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MICROGAS DISPERSION FOR FINE COAL CLEANING

Technical Progress Report for March 1—August 31, 1981

By
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G. S. Halsey
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Work Performed Under Contract No. FG22-80PC30234

Virginia Polytechnic Institute and State University
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Technical Progress Report
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MICROGAS DISPERSION* FOR FINE COAL CLEANING

by

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* This term has been changed to Colloidal Gas Aphrons (CGA).

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INTRODUCTION

Froth flotation is a very versatile process that is extensively used to concentrate a wide variety of minerals and coal. It is unfortunate, however, that its effectiveness is limited to a relatively narrow size range. Generally, the best recoveries are obtained for intermediately sized particles from 10 to 100 μm. Due to the difficulties encountered when floating fines, it is not uncommon for fines to be discarded without further processing.

The process of bubble-particle adhesion can be envisioned as taking place via: (1) a collision of particles with bubbles, (2) a thinning of the water film between the particle and the bubble, and (3) a rupture of the residual film resulting in the formation of a three-phase contact between the bubble, the particle, and water. The difficulty in floating small particles is attributed to the fact that small particles do not have enough momentum to thin the disjoining film between the particle and the bubble. Scheludko et al. (1976) conducted thermodynamic analysis at the three-phase contact, and concluded that a flotation limit exists in terms of particle size. More recently, however, Derjaguin and Dukhin (1979) suggested that fine particle flotation is possible without forming the three-phase wetting perimeter, i.e., flotation can occur with zero contact angle. This 'contactless flotation' concept was based on the possibility that small particles can be held to the bubbles by London dispersion forces. For the flotation of large particles, however, 'contact flotation' is necessary because the only way to retain a large particle on the bubble is by forming a three-phase wetting perimeter that will be able to resist the greater tearing-off forces. Derjaguin and Dukhin (1979) noted that the tearing-off force of a 100 μm particle is 10^6 times greater than that of a 1 μm particle, as the force is proportional to the volume of the particle.

Sebba and Yoon (1981) constructed a microflotation cell that can be mounted on a microscope stage, and observed the flotation of micron-sized graphite particles using small air bubbles. Their observations revealed that the bubble-particle attachment occurs tenuously at a single point, which appears to support the 'contactless flotation' theory proposed by Derjaguin and Dukhin (1979). This finding imposes an important constraint on fine particle flotation, i.e., the flotation must be carried out under quiescent conditions.

There are two other essential requirements for fine particle flotation. Firstly, it is desirable to have small air bubbles for improved collision efficiency, as has been shown by Anfruns and Kitchener (1976, 1977). Secondly, as another means of improving the probability of collision, it is best to use a 'cloud' of sparsely dispersed microscopic air bubbles (Packham and Richards, 1975). However, the use of too many bubbles may increase the probability of mechanically entrapping unwanted particles, thus reducing the selectivity of the process.

The purpose of this investigation is to study the application of small gas bubbles, named Colloidal Gas Aphrons (CGA: formerly called Microgas Dispersion; Sebba, 1971), for fine coal flotation. Results presented in this report are from the work done during the second semi-annual reporting period of the two-year project.

EXPERIMENTAL

Samples

a) For Electrophoretic Measurements

A R-O-M coal sample from the Jawbone seam (Hurricane Creek Mine) was provided by Clinchfield Coal Company. A quartz sample was purchased from Ward's Natural Science Establishment, Incorporated.

The coal sample was crushed and screened to obtain a -1/4 inch +10 mesh fraction, which was then cleaned of its ash by heavy medium separation at a specific gravity of 1.30. Magnetite was used as the dense medium. Care was taken to remove the magnetite particles adhering to the clean coal by repeated washings with water. This clean coal sample was then crushed with a micro-analytical mill (Tekmar A-10) and pulverized with an agate mortar and pestle. The -3 μ fraction of the pulverized coal, as obtained by sedimentation using an Andreasen pipette, was used for microelectrophoresis.

The pure quartz sample was crushed, pulverized, and sized similarly for the electrophoretic measurements.

b) For Flotation Experiments

Two R-O-M bituminous coals were used for the flotation experiments. A sample from the Eagle seam assaying 36% ash was provided by United Coal Company, and a blended sample composed of the Meigs Creek (No. 9), Pittsburgh (No. 8), and Waynesburg (No. 11) seams assaying 20% ash was supplied by R & F Coal Company. Each sample was pulverized to 100% -100 mesh using a hammer mill and riffled into 200-gram lots. The coal samples, thus prepared, were kept under a nitrogen atmosphere to minimize oxidation. Just before the flotation experiments, the 200-gram sample was taken out of the nitrogen atmosphere and further riffled into 25-gram lots.

Table I gives the results of a dry-screen analysis of the Eagle coal sample with the ash content of each size fraction. As anticipated, the ash content was found to increase with decreasing particle size.

Reagents

The frothers used in the present work are as follows:

Aerofroth 73	American Cyanamid Company
UCC Silicone L7001	Union Carbide Company
Tergitol TMN-6	Union Carbide Company
Tergitol 15-S-9	Union Carbide Company
MIBC	Union Carbide Company
MIBC	Consolidation Coal Company
Pine Oil	Hercules Incorporated
Dowfroth 200	Dow Chemical Company
Dowfroth 250	Dow Chemical Company
Dowfroth 400	Dow Chemical Company
Dowfroth M150	Dow Chemical Company
Dowfroth M210	Dow Chemical Company
Dowfroth E1128	Dow Chemical Company

Collectors used for flotation experiments included kerosene and No. 2 diesel oil supplied by Consolidation Coal Company.

TABLE I.

Results of a Dry-Screen Analysis Conducted on the Eagle Coal Sample

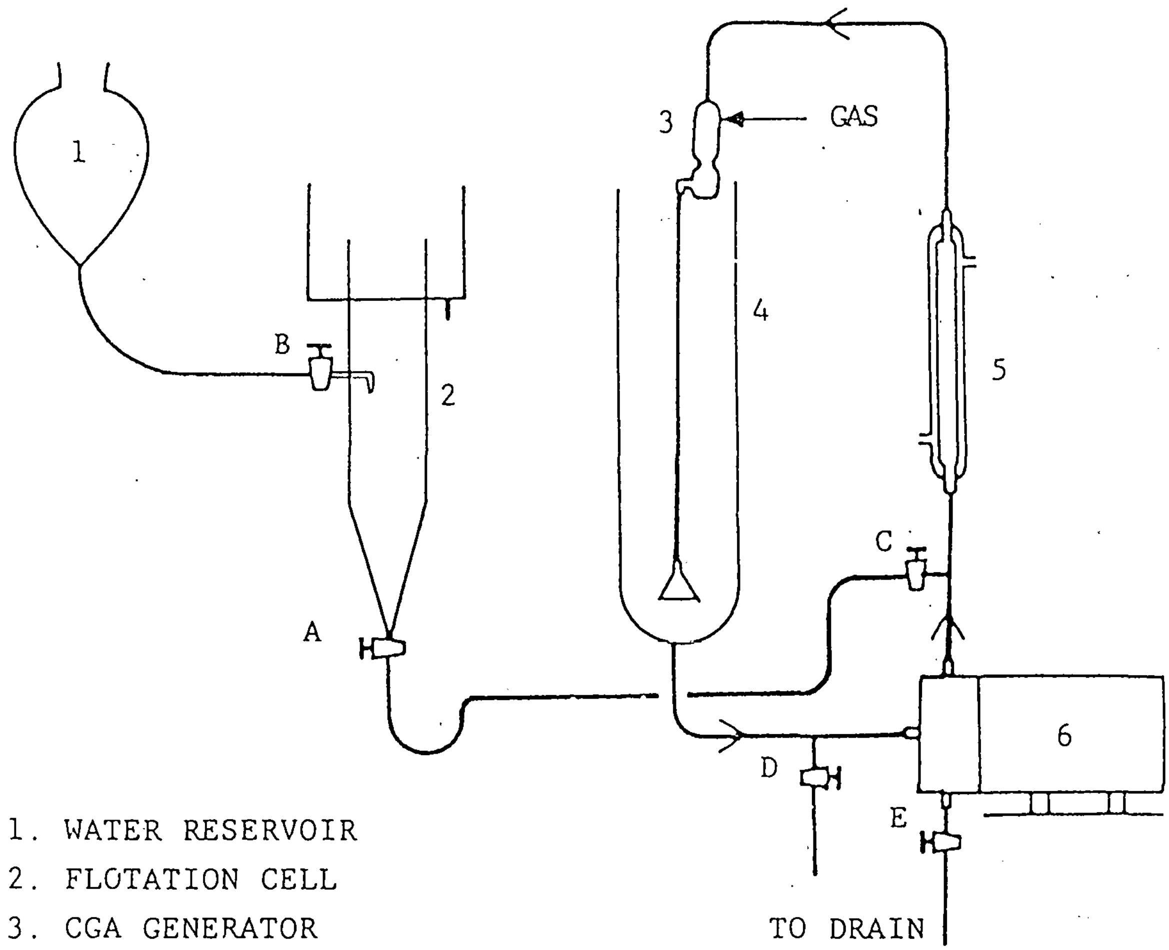
Screen Mesh	Weight (%)	Analysis (%)		Distribution (%)	
		Ash	Coal	Ash	Coal
-100 +150	2.7	28.2	71.8	2.1	3.1
-150 +200	16.3	32.3	67.7	14.4	17.4
-200 +250	18.4	36.8	63.2	18.5	18.3
-250 +325	21.7	37.5	62.5	22.2	21.4
-325 +400	19.2	38.6	61.4	20.3	18.6
-400	21.7	38.0	62.0	22.5	21.2
	100.0	36.6	63.4	100.0	100.0

Equipment

The streaming currents were measured in a bubble column, as described in the previous report (Yoon and Sebba, 1981), to obtain information regarding the charges of bubbles generated using various frothers. A Rank Brothers' Particle Electrophoresis Apparatus was used to obtain information on the surface charge of coal and quartz particles suspended in various frother solutions.

The flotation apparatus shown in Figure 1 is similar to the one described in the previous report. It incorporates a glass aspirator developed by Sebba (1971) to generate CGA. The volume of the cylindro-conical flotation cell is 1750 ml. The stopcock located on the side of the flotation column is used to flood the froth product into the catch pan without disturbing the settled refuse and the froth layer. A 1 hp centrifugal pump (Eastern Model MDH-32) circulates the frother solution past the glass aspirator to generate the CGA.

The method of generating CGA using the glass aspirator may prove difficult to apply industrially. Figure 2 shows a flotation apparatus in which CGA is generated in a much simpler way and used for flotation. It consists of a blender (Waring Model 31BL42) and a cylindrical separatory funnel. Small bubbles are generated in the blender by high shear agitation. The mechanism of generating small bubbles using a blender is simply that the large bubbles formed around the vortex are shredded into smaller ones by the high speed (10 to 12,000 rpm) impeller. The bubbles formed in the blender are pumped via a peristaltic pump to the bottom of the separatory funnel which is used as the flotation cell.



1. WATER RESERVOIR
2. FLOTATION CELL
3. CGA GENERATOR
4. CGA RESERVOIR
5. HEAT EXCHANGER
6. CENTRIFUGAL PUMP

A, B, C, D & E. TEFLON STOPCOCKS & VALVES

Figure 1. Apparatus for CGA flotation.

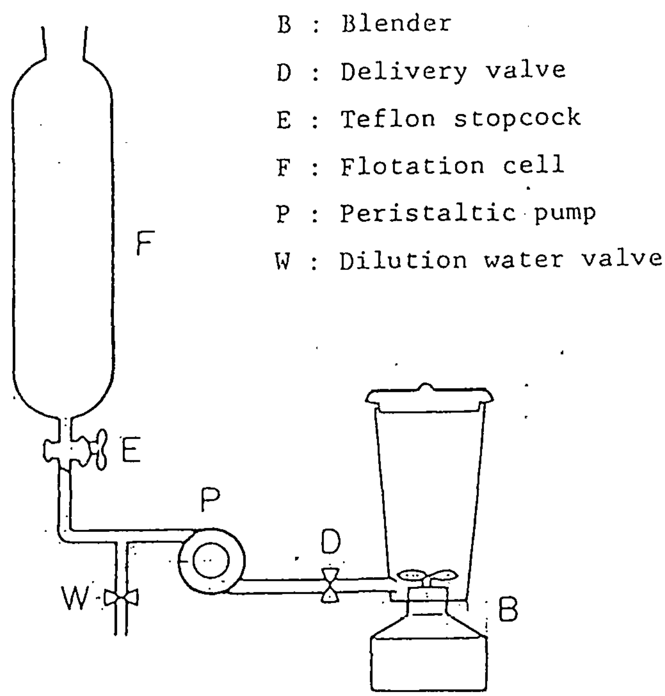


Figure 2. Bench-scale batch flotation apparatus using high speed blender.

