changing the volume of the slurry to be filtered in the range of 100 to 500 ml. The results are shown in Figure 3. As expected, the cake moisture increased with increasing cake thickness. As the cake thickness increased from 0.1 to 0.5 inches, the cake moisture increased from 13.1 to 20.5%,

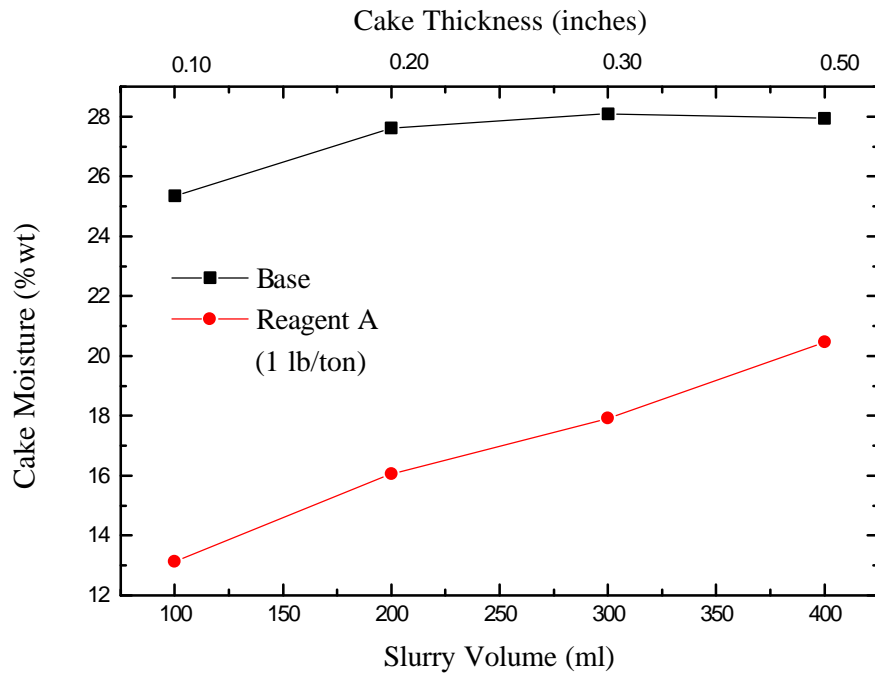


Figure 3. Effects of cake thickness on the cake moisture. Filtration tests were conducted on the flotation products (100 mesh x 0) obtained at the Middle Fork preparation plant with and without dewatering aid (1 lb/ton of Reagent A). All tests were conducted at 5 minutes of drying cycle time.

respectively. Thus, the advantage of using the dewatering aid diminished considerably at higher cake thicknesses. However, the cake moisture of the control tests also increased with increasing cake thickness. Therefore, the advantage of using Reagent A was evident even at higher cake thicknesses. At 0.5 inches of cake thickness, for example, the cake moisture obtained at 1 lb/ton of Reagent A was approximately 8% lower than the control tests. This difference may increase at higher dosages of Reagent A.

Figure 4 shows the results of the filtration tests conducted on the flotation products from Middle Fork using varying amounts of Reagent A. All of the tests were conducted with 200 ml of slurry, which gave approximately 0.2 inches of cake thickness.

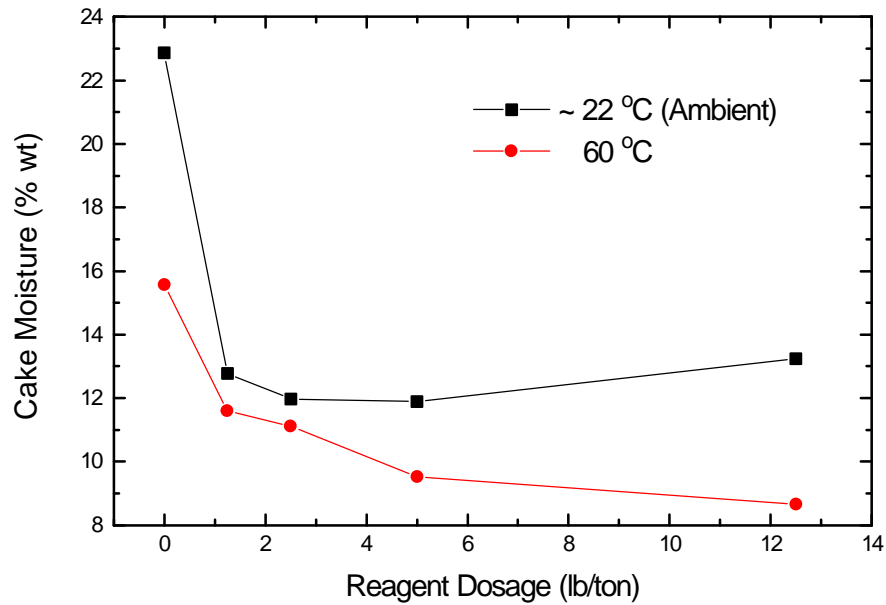


Figure 4. Results of the filtration tests conducted on the flotation products from the Middle Fork coal preparation plant as a function of reagent dosage (Reagent A). The tests were conducted at 0.2 inches of cake thickness and at two different temperatures.

At ambient temperature, the cake moisture obtained without using dewatering aid was 22.9%. The cake moisture decreased when the reagent dosage increased. At 5 lb/ton, the cake moisture became as low as 11.9%. Note, however, that there were no significant benefits for increasing reagent additions beyond 2.5 lb/ton when the coal slurry was filtered at ambient temperature.