Procedures

a) Streaming Current Measurements

The potential (E) across the Ag/AgCl electrodes was measured with an electrometer. The resistance (R) of the solution across the electrodes was measured by means of a conductivity bridge. From the values of E and R, thus measured, the streaming current (I) was calculated. These measurements were made with and without passing CGA bubbles, and the difference was taken to be the streaming current generated by the bubbles.

b) Electrophoretic Measurements

Having obtained the -3 μm fractions of the pulverized samples, each was then diluted to a desired concentration and a measured volume of the suspension was transferred to a 100 ml volumetric flask. The pH was adjusted to a desired value by adding HCl and NaOH, and a known amount of frother was added. After 30 minutes of conditioning, the suspension was transferred to a flat electrophoretic cell to measure the mobility. Usually, 20 measurements were taken on each sample and averaged. Platinized platinium electrodes were used with the cell.

c) Measurements of CGA Stability

To assess the stability of CGA, the following procedure was employed. A fixed volume (100 ml) of CGA was injected into a graduated cylinder, and, as the bubbles rose, the rising boundary separating the clear solution from the cloudy aphron suspension was observed. The time required for this boundary to reach finite volumes was measured, and the two were plotted against each other. It should be noted that this measurement is subjective and is intended only for comparative purposes.

When comparing the effects of various frothers, a concentration of 1 ml/ℓ was used in each case. For the aspirator-generated CGA, a circulating flow was established and then stopcock C (Figure 1) was opened and CGA was injected into a cylinder. For the blender-generated CGA, the surfactant solution was agitated in a blender at its highest speed (12,000 rpm, free running) and then transferred via a peristaltic pump into the graduated cylinder.

d) Flotation Tests

The conditioning process for the coal samples prior to flotation was as follows: A mixture of 25 g of coal and 200 ml of tap water was agitated in a Waring blender for 4 minutes to wet the coal sample. A volume of kerosene was then added with a microliter syringe and conditioning continued for 4 minutes. This slurry was then poured into the flotation cell.

For the aspirator-generated CGA tests, a volume of CGA was injected into the cell through stopcocks A and C (Figure 1). When injecting a quantity of CGA less than 1000 ml, additional water had to be added prior to flotation to ensure that the froth layer rose above the level of stopcock B. After the injection of the CGA, the mixture was allowed to stand for 5 minutes (less if the froth layer began to noticeably break), during which the CGA levitated the coal particles while the refuse particles settled to the bottom. Stopcock B was then opened and the froth product floated over into the catch pan. The refuse was drained into a beaker by opening stopcock A.

When doing a two-stage test, the refuse was first drained, and then the froth product was repulped within the flotation cell. Additional CGA was then generated and injected into the cell, and the single-stage flotation procedure employed.

For the blender-generated CGA tests, a 1 ℓ cylindrical separatory funnel was used as the flotation cell. A known volume (usually 300 ml) of CGA was pumped with a peristaltic pump through Tygon tubing into the funnel. This was allowed to stand as described before, and then the refuse and froth product were each drained into a beaker.

The flotation products obtained with each technique were then filtered, dried, weighed, and assayed for ash.

RESULTS

Stability of CGA

a) On CGA Produced by the Aspiration Technique

In previous work (Yoon and Sebba, 1981), CGA was prepared by the glass aspirator technique using a centrifugal pump with a 1/3 hp motor. Hoping that a more powerful pump would produce more stable CGA, a 1 hp centrifugal pump was used during this reporting period. Figure 3 shows the stability of the CGA produced with various frothers using this pump. It was found, however, that the CGA produced in this way was not significantly more stable than that prepared by using a 1/3 hp pump.

Figure 3 also shows that Dowfroth M150 produces the most stable CGA and, presumably, the smallest air bubbles, and MIBC the least

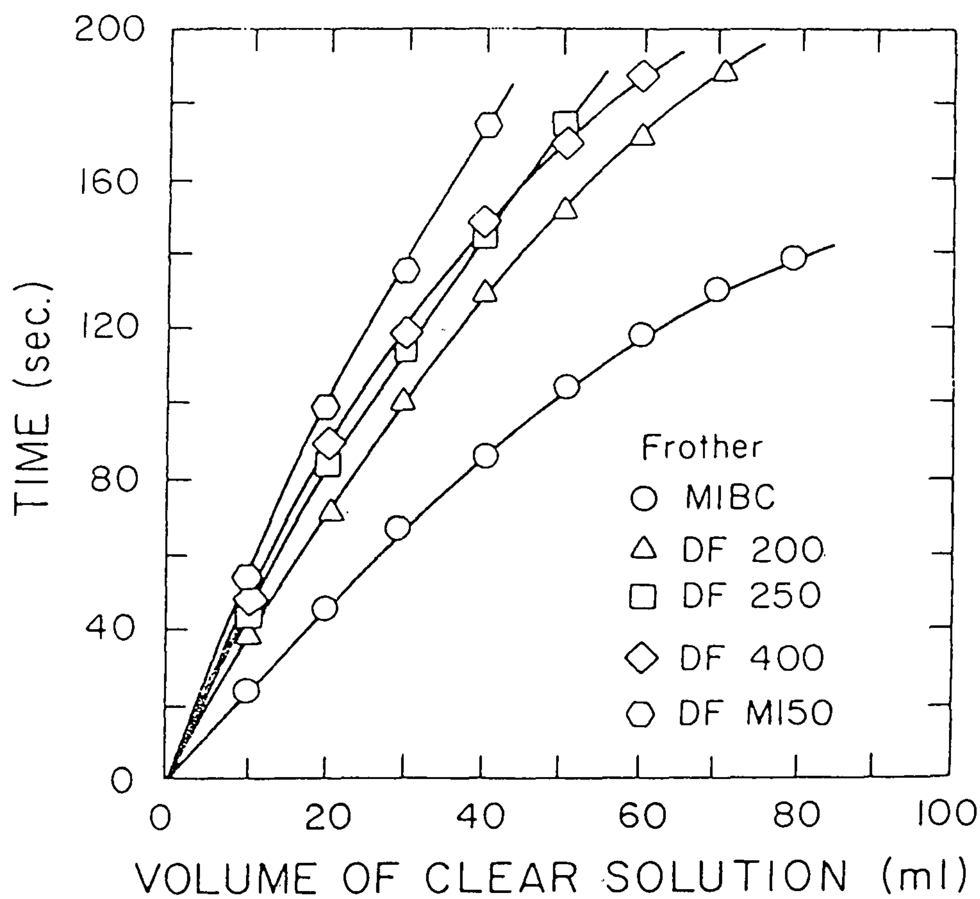


Figure 3. Stability of CGA produced by the aspirator technique using various frothers at a concentration of 1 ml/ℓ.

stable. Dowfroth 400 and 250 have approximately the same stability, followed by Dowfroth 200.

Using 0.07 ml/ℓ of Dowfroth M150, the stability tests were conducted as a function of aspiration time. The stability was found to increase with time during the first 1 and 1/2 minutes, level off during the next 2 and 1/2 minutes, and then begin to decrease. This deterioration may have been caused by the rising temperatures associated with increasing aspiration time. Tests were also conducted varying the frother (Dowfroth 400) concentration from 0.1 to 8 ml/ℓ, and the CGA stability was found to increase slightly with increasing concentration. Similar tests were also performed with MIBC, where it was found that this reagent did not produce a stable CGA even at high concentrations.

b) On CGA Produced by the Blender Technique

Figure 4 shows the stability of CGA produced by the blender technique using seven different frothers. As is the case with the aspirator-generated CGA, MIBC produces a much less stable CGA than the Dowfroth frothers. Among the Dowfroth homologues, DF E1128 generates the most stable CGA, which may be attributed to its large molecular weight.

Silicone L7001 and Tergitol TMN-6 produce very stable CGA due to their large molecular weights. With the CGA generated with the former, as much as 80% of the volume is air. The CGA produced with pine oil and Dowfroth M210 is unstable, even more so than that produced with MIBC.

The most notable feature of Figure 4 is that the CGA produced by the blender technique is as stable as those produced by the aspirator technique. The blender technique is much simpler, however, and less likely to

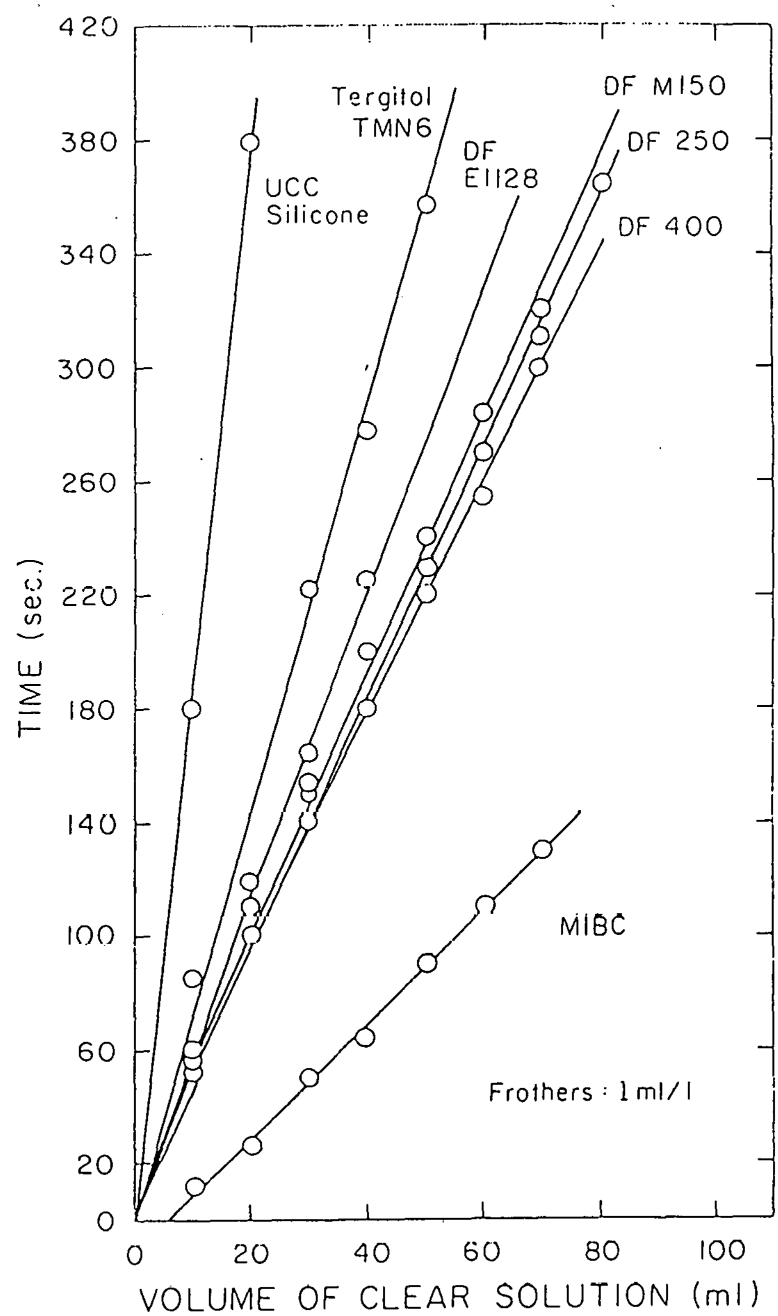


Figure 4. Stability of CGA produced by the blender technique using various frothers at a concentration of 1 ml/ℓ.

impose problems in scale-up. Factors affecting the CGA stability would be impeller rpm and the shape and dimensions of both the impeller and the container. It would be interesting to investigate these factors in a future study.

Electrophoretic Mobility

Figure 5 represents the results of electrophoretic measurements conducted on coal and quartz particles at a pH of 7.0 as a function of Dowfroth M150 addition. In this case, mobility is shown to become more negative with increasing M150 addition. This indicates that the frother adsorbs considerably on both coal and quartz, making the surface more negative.