Also shown in Figure 4 are the results obtained at 60 °C. In general, the cake moistures obtained at this elevated temperature were substantially lower than at the ambient condition. When no dewatering aids were used, the cake moisture was only

15.6%, which was substantially lower than obtained (22.9%) at the ambient temperature. The cake moisture decreased further as Reagent A was added to aid the filtration. It is interesting that the cake moisture continued to decrease with increasing reagent dosage without leaching a plateau, which is different from what was observed at the ambient temperature. The beneficial effects of filtering fine coal slurry at an elevated temperature may be attributed to the reduction in the viscosity of the water trapped in the capillary formed between the particles. Apparently, there is a synergy between using Reagent A and doing filtration at an elevated temperature.

Figure 5 shows a similar set of test results obtained with the flotation product (28 mesh x 0) from the Elkview Mining Company using Reagent A. The tests were conducted both at the ambient temperature (~22 °C) and at 60 °C using 200 ml of coal slurry in each test. The cake thicknesses measured after the drying cycle time were in the neighborhood of 0.2 inches. When the tests were conducted without using dewatering aid, the cake moistures were 20.8 and 18.9% at 22 and 60 °C, respectively. With the addition of Reagent A, the cake moisture decreased substantially. The moisture reduction increased with increasing reagent dosage. At 2 lb/ton, the cake moisture decreased to 13.7% at 22 °C. At the elevated temperature, it was further reduced to 12.6% at the same reagent dosage. It is interesting that, unlike the case with the Middle Fork coal, the cake moisture continued to decrease beyond 2 lb/ton, which was the maximum dosage used for the

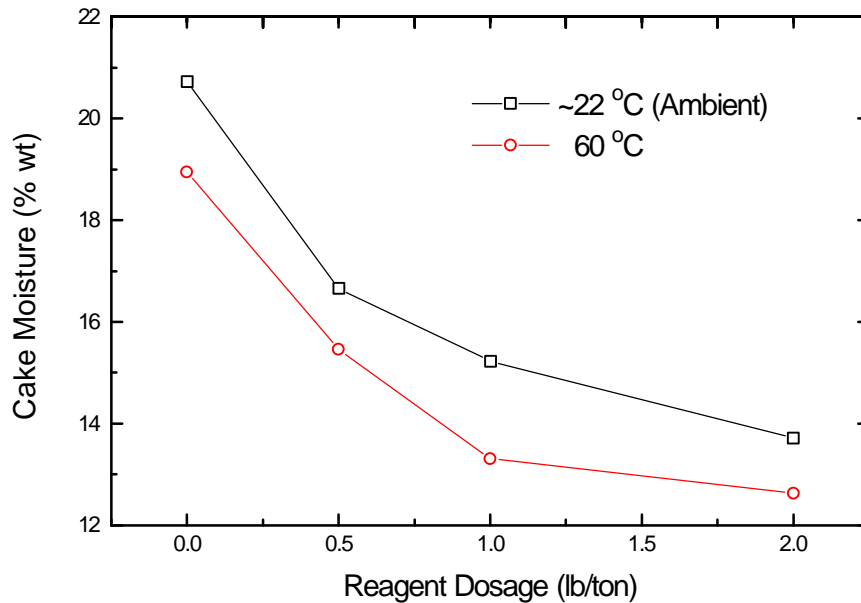


Figure 5. Results of the filtration tests conducted on the flotation products (28 mesh x 0) from Elkview Mining Company as a function of reagent dosage (Reagent A). The tests were conducted at 0.2 inches of cake thickness and at two different temperatures.

Elkview coal. It is possible that the moisture reduction reaches a plateau at higher reagent dosages, which should be determined in the future.

In addition to helping to reduce the cake moisture, Reagent A is capable of improving the kinetics of dewatering. When no reagent was used, the cake formation time was 20 seconds. At 2 lb/ton of Reagent A, the cake formation time decreased to 7 seconds. At 60 °C, the cake formation time was further reduced to 5.

The dewatering aid used in the present work is sensitive to the oxidation of a coal sample. Table 1 shows the results obtained after aging the Elkview coal sample for four weeks at the ambient temperature before filtration tests. Although Reagent A was able to

Table 1. Results Obtained Using Reagent A on the Elkview Coal Sample (28 mesh x 0)

Reagent Dosage (lb/ton)	Cake Formation Time (sec)	Product Moisture (% weight)
0	25	24.37
0.5	20	21.24
1	18	19.48
2	15	16.64

reduce the cake moisture substantially below the level that can be achieved without dewatering aid, the final moisture was not as low as obtained when the tests were conducted soon after the sample had been received. It is likely that coal particles are superficially oxidized during the process of aging, which may be detrimental to the adsorption of the dewatering aid used in the present work.

Table 2 shows the results of the filtration tests conducted on the flotation product from CONSOL, Inc., which was a 28 mesh x 0 Pittsburgh seam coal. Each test was conducted using 200 ml of coal slurry, which gave approximately 0.2 inches of cake thickness. As shown, the cake moisture was lower than that obtained without dewatering aid by 4% only at 2 lb/ton of Reagent A. This result was very poor as compared to the result obtained with another Pittsburgh coal sample received from the same company. As shown in our previous quarterly report, Reagent A was able to reduce the cake moisture of a Pittsburgh coal to less than 10%. The poor results obtained with the sample tested during the current reporting period may be attributed to the possible contamination of the coal sample by the flocculants during plant operation.

Table 2. Results Obtained Using Reagent A on the Pittsburgh Coal Sample (28 mesh x 0)

Reagent Dosage (lb/ton)	Cake Formation Time (sec)	Product Moisture (% weight)
0	61	25.57
0.5	48	23.96
1	36	22.42
2	36	21.92

SUMMARY

During the current reporting period, three different coal samples were subjected to laboratory vacuum filtration tests using one novel dewatering aid (Reagent A) developed at Virginia Tech. The results may be summarized as follows:

- The tests conducted on the Middle Fork coal sample (100 mesh x 0) showed that use of Reagent A in the amount of 1 lb/ton reduced the cake moisture by more than 10%, which accounted for a 50% moisture reduction. The tests were conducted using a relatively thin cake (0.1 inches) and long drying cycle times. However, extending the drying cycle time beyond 3 to 5 minutes did not make a significant difference.
- The test results obtained with the Middle Fork coal sample using 1 lb/ton of Reagent A showed that cake moisture increases with cake thickness. However, the same trend was observed when no dewatering aid was used. Thus, the use of Reagent A showed an advantage in moisture reduction by

approximately 12% at 0.1 inches cake, which diminished to 8% at 0.5 inches cake thickness.

- Tests were conducted on the Middle Fork coal sample by using different amounts of Reagent A at 0.2 inches of cake thickness and at two different temperatures. At the ambient temperature, the cake moisture was reduced sharply at 1 lb/ton and reached a plateau at 2.5 lb/ton. At 60 °C, however, the cake moisture continued to decrease with increasing reagent addition. In general, significantly lower cake moistures could be obtained at the elevated temperature. The improved moisture reduction at the higher temperature may be attributed to a decreased in the viscosity of the capillary water.
- Effects of reagent dosage on the cake moisture were also studied on the Elkview coal (28 mesh x 0 coal). The tests were conducted at 0.2 inches of cake thickness and at two different temperatures. The cake moistures obtained at 60°C were considerably lower than those obtained at the ambient temperatures. The results showed that judicious use of Reagent A can reduce the cake moisture to 12 to 14% range.
- A series of tests were conducted on the Elkview coal sample after aging the coal sample for a period of four weeks at ambient temperature. Although the results showed substantially improved moisture reductions, the improvement was less than obtained with fresh sample. The detrimental effect of aging may be attributed to the superficial oxidation of the coal sample.
- Tests conducted on the Pittsburgh coal from CONSOL, Inc. showed that use of Reagent A reduced the cake moisture by approximately 4%, which was

poor as compared to those obtained with another Pittsburgh coal sample. The poor performance of the novel dewatering aid may be attributed to the possible contamination of the coal sample by a flocculant during plant operation.

Quarterly Technical Progress

Report 5

October-December, 1996

**STUDY OF NOVEL APPROACHES FOR
DESTABILIZATION OF FLOTATION FROTH**

*Prepared for
The Appalachian Clean Coal Technology Consortium*

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STUDY OF NOVEL APPROACHES FOR DESTABILIZATION OF FLOTATION FROTH

ABSTRACT

Fine coal recovery from fine waste stream using froth flotation technique is becoming an important and integral part of coal preparation plans. Column flotation technique has proven to be the most effective and cost-efficient in recovery of fine coal. However, in some columns use of glycol-based frother produces a stable froth, which is not destroyed easily. The main objective of this program is to develop novel approaches for destabilization of stable froth. The approaches involve addition of either chemically treated coarse coal or addition of chemically treated magnetite-limestone suspended in an oil medium to the froth to break it down. Mechanical approaches to be tested involve utilization of ultrasonic energy or cyclone or vacuum. This report discusses technical progress made during the quarter from January to March 1996.

INTRODUCTION

In froth flotation process, frothers are utilized to produce stable small bubbles which carry the floatable particle. In case of an ideal froth flotation, the froth should have good fluidity and optimal rigidity. The froth should be just stable enough to carry the floated particles out of the flotation cell. Once the froth is scraped out the cell, it should collapse to free and concentrate the volume of floated particles. Such a froth will minimize the entrainment of undesired mineral particles

into the concentrate and can provide additional selectivity for the flotation process. Too stable or too unstable froths penalize the separation efficiency and operation smoothness. If the froths are too stable, they will not break up after being scraped out of the flotation cell. Usually, such overly stable froths have low fluidity. The unwanted excessive stability can cause serious problems for downstream processes, such as dewatering of flotation concentrate, tailings handling, and re-use of processing water.

Generally, alcohol-based frothers provide froth which collapses as soon as froth is removed from the flotation cell and sprayed with water. Glycol-based frothers on the other hand produce a much stronger froth which is difficult to break by simple physical techniques and requires a large amount of chemicals to break the froth, which adds to the processing cost.

OBJECTIVES AND SCOPE

The main objective of the proposed project is to develop new defoaming techniques for destruction of overly stable froths produced in fine coal froth flotation.

It is also the objective of the project to study chemical dynamics of the three phase froth.

The focus of this project will be to investigate new defoaming techniques. The existing commercial defoamers, such as surfactants, copolymers, hydrophobic silica, amide particle, are expensive and thus economically unfavorable for breaking up fine coal froths. In this project, new techniques for defoaming coal froth with less expensive chemical will be developed. Two approaches will be studied. One will

involve use of coarse coal particles. To ensure that the surfaces of coal particles are sufficiently hydrophobic, the coal particles will be pretreated with cationic surfactant. The second approach will be to use magnetite and limestone particles. Since both magnetite and limestone particles are naturally hydrophilic, it is necessary to coat the particles with specific chemicals to make the surface highly hydrophobic (contact angle $>120^\circ$). The hydrophobic particles are then dispersed in oil. It is anticipated that these new defoamers will be advantageous over existing defoamers. Besides their low cost, the use of hydrophobic limestone particles can also help to capture the sulfur dioxide during combustion and thus reduce the mission.

APPROACH

The project consists of four (4) tasks. Progress made in each task during the period of October-December, 1996 is described below.