Figure 6 shows, on the contrary, that the mobility of quartz and coal decreases slightly with increasing MIBC addition. This may indicate either that MIBC does not adsorb on coal and quartz appreciably, or that the adsorbed MIBC tends to make the surface less negative. Similar results have been obtained with Tergitol 15-S-9, as shown in Figure 7.

Streaming Current of CGA

Figure 8 shows the results of the streaming current measurements conducted on CGA prepared with frother solutions containing varying amounts of Tergitol 15-S-9 at a pH of 6.2. It is shown that, at low frother concentrations, the bubbles are slightly negatively charged, which conforms to the results obtained by Dibbs et al. (1974). The bubbles become positively charged, however, with increasing frother addition. Comparing this finding with the results of the electrophoretic measurements conducted on quartz and coal particles in the same frother solutions,

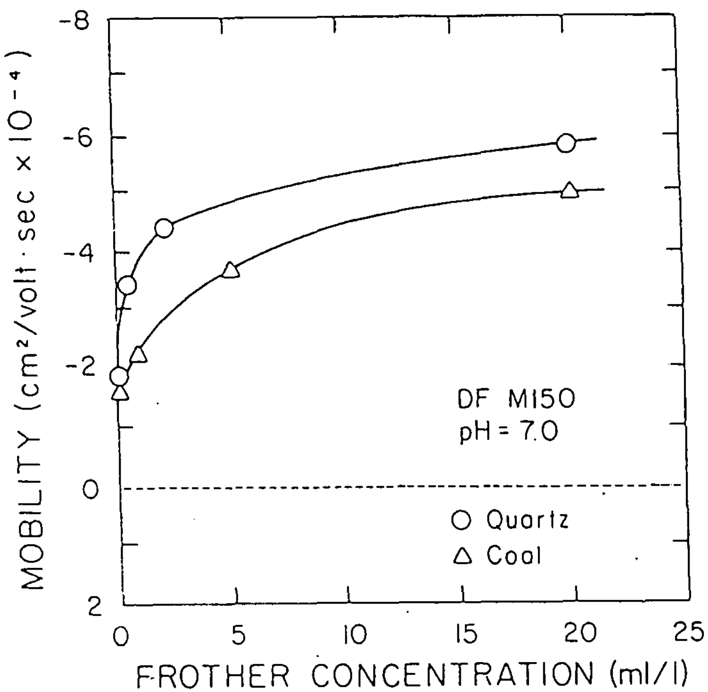


Figure 5. Electrophoretic mobility of coal and quartz particles at pH 7.0 as a function of Dowfroth M150 addition.

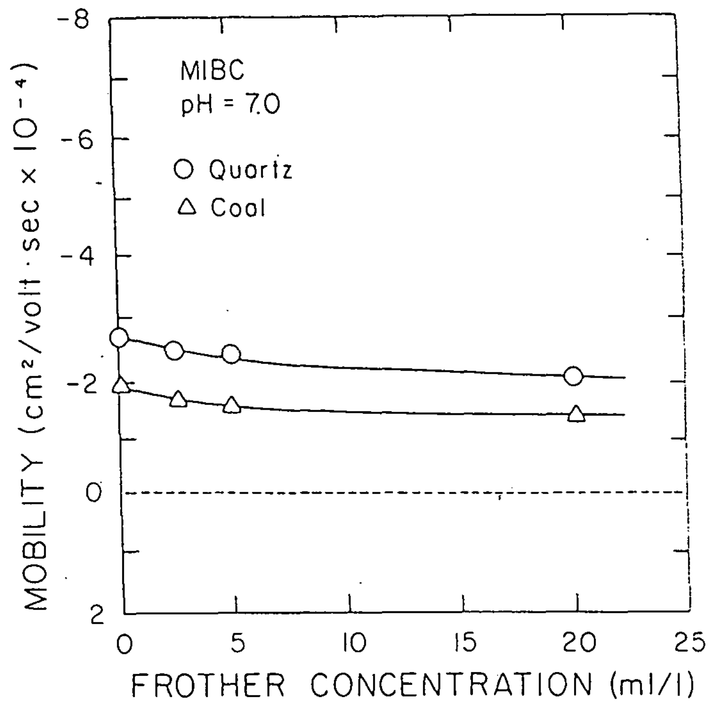


Figure 6. Electrophoretic mobility of coal and quartz particles at pH 7.0 as a function of MIBC addition.

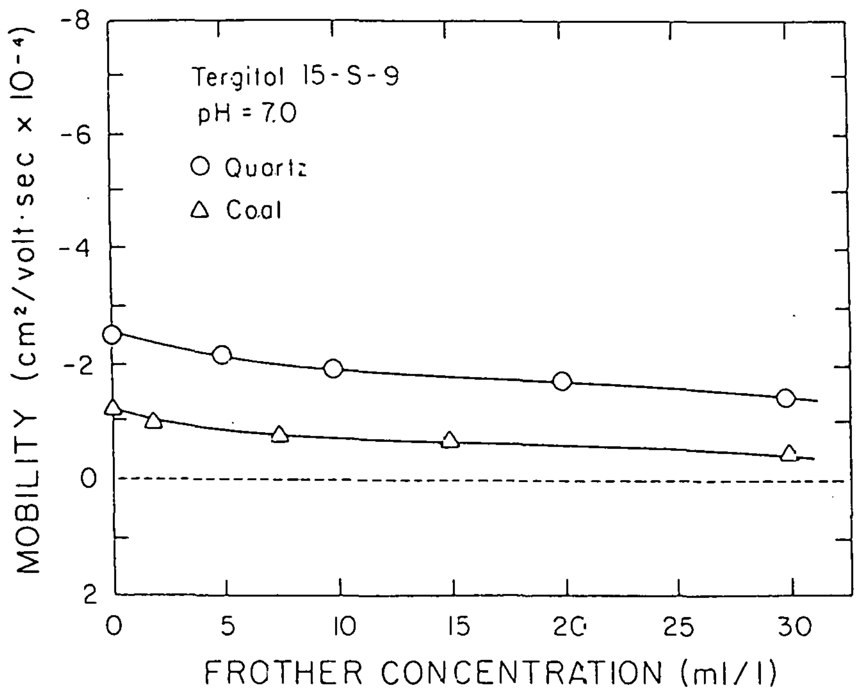


Figure 7. Electrophoretic mobility of coal and quartz particles at pH 7.0 as a function of Tergitol 15-S-9 addition.

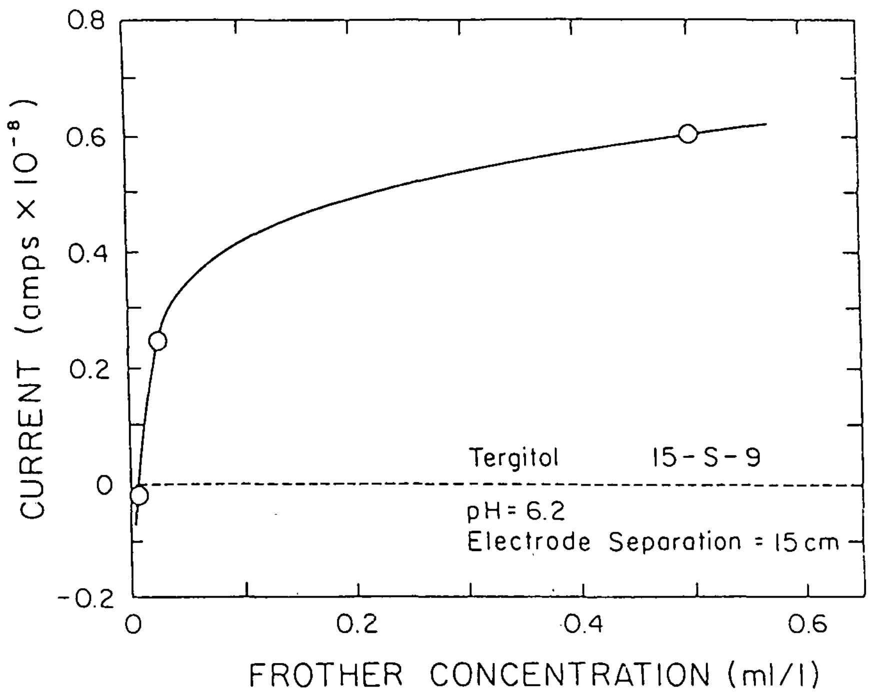


Figure 8. Results of the streaming current measurements conducted on CGA generated with varying amounts of Tergitol 15-S-9 at a pH of 6.2.

one can see that the presence of Tergitol molecules at an interface makes it more positive. This may possibly be explained considering the structure of Tergitol 15-S-9:

$$CH_3\ (CH_2)_n - \underset{\displaystyle |\atop \displaystyle O\ (CH_2 \cdot CH_2O)_q \cdot H}{CH} - (CH_2)_m \cdot CH_3$$

The H^+ (aq) ions may adsorb on the basic oxygen atoms to make the molecule positive. This will be tested by measuring the bubble charge as a function of pH. The molecular weight of this frother is 596.

Similar results have been obtained with other frothers such as Dowfroth M150 and MIBC, etc. These results are not included in this report, however, since a much better technique for measuring streaming currents has just been established in our laboratory.

Flotation Tests

a) Effect of Frothers

Figure 9 shows the results of flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated with varying amounts of Aerofroth 73. The CGA was generated using a blender in which 300 ml of tap water was mixed with a measured volume of the frother. The entire volume was then pumped into a separatory funnel containing the conditioned coal to begin the flotation. As shown, a clean coal containing less than 8% ash was obtained, but even with reagent consumption as high as 25 lb/ton, a yield of only 36% was achieved. This may indicate that Aerofroth 73 does not have collecting properties for coal flotation with this sample.

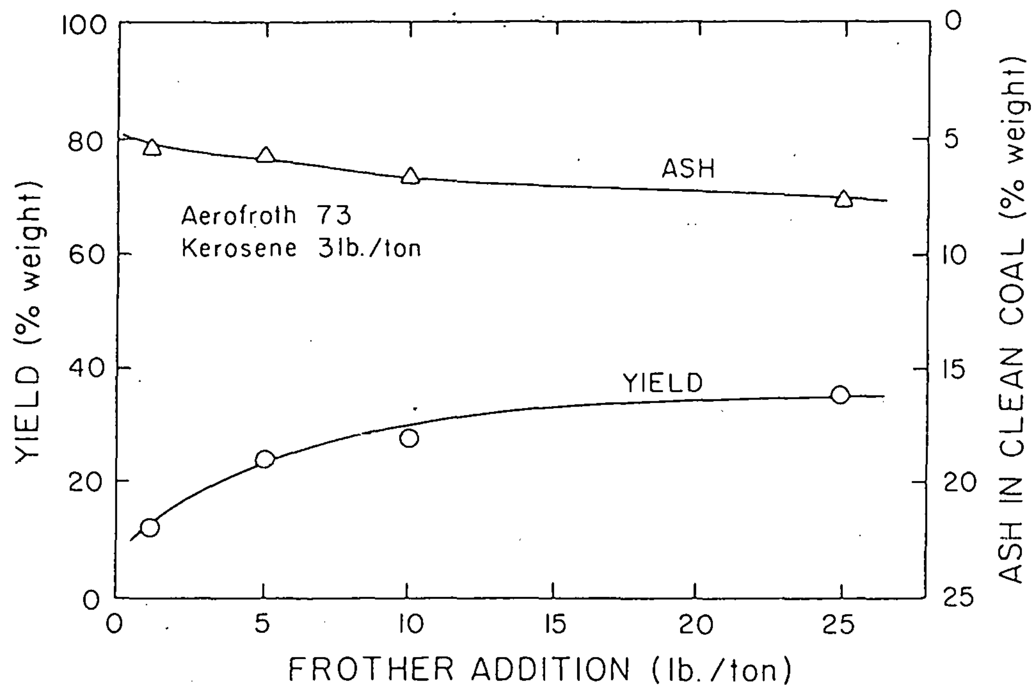


Figure 9. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of Aerofroth 73.