Task 1. Sample Acquisition and Characterization

Destabilization studies of flotation froth were conducted with both flotation feed and froth product. The samples were obtained from the Pittston Coal Company which was recommended by the ACCTC. The flotation feed contains 32.93% ash and column froth product 8.54% ash. Size analysis of the feed and product samples have been performed and results included in the previous quarterly report. The major findings are that D_{50} is 40 and 80 mm for the feed and product, respectively and the froth product had 48 weight percent particles finer than 200 mesh (74 mm) and the feed had 63 weight percent particles finer than 200 mesh.

Task 3. Fine Coal Froth Destabilization Studies

This task consists of studies on chemical and mechanical defoaming techniques for fine coal flotation froth. The chemical process involves use of coal, magnetite, or limestone particles coated with surfactant for froth spraying. The mechanical process utilizes cyclone, ultrasonic energy, or vacuum to destabilize froth. In the last quarter major efforts were devoted to the investigation of chemical defoaming techniques. The tests were conducted with 200 ml slurry containing 7.1% solids.

Figure 1 shows changes in froth surface area as a function of the dosage of fuel oil used as collector and Allied Colloids 944 B frother. The surface area was that of froth remaining in the column after 10 min froth drainage time, determined from the measured pressure differential. The ratio of collector and frother dosage was kept constant at unity in these tests. The froth surface area increased consistently with reagent dosage, due to reduced bubble size and increased froth stability. There is an essentially linear relationship between the surface area and the dosage in the range of 100-500 g/t.

Figure 2 demonstrates effects of coal slurry solids concentration on surface area of froth remaining after 10 minute drainage. The surface area increased

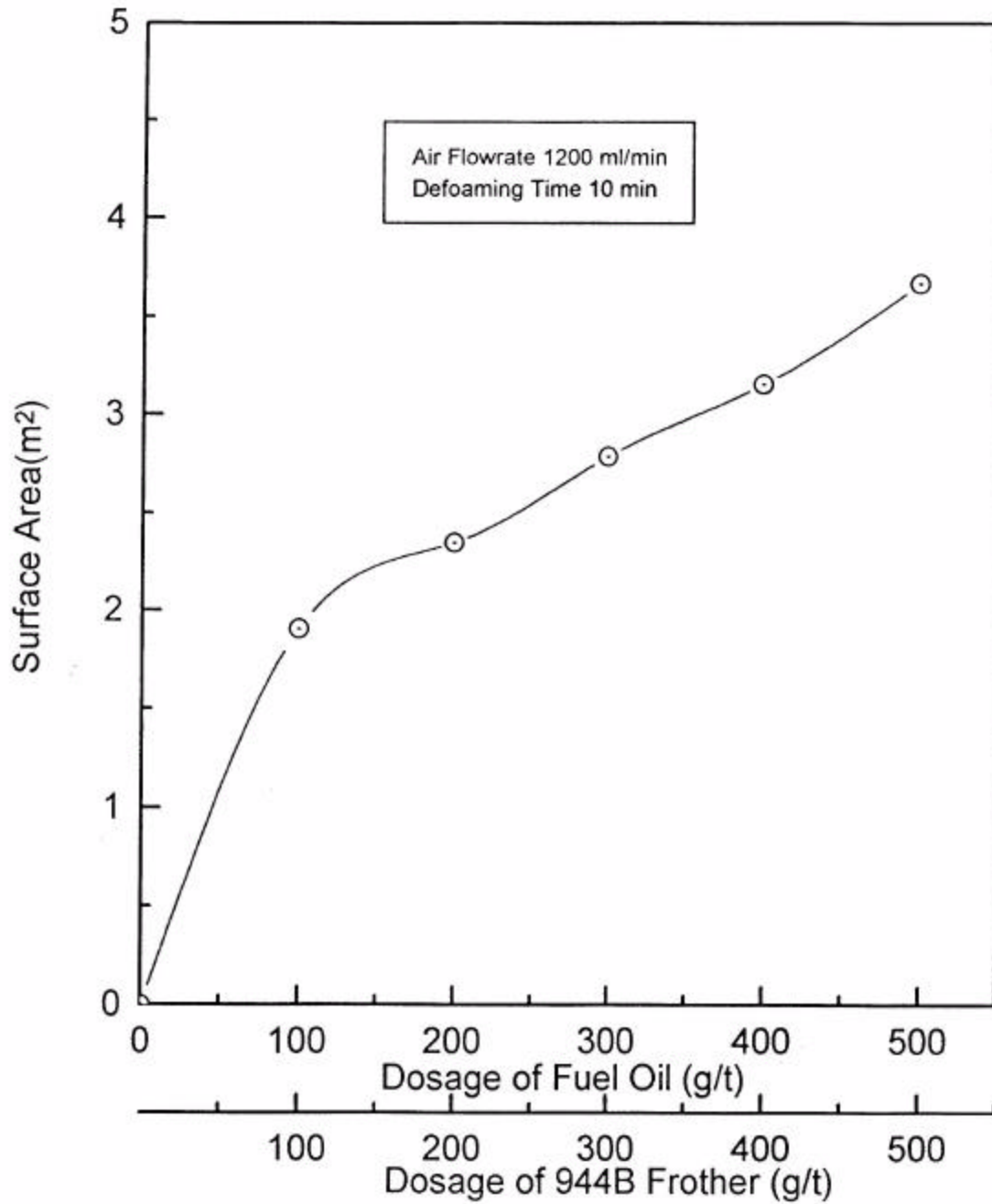


Figure 1. Effects of reagent concentration on surface area of froth remaining after 10 minutes.

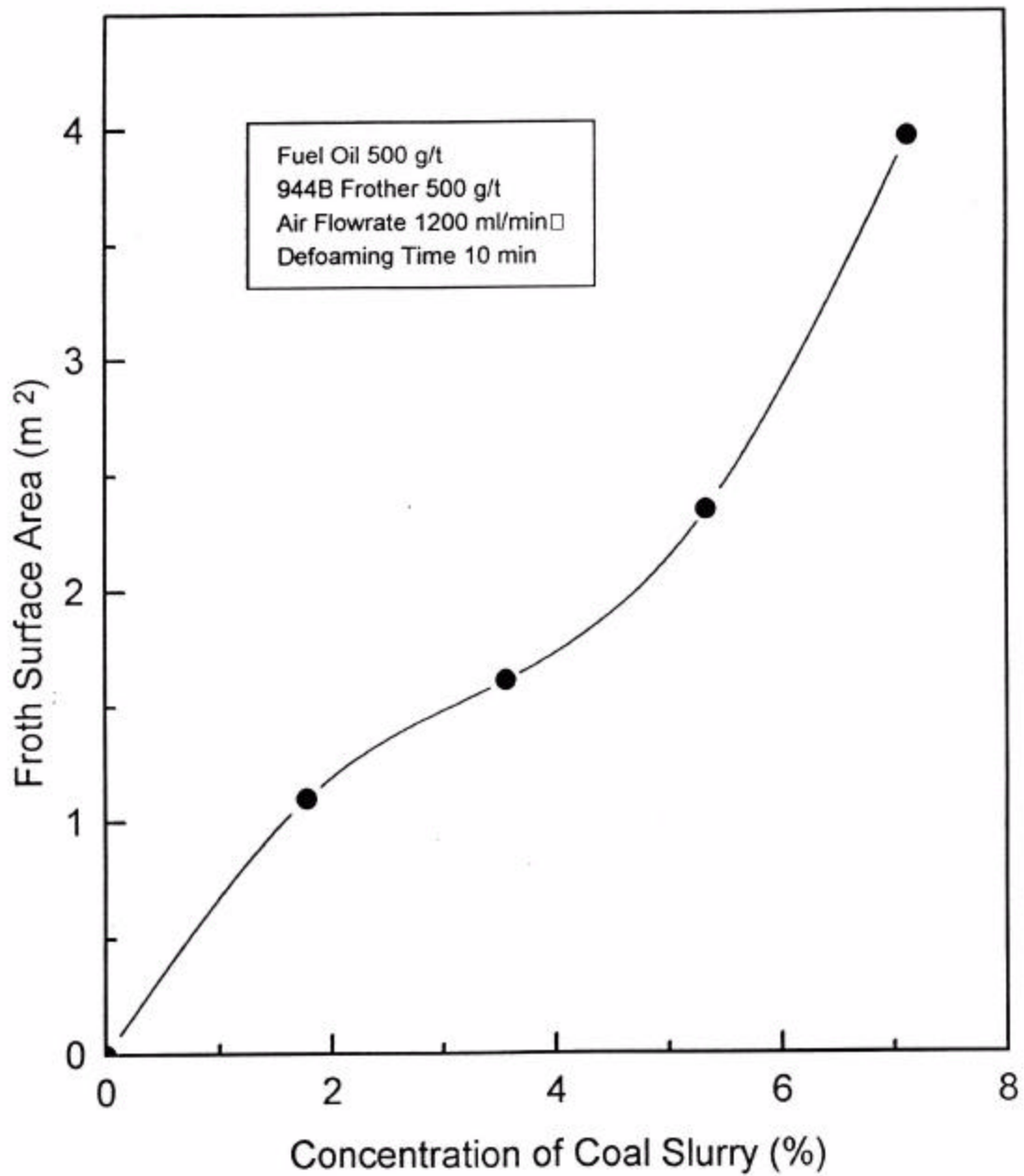


Figure 2. Effects of solids concentration in slurry on surface area of froth remaining after 10 minutes.

sharply in the presence of coal particles. Higher solids concentration generated more froth surface area. Obviously coal particles have remarkable impacts on froth stability.

Figure 3 shows froth destabilizing effects of limestone particles that are coated or not coated with the surfactant cetylpyridinium chloride (1-hexadecylpyridinium chloride) in fuel oil #2. The surface area of froth decreased dramatically with increasing the limestone dosage (expressed as weight percentage of coal slurry) up to 0.4%. Further increase in limestone dosage had less pronounced but still significant froth destabilizing effects. Use of about 0.85% 40-50 mesh coated limestone particles reduced froth surface area from 2.75 to 0.4 m², representing an 85% reduction. By comparison, uncoated limestone particles were less effective in destabilizing froth, due to its surface hydrophilicity.

Figure 4 shows effects of particle size of uncoated limestone particles on froth destabilizing ability. Smaller froth surface area indicates a stronger froth destabilizing ability, which means less froth remained after limestone particles were applied to the froth. The results suggested that the particle size did not have explicit effects on froth destabilizing ability. This is possibly because particle size has two counteracting effects: smaller particles possess larger surface area but they also have weaker penetration capability in the froth due to their small inertia.