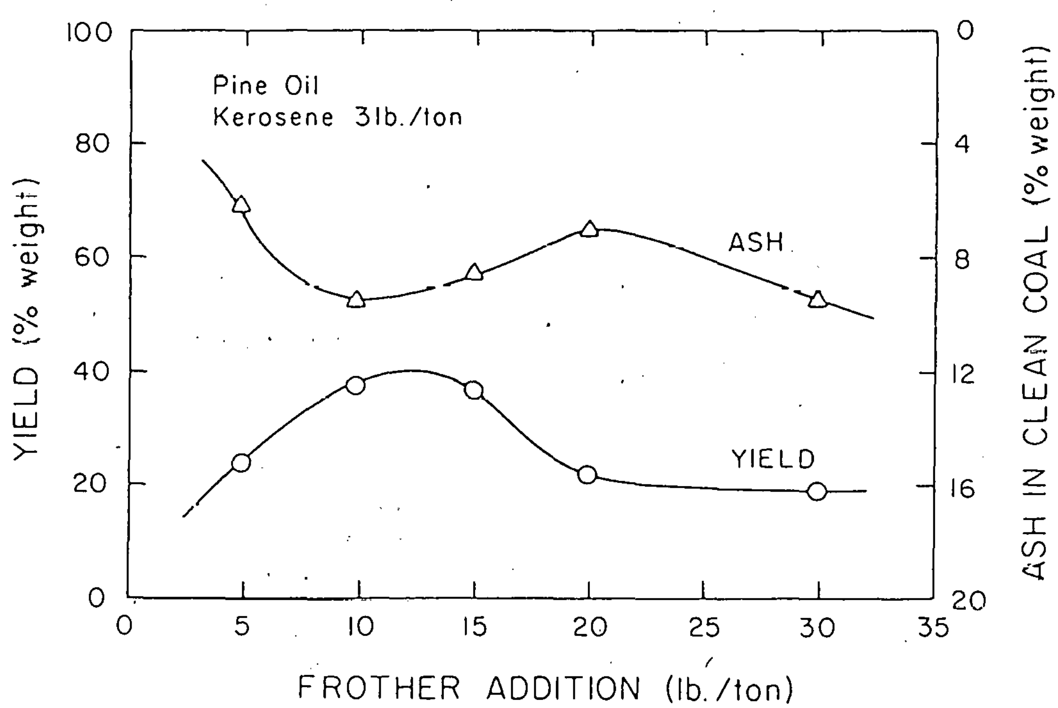


Figure 10. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of pine oil.

The next series of experiments was conducted using pine oil, and the results are shown in Figure 10. A maximum yield of 38% was obtained at approximately 12 lb/ton of pine oil, with a corresponding clean coal content of 9%. It was also found that increasing the frother addition beyond this point resulted in poor yields, due to the low stability of CGA produced with pine oil.

Similar sets of tests were made on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene with Dowfroth M210, Dowfroth E1128, and Tergitol TMN-6. These results are shown in Figures 11, 12, and 13, respectively. Dowfroth M210 gave the poorest results with yields no higher than 13% and ash contents no less than 9%. This may be attributed to the poor CGA; the color was less intense than that produced by other frothers, and during the stability measurements, no rising boundary was visible. On the contrary, Dowfroth E1128 produced one of the most stable CGA (Figure 4), and as a consequence, the yield was as high as 73%. The clean coal products contained relatively high ash, however, when more than 10 lb/ton of the frother was used. Tergitol TMN-6 also produced a very stable CGA, as shown in Figure 4, and achieved a maximum yield of 69% at 10 lb/ton. At higher frother additions, however, this yield decreased substantially. Two possible explanations for this are: (1) that the frother molecules may be inversely oriented in the second adsorbed layer with their hydrophilic polar groups pointing toward the aqueous phase, or (2) that the excess frother molecules may act as a detergent and remove adsorbed kerosene from the coal surface.

Figure 14 represents the results obtained with the R & F coal (-100 mesh) using 3 lb/ton of kerosene and varying amounts of Silicone L7001.

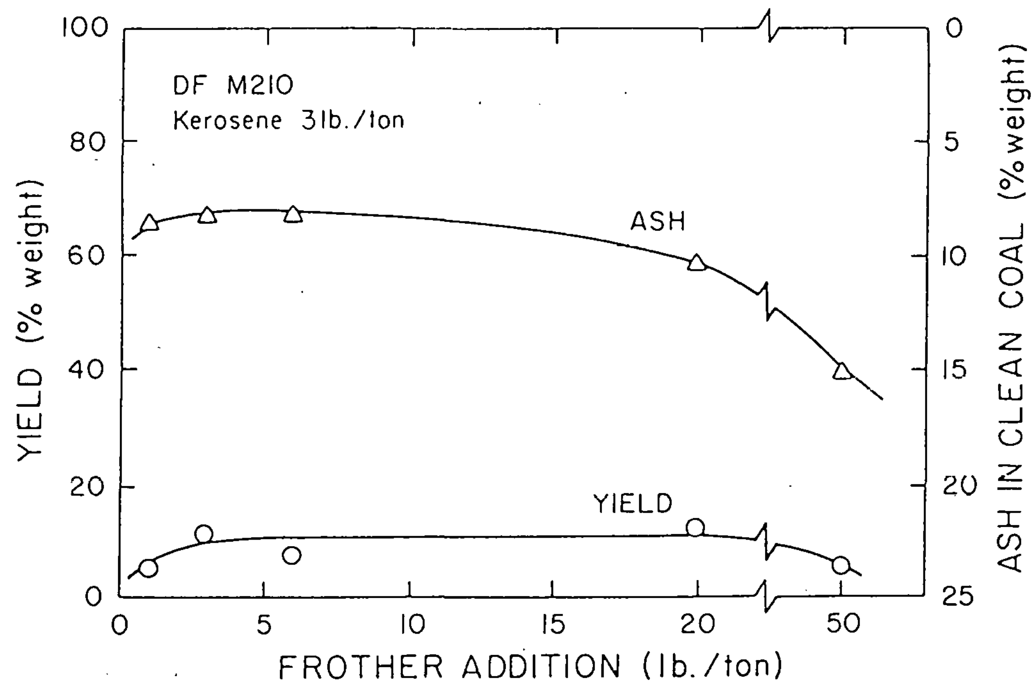


Figure 11. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of Dowfroth M210.

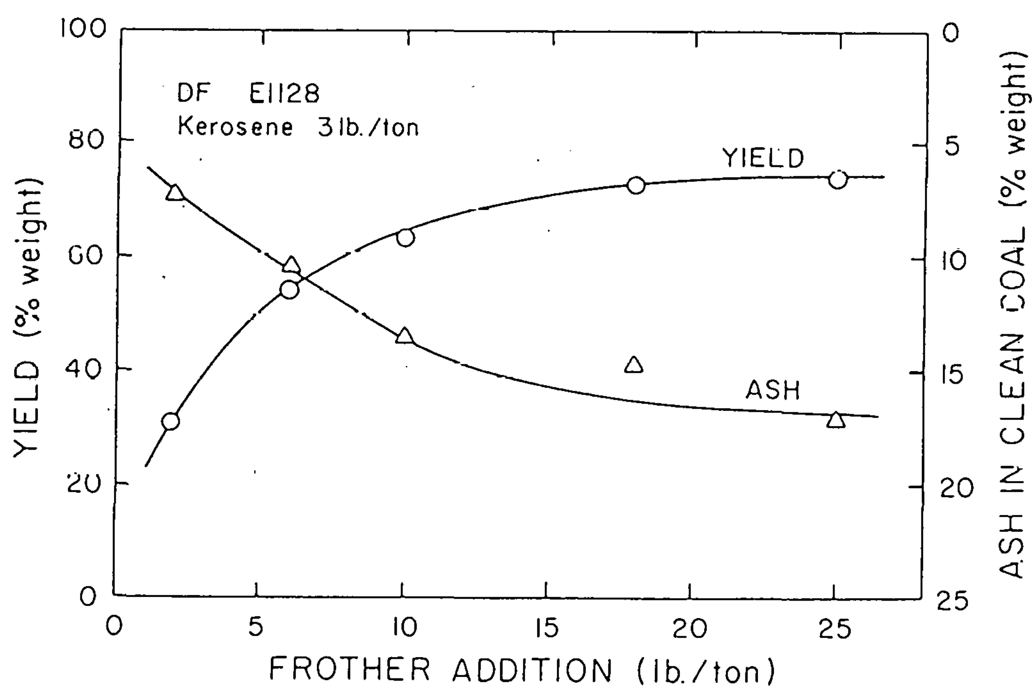


Figure 12. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of Dowfroth E1128.

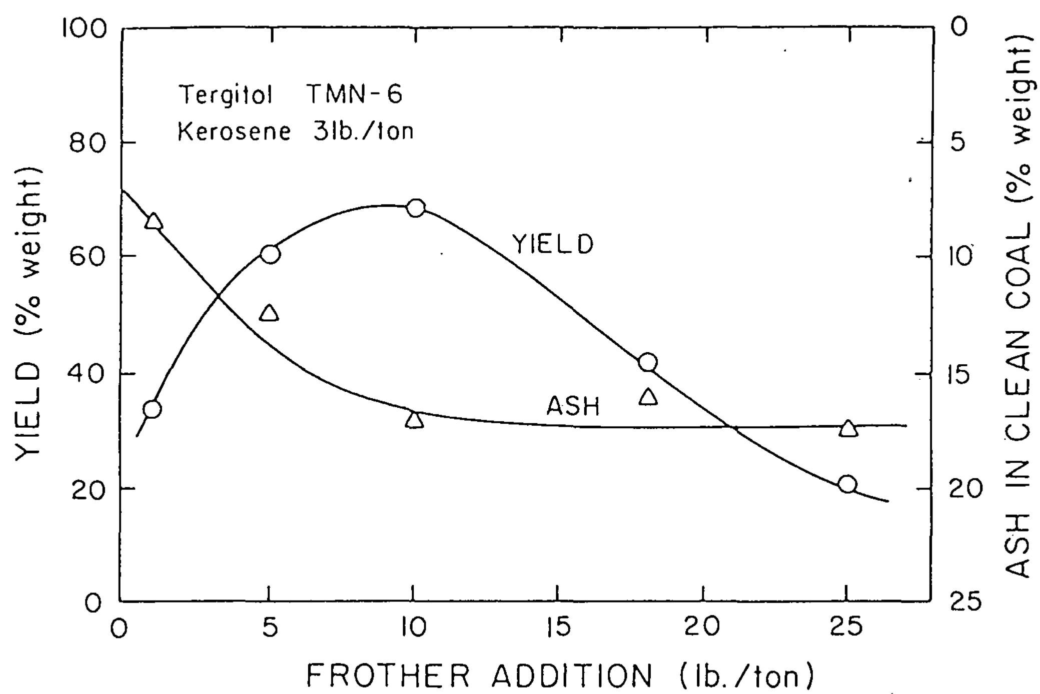


Figure 13. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of Tergitol TMN-6.

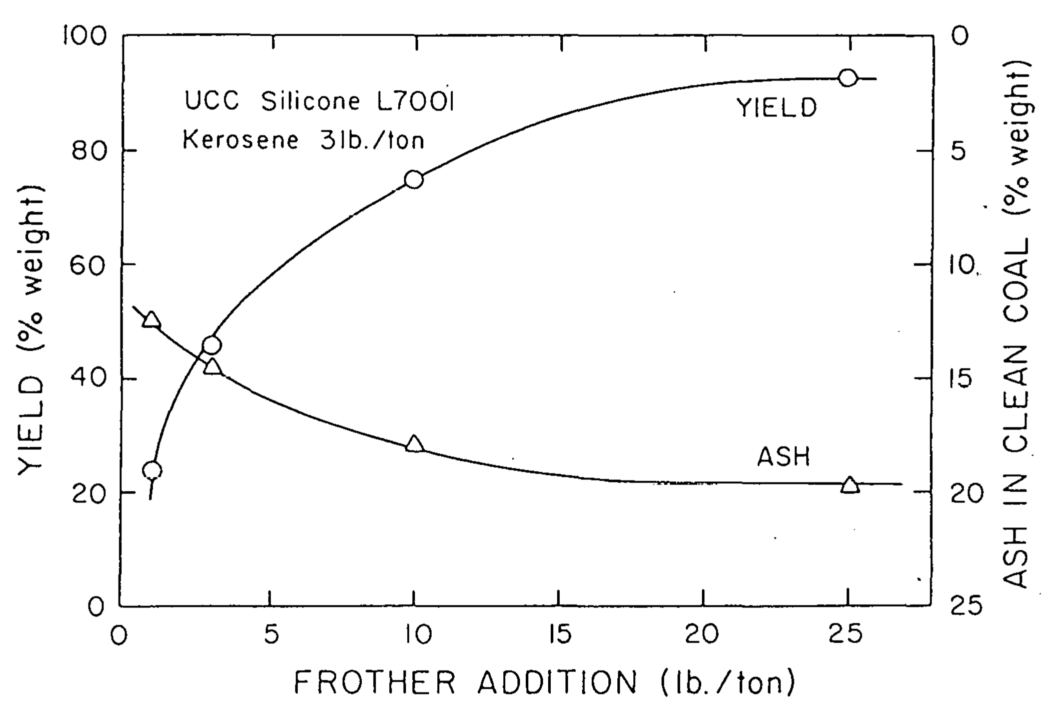


Figure 14. Results of the flotation tests conducted on the R & F coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of Silicone L7001.