Figure 5 shows froth destabilizing effects of concentration of minus 100 mesh (150 μm) coated limestone. Froth surface area decreased substantially with increasing limestone concentration up to about 0.8-1% and then it leveled off. The

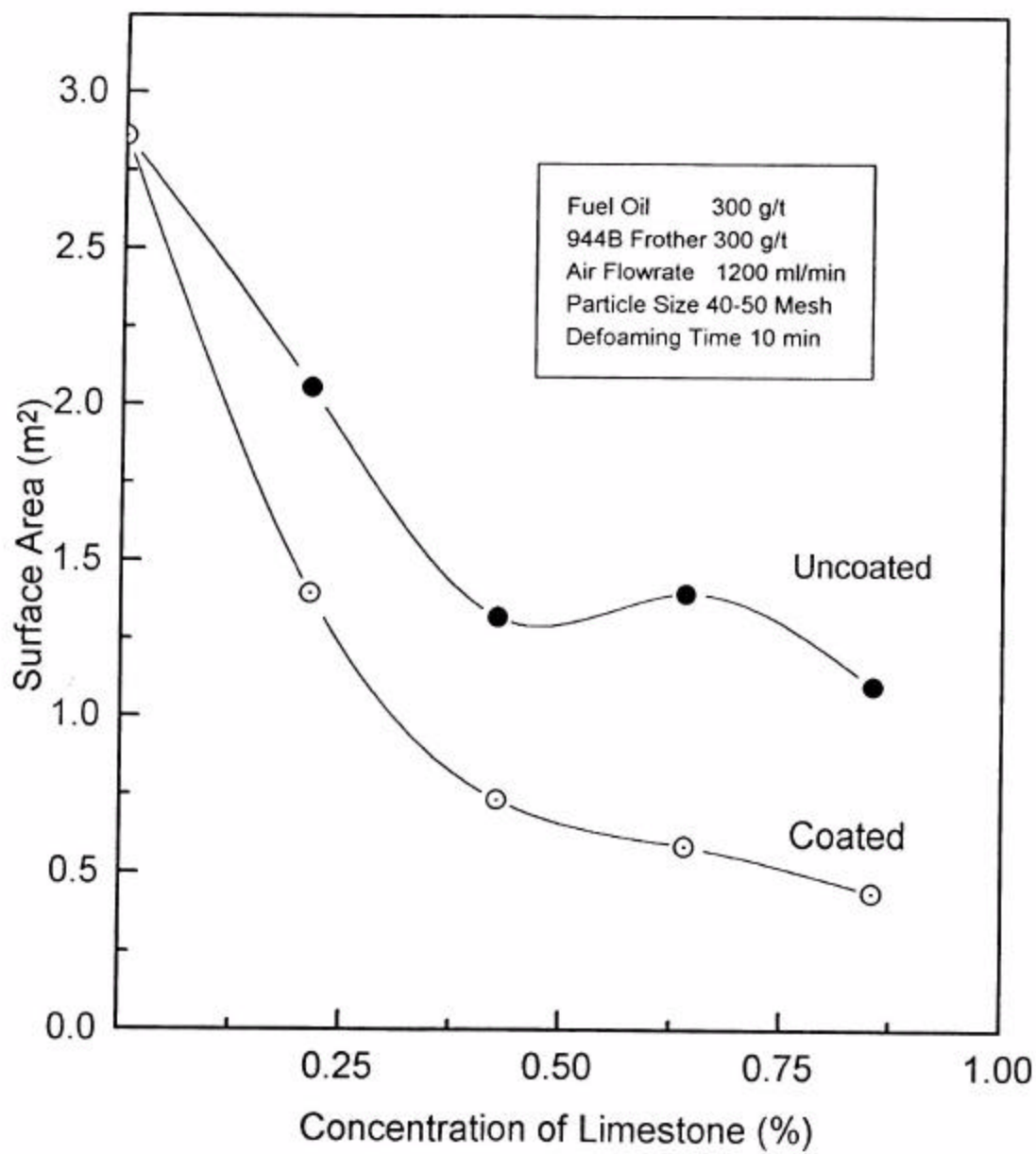


Figure 3. Effects of limestone concentration on surface area of froth remaining after 10 minutes.