According to our froth stability measurements (Figure 4), this frother produces the most stable CGA. As a result, high yields were obtained, but the froth product contained high ash.

The next few series of experiments were conducted using MIBC. Figure 15 shows the results obtained with the Eagle coal (-100 mesh) using 1 lb/ton of kerosene and varying amounts of MIBC. The maximum yield achieved was 24% at 6.3 lb/ton of frother, but the ash content of the clean coal was as low as 7%. In these tests, the CGA was produced using the aspirator technique.

Hoping to increase the yield with MIBC, the next series was conducted using 3 lb/ton of kerosene. Two sets of experiments were conducted. In one, the blender technique was used to generate CGA, and, in the other, the aspirator technique was employed. The results are given in Figures 16 and 17, respectively. (The MIBC used in the blender-generated series was from Consolidated Coal Company.) From these, it appeared that the blender-generated CGA gave better selectivity than the aspirator-generated CGA. The clean coal products obtained with the former assayed less than 5% ash, but the maximum yield was only 26% at 6 lb/ton of MIBC. With the aspirator-generated CGA, higher yields were obtained, but with correspondingly higher ash. It is rather surprising, however, that yields did not increase significantly with frother additions of up to 25 lb/ton.

More promising results were obtained with Dowfroth M150. This reagent is less selective than MIBC, but gives respectable yields. Figure 18 shows the results of two series of flotation tests conducted on the Eagle coal (-100 mesh) using 1 lb/ton of kerosene and varying amounts of frother, where the CGA was produced

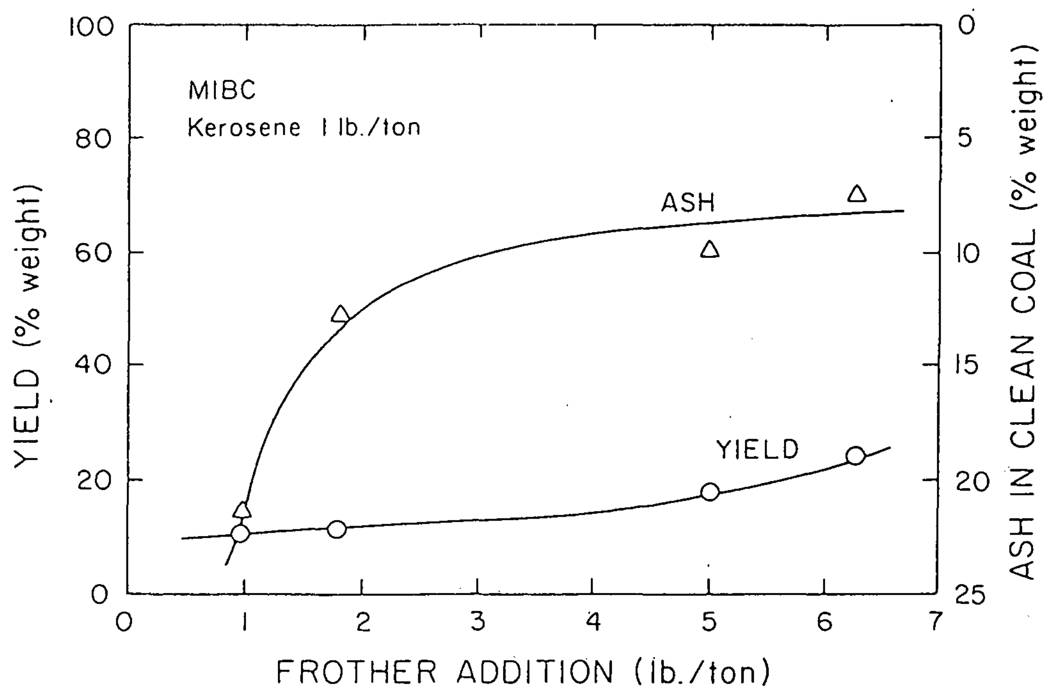


Figure 15. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 1 lb/ton of kerosene and CGA generated by the aspirator technique with varying amounts of MIBC.

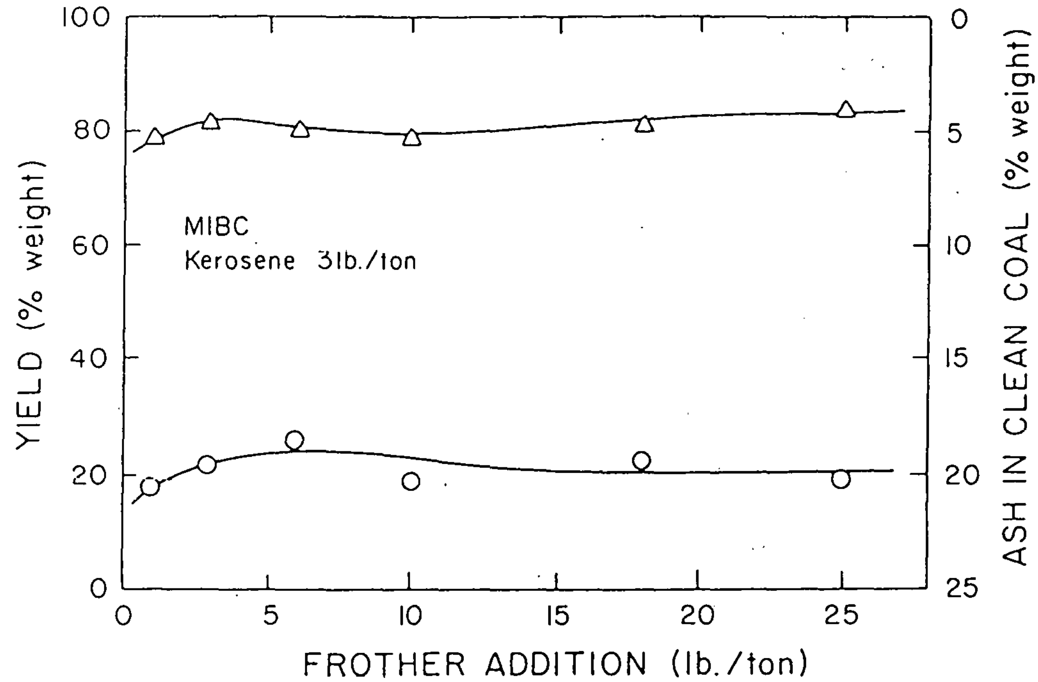


Figure 16. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of MIBC.

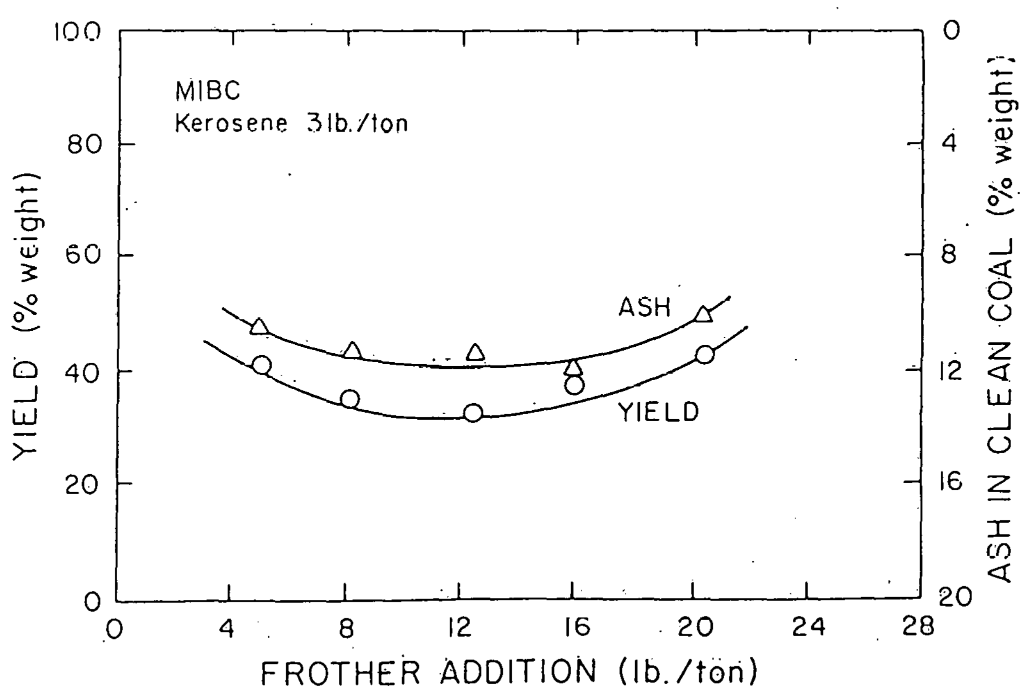


Figure 17. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the aspirator technique with varying amounts of MIBC.

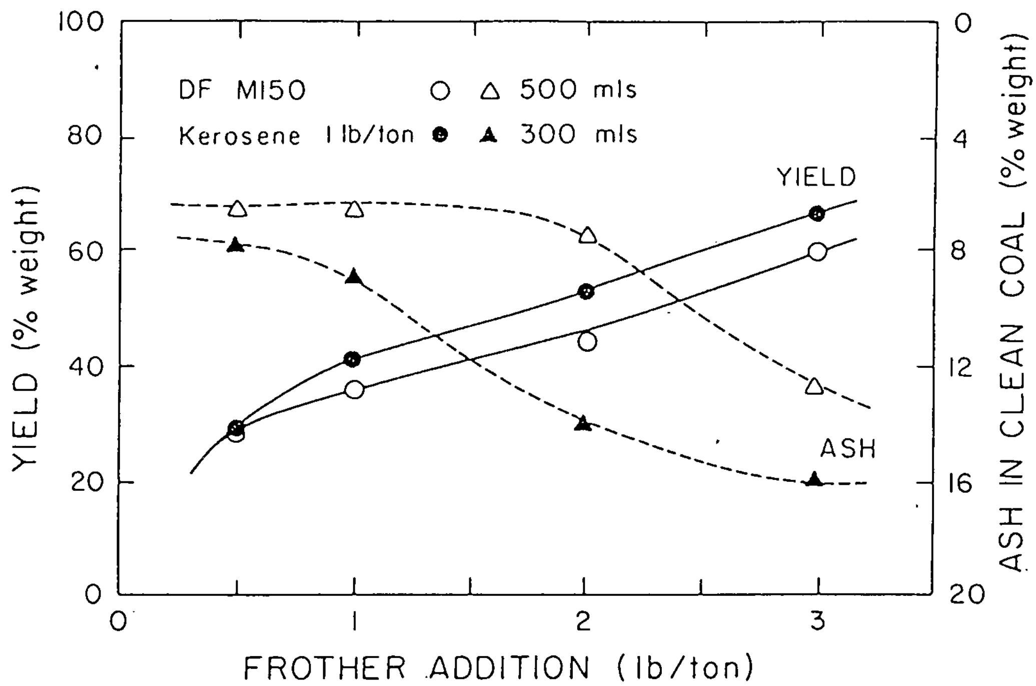


Figure 18. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 1 lb/ton of kerosene and CGA generated by the blender technique with varying amounts of Dowfroth M150. In each series, the volume of CGA injected into the separatory funnel was varied.

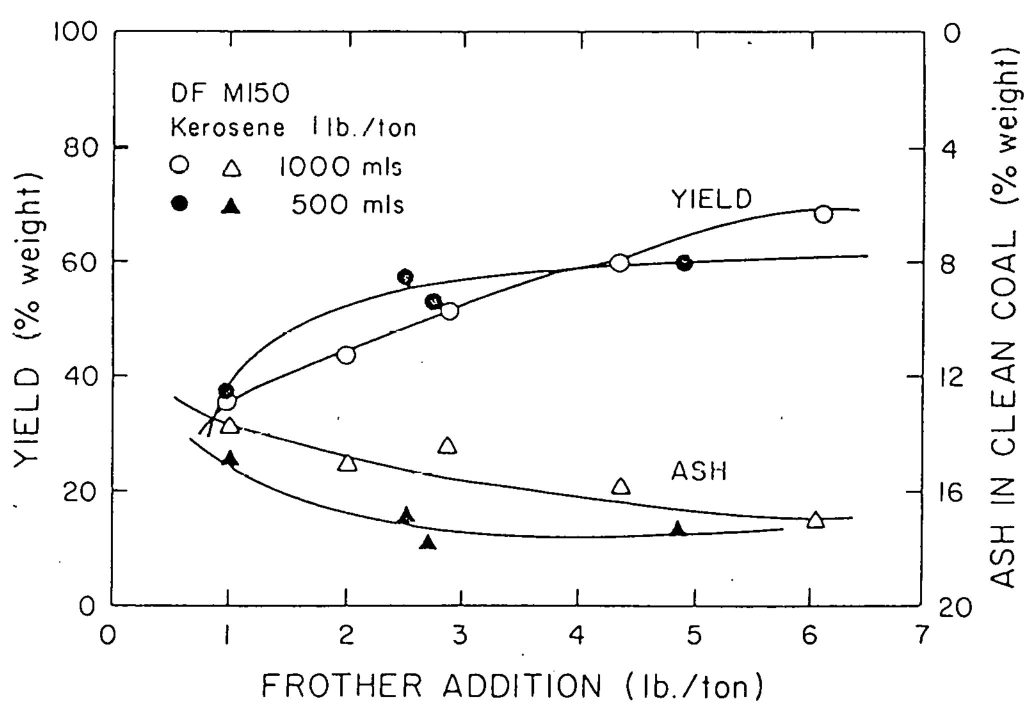


Figure 19. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using 1 lb/ton of kerosene and CGA generated by the aspirator technique with varying amounts of Dowfroth M150. In each series, the volume of CGA injected into the flotation cell was varied.

by the blender technique. In one series, a total of 300 ml of CGA was injected into the flotation cell, and in the other, 500 ml of CGA was used. It should be noted that the CGA of the former series was more stable than those of the latter since the frother solutions were at a higher concentration for a given frother addition.

These two sets of experiments produced remarkably different results. The yields were higher by about 5% with 300 ml of CGA, but the ash contents of the clean coal products were significantly lower when 500 ml of CGA was used. For example, at 2 lb/ton of Dowfroth M150, the clean coal product obtained with 300 ml of CGA contained approximately 8% lower ash than that obtained with 500 ml of CGA. This suggests that when using a less stable CGA, mechanical entrapment is less likely to occur, which explains why MIBC has proven to be the most selective frother.

Figure 19 shows similar results obtained using CGA produced by the aspirator technique. The beneficial effect of using more dilute frother solutions, and, hence, less stable CGA was less dramatic than shown in Figure 18, but is still evident.

Dowfroth M150 was also tested with the R & F coal (-100 mesh). The results shown in Figure 20 were obtained by using 3 lb/ton of kerosene and CGA produced using the aspirator technique. A maximum yield of 87% was obtained at 6.3 lb/ton of frother addition with 16% ash in the clean coal. The ash content remained relatively constant at frother additions higher than 1 lb/ton, while the yield increased steadily. The yield achieved with the R & F coal was higher than that with the Eagle coal, primarily due to its lower feed ash content.

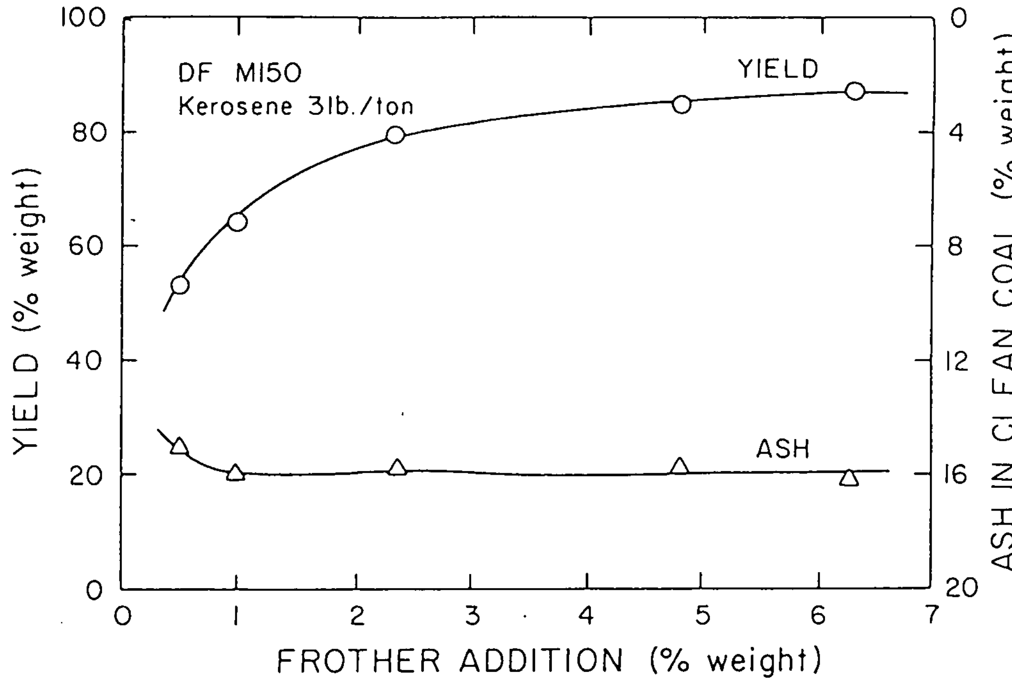


Figure 20. Results of the flotation tests conducted on the R & F coal (-100 mesh) using 3 lb/ton of kerosene and CGA generated by the aspirator technique with varying amounts of Dowfroth M150.

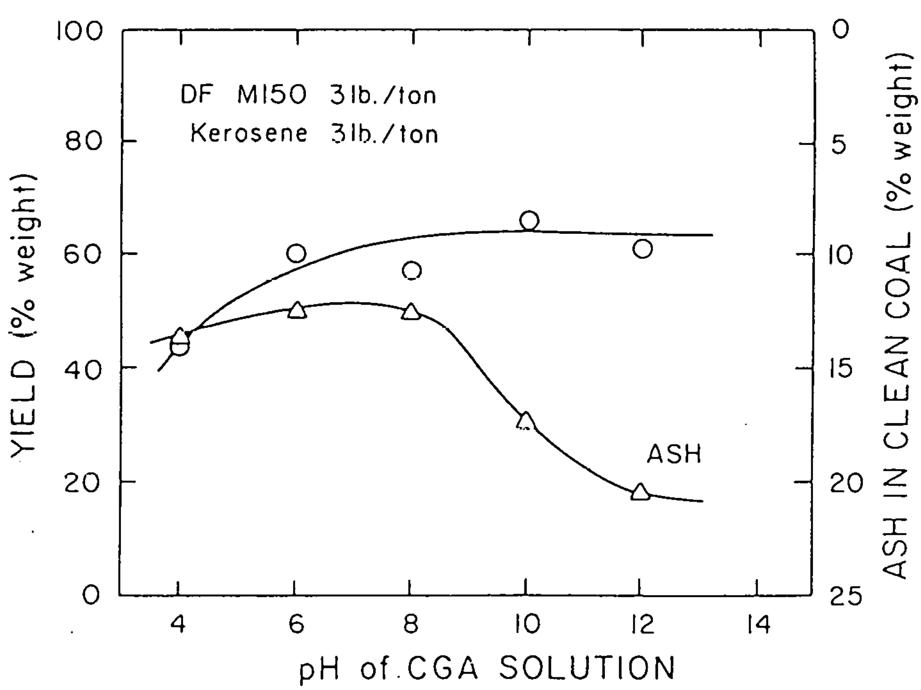


Figure 21. Effect of the pH of the CGA on the flotation of the Eagle coal (-100 mesh) using 3 lb/ton of kerosene. The CGA was generated by the blender technique using 3 lb/ton of Dowfroth M150.

b) Effect of pH

Figure 21 represents the results of flotation tests conducted to investigate the effect of pH on the CGA. The tests were made on the Eagle coal (-100 mesh) using 3 lb/ton of Dowfroth M150 and 3 lb/ton of kerosene. The coal samples were conditioned with kerosene at natural pH, and the CGA was prepared at various pH's using the blender technique. The yield was relatively constant above a pH around 6, while the ash content of the clean coal increased substantially at alkaline pH values. Presumably, this might be due to the possibility that the CGA produced by Dowfroth M150 is less stable at lower pH values. As shown in the foregoing section (Figure 18), selectivity improves when more dilute, or less stable, CGA is used for flotation. In this regard, an investigation of the effect of pH on the stability of CGA is planned.

Effect of Collector Additions

It has been established that Dowfroth M150 gives high yields with a relatively low ash clean coal. MIBC is more selective, but the yields are relatively low. To investigate the effect of collector additions, a stable CGA was desired, and, therefore, Dowfroth M150 was chosen as the frother.

Figure 22 shows the results of the two-stage flotation tests conducted on the Eagle coal (-100 mesh) as a function of kerosene addition. The CGA was generated with the aspirator technique, using 6 lb/ton of Dowfroth M150 in the first stage and 3 lb/ton in the second. A maximum yield of 72% was obtained at 6 lb/ton or more of kerosene. An approximate loss of 5% in yield occurred with the second cleaning, but

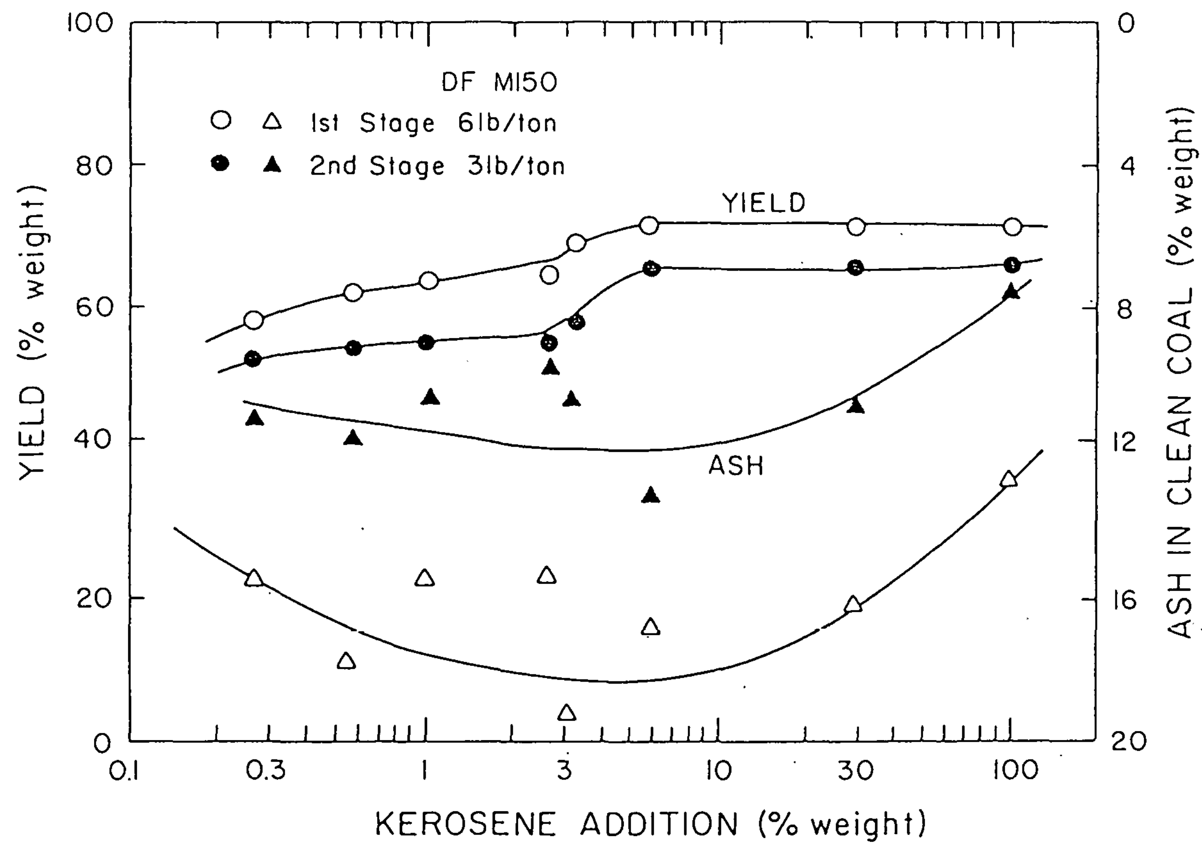


Figure 22. Results of the two-stage flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the aspirator technique with 6 lb/ton of Dowfroth M150 in the first stage and 3 lb/ton in the second. The coal was conditioned with varying amounts of kerosene.

at the same time, the ash content of the clean coal was reduced by approximately 5-6%. Note that the ash content dropped significantly above 10 lb/ton of kerosene addition, which may indicate that an oil agglomeration mechanism begins to operate in this region.