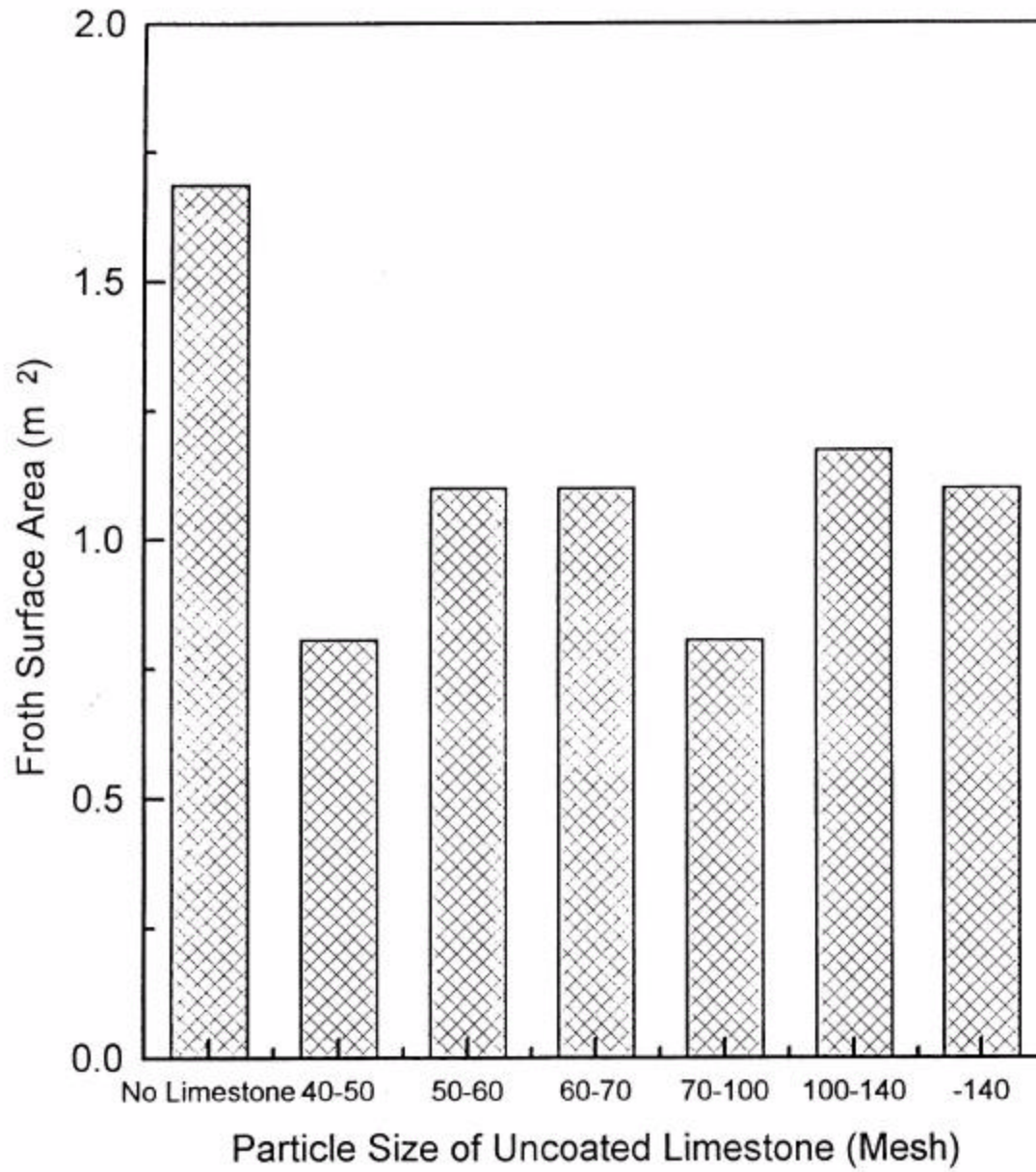


Figure 4. Effects of uncoated limestone particle size on surface area of froth remaining after 10 minutes.

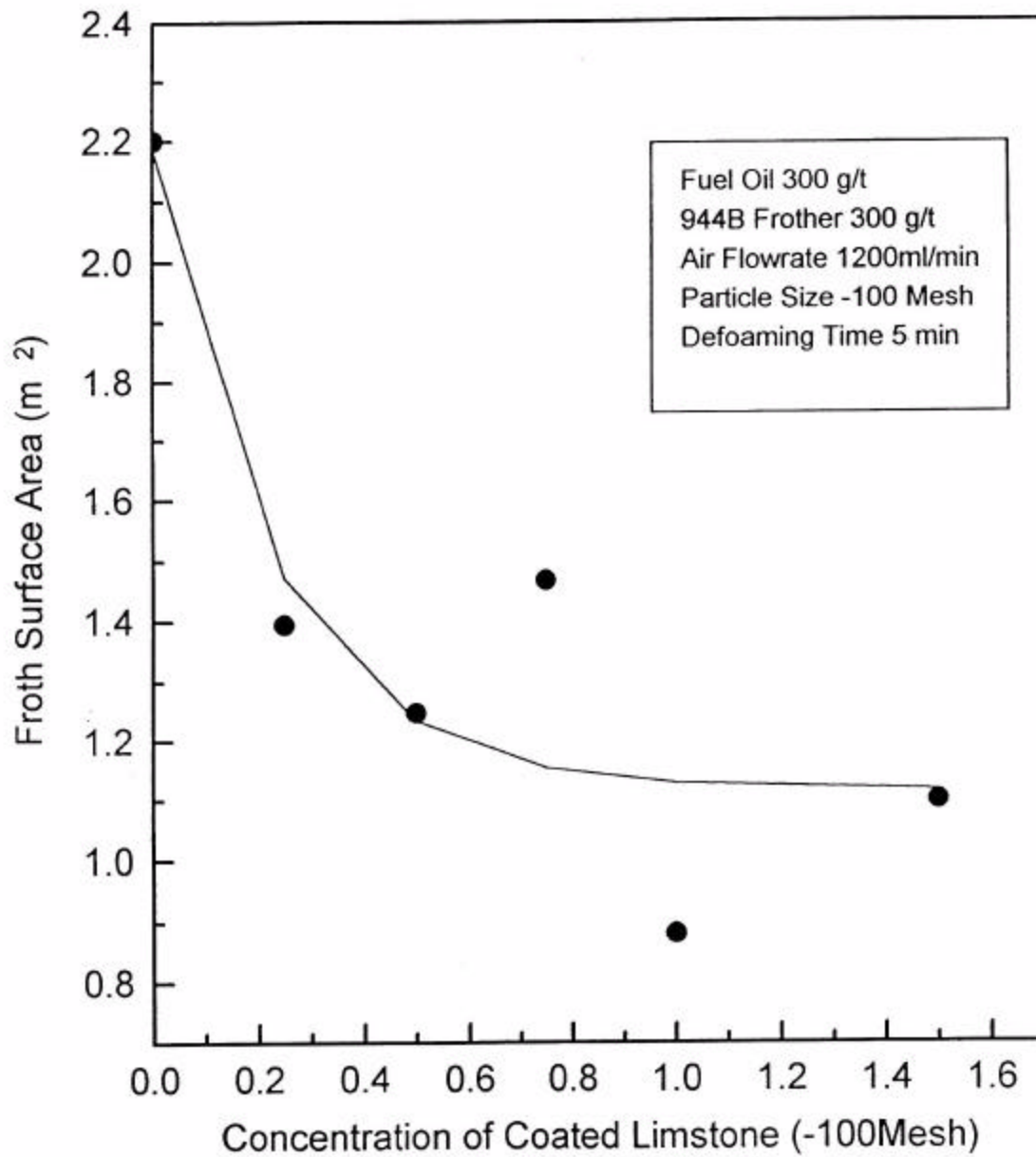


Figure 5. Effects of coated limestone concentration on surface area of froth remaining after 5 minutes.

concentration of limestone particles is an important factor determining froth destabilizing ability.