A similar series of experiments has been performed on the same sample using CGA produced by the blender technique. These results are shown in Figure 23. The loss of yield with the second stage flotation is considerably less in this series than that shown in Figure 22, as is the ash rejection. This may be attributed to the experimental procedure: in the aspirator-generated CGA flotation tests, larger volume of less concentrated CGA was used with a lower pulp density in the flotation cell. These results again demonstrate that use of more dilute CGA provides better selectivity. Surprisingly, with the blender-generated flotation tests (Figure 23), the oil agglomeration effect is not evident at high kerosene additions.

Figure 24 gives the results of a series of flotation tests conducted on the Eagle coal (-100 mesh) using No. 2 diesel oil as the collector. CGA was generated using the blender technique with 6 lb/ton of Dowfroth M150. As compared to the test results obtained using kerosene (Figure 23), slightly higher yields (by about 2-3%) were obtained with a concurrent increase in the ash content of the clean coal. This suggests that No. 2 diesel oil is a more powerful collector than kerosene, and, as such, selectivity is reduced. Unlike with the aspirator-generated flotation tests (Figure 22), no agglomeration effect was visible at higher collector additions, and, in fact, the ash content of the clean coal increased above 3 lb/ton. If tests had been conducted at kerosene additions

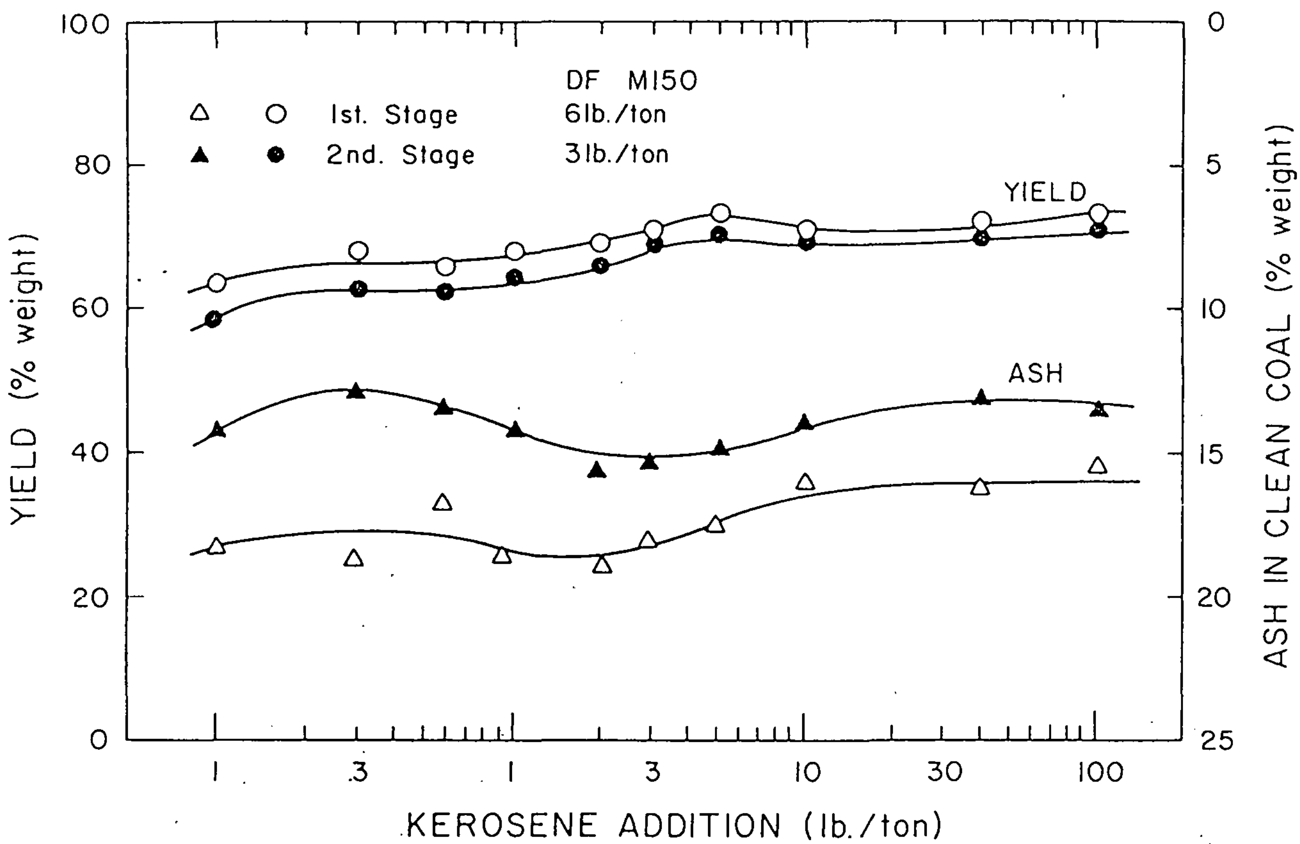


Figure 23. Results of the two-stage flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 6 lb/ton of Dowfroth M150 in the first stage and 3 lb/ton in the second. The coal was conditioned with varying amounts of kerosene.

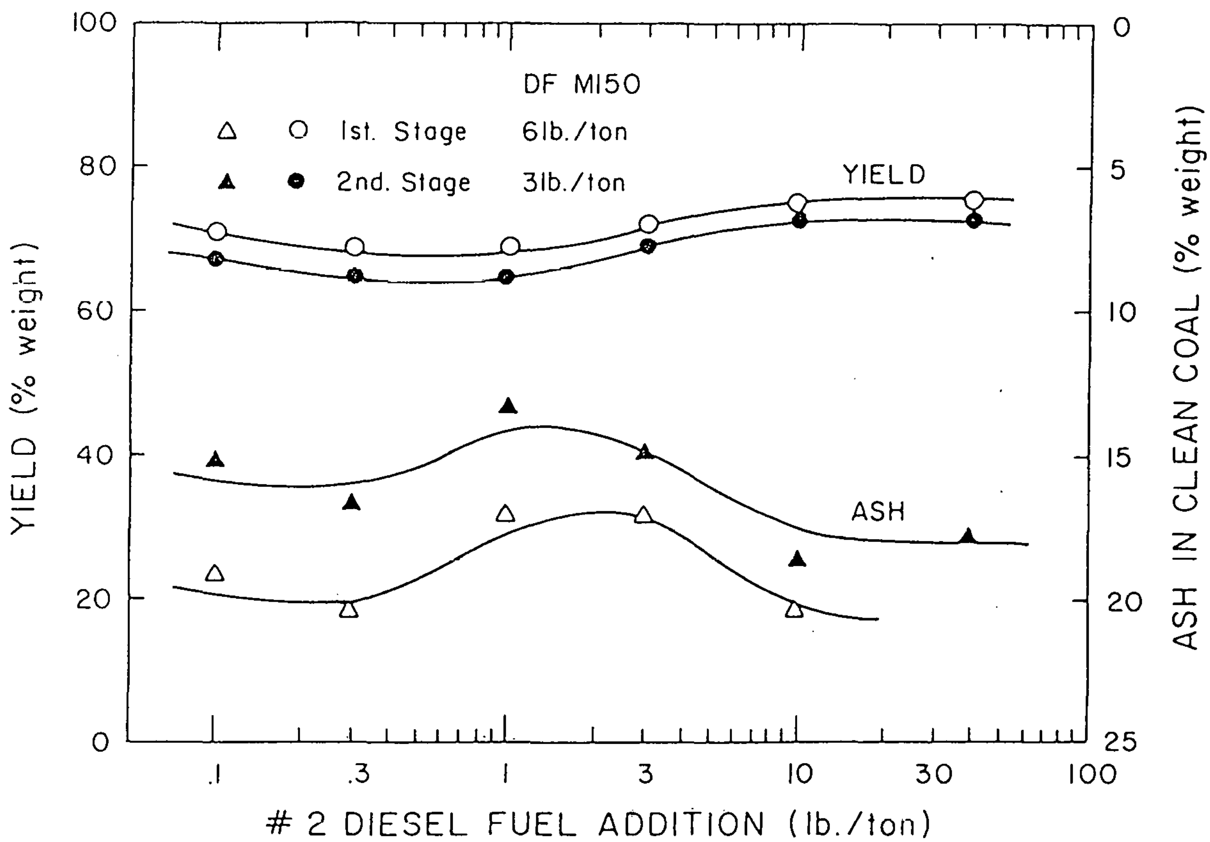


Figure 24. Results of the two-stage flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 6 lb/ton of Dowfroth M150 in the first stage and 3 lb/ton in the second. The coal was conditioned with varying amounts of No. 2 diesel fuel.

higher than 40 lb/ton, however, the agglomeration effect might have become noticeable.

A similar series of flotation tests was conducted on the R & F coal sample (-100 mesh) to investigate the effect of kerosene addition. These results are shown in Figure 25. CGA was produced using the aspirator technique with 3 lb/ton of Dowfroth M150 in the first stage and 1.5 lb/ton in the second. It is shown that ash rejection is improved by 2-3% with the second stage flotation. The yields are higher as compared to those of the Eagle coal due to the lower feed ash content of the R & F coal.

Effect of Kerosene Additions to CGA

It was thought that the CGA could be coated by hydrocarbon oils, such as kerosene. By using the oil-coated CGA, one could then reduce the consumption of oil during flotation. Attempts were made to oil-coat the bubbles by adding kerosene to the blender in which the CGA was being generated.

Figure 26 shows the results obtained by using a total of 3 lb/ton of kerosene. In each experiment, different proportions of the kerosene were added directly to the coal during conditioning and to the CGA in the blender. The Eagle coal sample (-100 mesh) was used and Dowfroth M150 addition was kept constant at 6 lb/ton. A fairly uniform yield was obtained, but the ash content of the clean coal appeared to increase when all of the 3 lb/ton of kerosene was added to the CGA.

The next series of experiments was made by adding all of the kerosene to the CGA, again using the Eagle coal (-100 mesh) and 6 lb/ton of Dowfroth M150. The kerosene addition was increased to a maximum of 30 lb/ton,

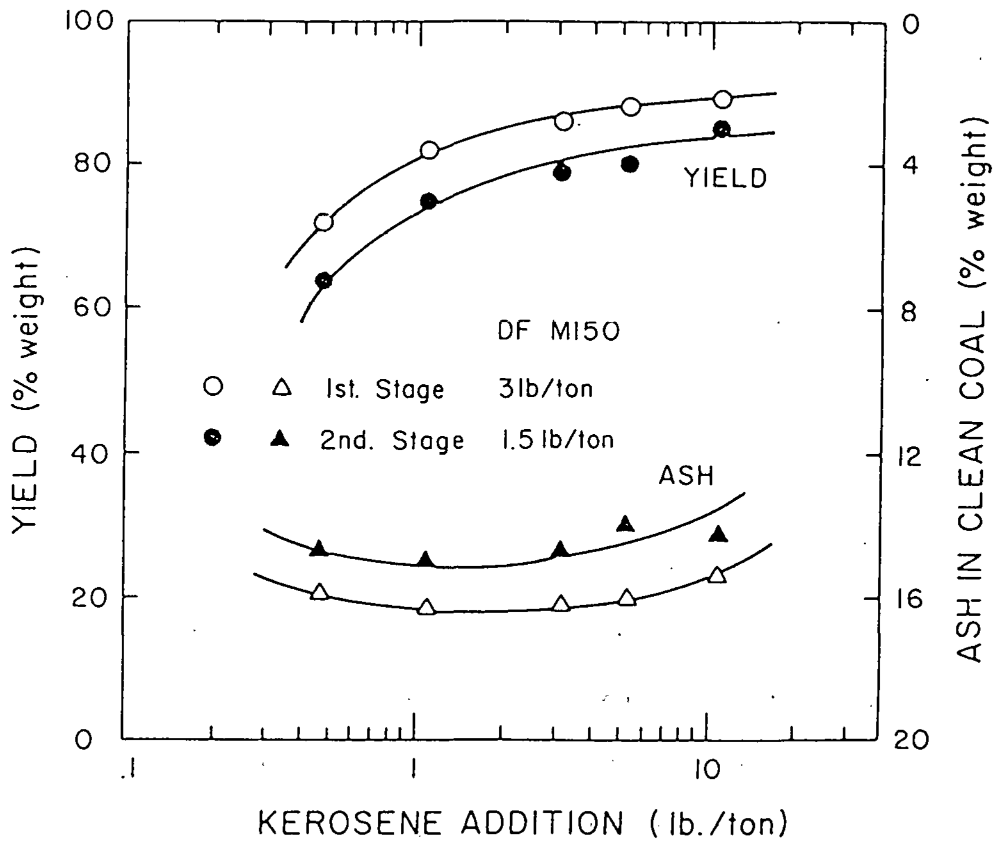


Figure 25. Results of the two-stage flotation tests conducted on the R & F coal (-100 mesh) using CGA generated by the aspirator technique with 3 lb/ton of Dowfroth M150 in the first stage and 1.5 lb/ton in the second. The coal was conditioned with varying amounts of kerosene.