Magnetite particles (9-23 μm) were also used to destabilize froth. The results are shown in Figure 6. Uncoated particles were not effective in destabilizing froth. The maximum froth area reduction, from 2.0 to 1.75 m^2 , was observed at a dosage of 0.43%. Coated magnetite particles were much more effective than the uncoated ones for destabilizing froth. Even low dosage of magnetite up to 0.2% showed better froth destabilizing effects. At a dosage of 0.21% the froth area was reduced to 0.5 m^2 . But dosages higher than 0.2% had no significant additional benefits. Comparing the results obtained with limestone it appears that reagent coated magnetite particles are more effective in destroying froth.

CONCLUSIONS

- . Higher concentration of frother produced more stable froth. There is an almost linear relationship between froth surface area and Allied Colloid 944 B frother concentration up to 0.5 kg/t.
- . Increasing coal solids concentration in slurry substantially increased froth stability.
- . Limestone particles at 0.85 weight percent dosage were effective in destabilizing froth. Particle size and coating with surfactant showed relatively small impacts.

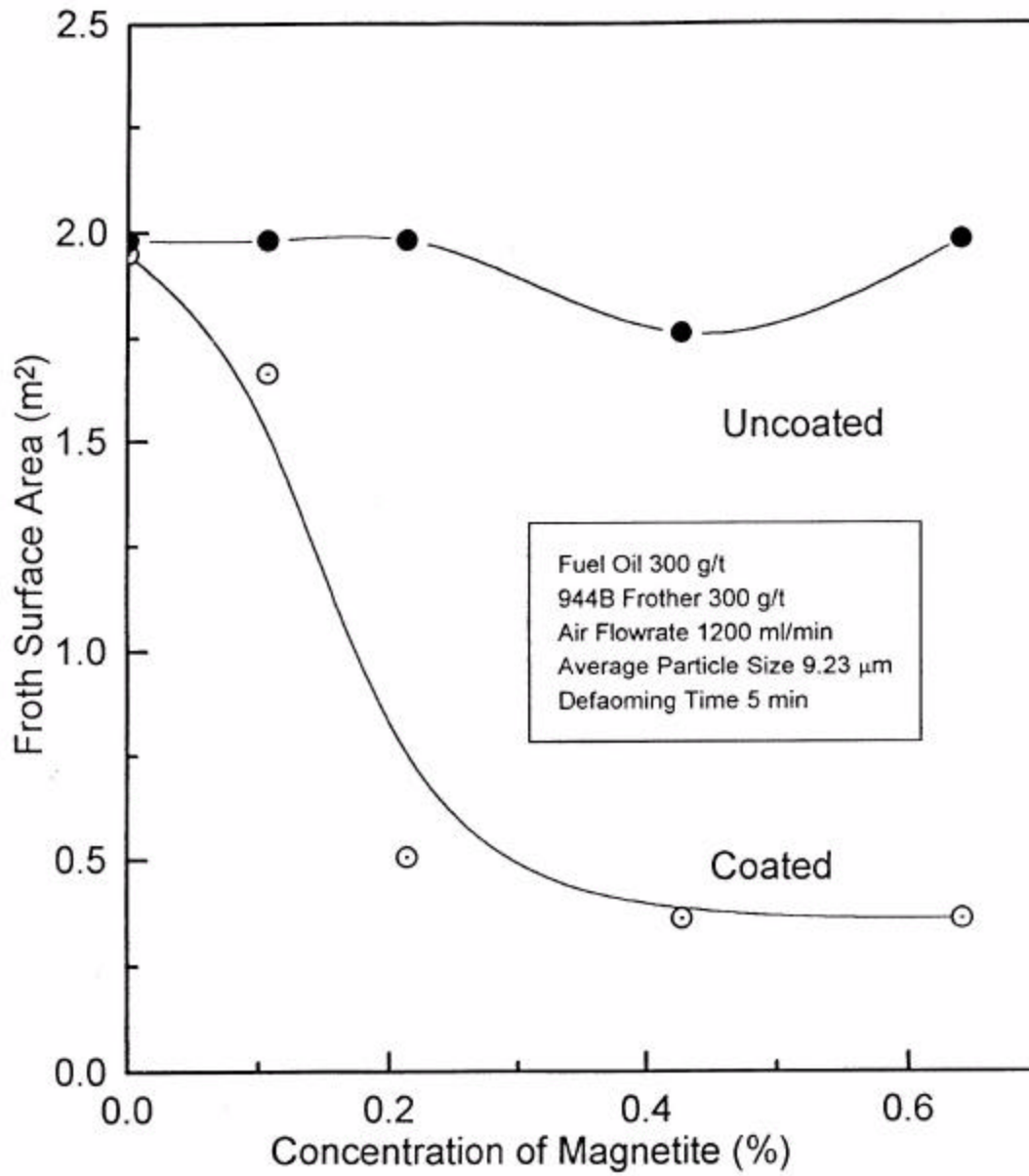


Figure 6. Effects of concentration of limestone coated/uncoated with surfactant on surface area of froth remaining after 5 minutes.

- About 0.2 weight percent of reagent coated magnetite was the most effective froth destabilizer.

ACTIVITIES FOR NEXT QUARTER

Since the most effective defoaming was observed with reagent coated magnetite, the future research work will be focused on the effects of size of magnetite particles coated with different surfactants that are used to increase surface hydrophobicity. The study will be conducted with respect to:

- particle size of magnetite
- surfactant type
- surfactant concentration
- conditioning time
- dosage of coated magnetite

A number of other defoaming experiments will also be conducted to study defoaming effectiveness using coated coal, limestone, smoke, low vacuum.