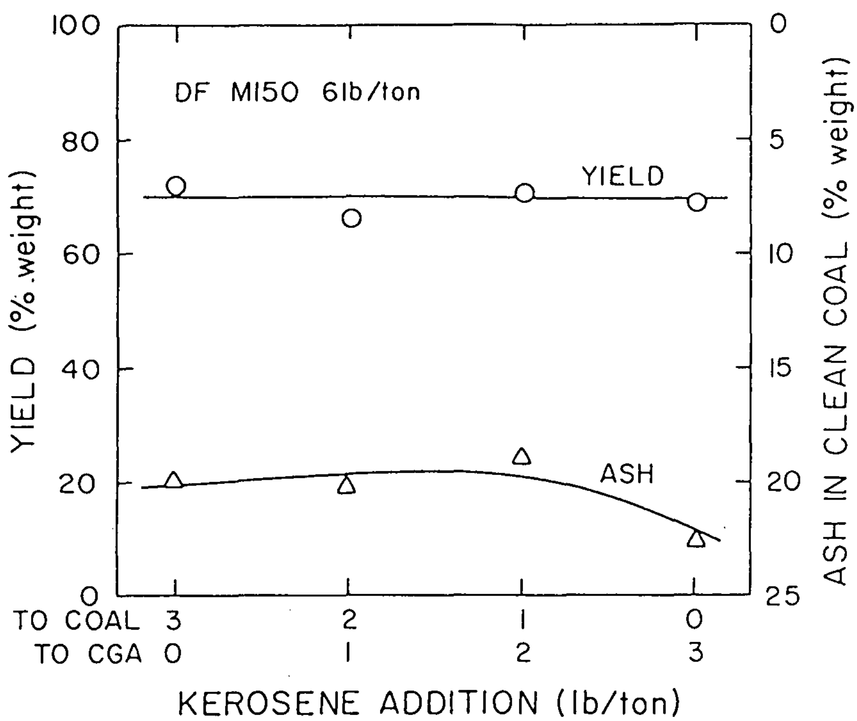


Figure 26. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 6 lb/ton of Dowfroth M150. A total of 3 lb/ton of kerosene was used, with varying amounts being distributed between the conditioning coal and the CGA solution.

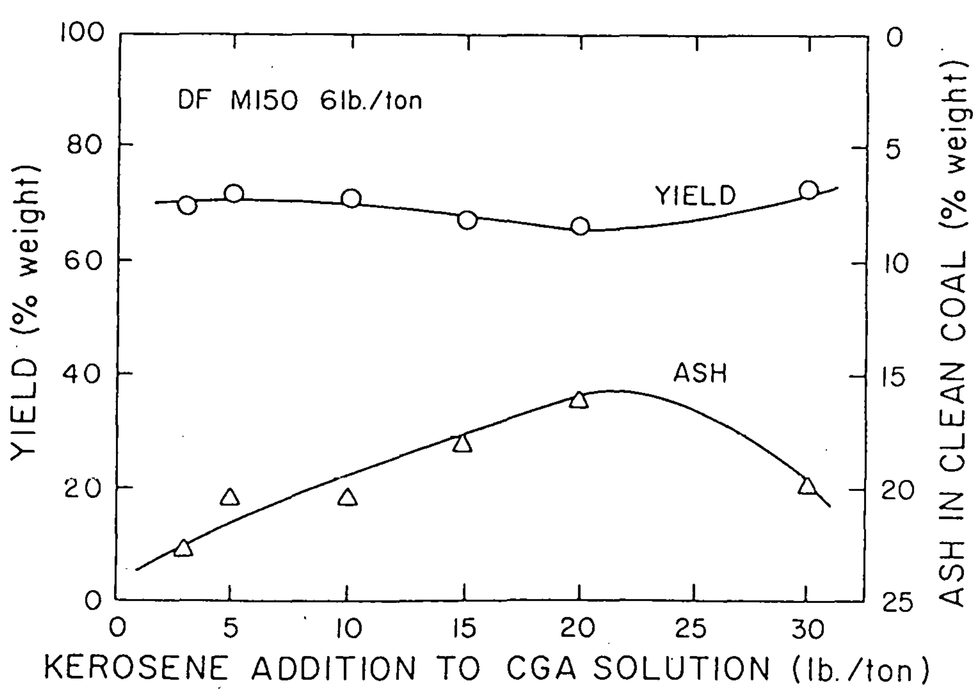


Figure 27. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 6 lb/ton of Dowfroth M150. The amount of kerosene added to the CGA solution was varied.

however. Ash rejection improved progressively with increasing kerosene additions up to 20 lb/ton with only a slight loss of yield. At higher dosages, both the yield and the ash of the clean coal increased.

Synergistic Effect of Frother Combinations

The results presented thus far indicate that MIBC was the most selective, i.e., that the clean coal products assayed low ash, but at the expense of the yield. On the other hand, Dowfroth M150 produced high yields but with higher ash contents. It was, therefore, thought that a combined use of these two reagents would produce a synergistic effect.

Figure 28 shows the results obtained by using 6 lb/ton of Dowfroth M150 and varying amounts of MIBC. The CGA was produced using the blender technique. The -100 mesh Eagle coal samples were conditioned with 6 lb/ton of kerosene. A comparison of these results with those obtained using 6 lb/ton of Dowfroth M150 and 6 lb/ton of kerosene alone indicates that the use of MIBC produced a considerably lower ash (by about 2%) clean coal product. Further improvements were made when the MIBC was added to the coal during conditioning, as shown in Figure 29. Ash rejection was improved by about 2% with virtually no decrease in yields.

It was thought that in these experiments the 6 lb/ton of Dowfroth M150 was overpowering the beneficial effect of the MIBC addition, and, thus, the next series of flotation tests was conducted with 6 lb/ton MIBC and varying amounts of Dowfroth M150. The Eagle coal (-100 mesh) was conditioned with 6 lb/ton of kerosene, and the results are shown in

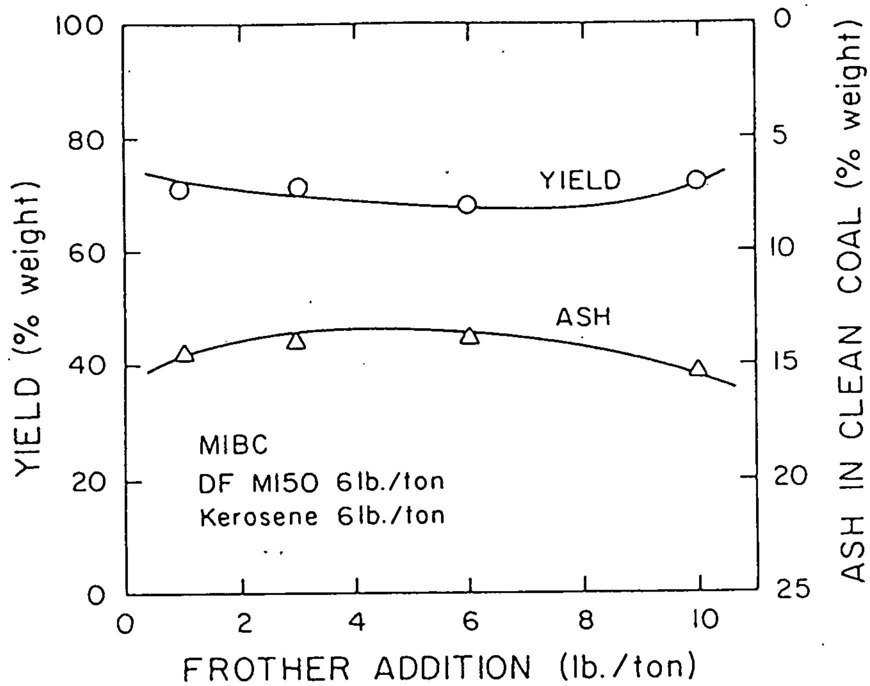


Figure 28. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 6 lb/ton of Dowfroth M150 and varying amounts of MIBC. The coal was conditioned with 6 lb/ton of kerosene.

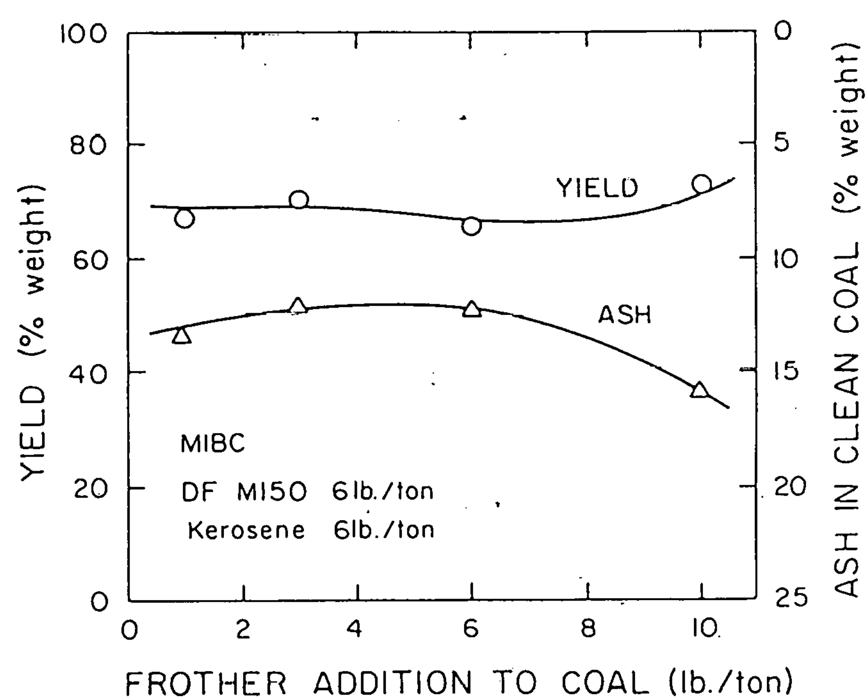


Figure 29. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 6 lb/ton of Dowfroth M150. The coal was conditioned with 6 lb/ton of kerosene and varying amounts of MIBC.

Figure 30. Yields remained fairly constant when using more than 3 lb/ton of Dowfroth M150, while the ash content of the clean coal increased steadily with increasing Dowfroth M150 addition throughout the range tested. The ash content of the clean coal increased by 5% as the frother addition was increased from 1 to 6 lb/ton.

The next series of experiments was conducted on the same coal with only 3 lb/ton of Dowfroth M150 and varying amounts of MIBC. The collector addition was also reduced to 3 lb/ton. In an attempt to maintain respectable yields, however, the more powerful No. 2 diesel oil was used. Figure 31 gives the results. As compared to the results obtained with twice as much reagent addition (Figure 28), the froth products contained less ash with only a slight loss of yield. Encouraged by this improved selectivity, the next series of experiments was conducted with as little as 1 lb/ton of Dowfroth M150 and varying amounts of MIBC. As shown in Figure 32, the froth products assayed only 9% ash at the most, while maintaining 50% yields.

Effect of Particle Size

Figure 33 represents the results of the flotation tests conducted on various size fractions of the Eagle coal as obtained by dry screening. The CGA was produced by the aspirator technique using 6 lb/ton of Dowfroth M150 and each coal sample was conditioned with 3 lb/ton of kerosene. As shown, the yield decreased drastically below 200 mesh. The improvement in ash rejection between 75 and 40 μm may be due to the high degree of liberation of ash particles from the coal and also to the simple fact that, when yields are low, the more floatable coal particles,

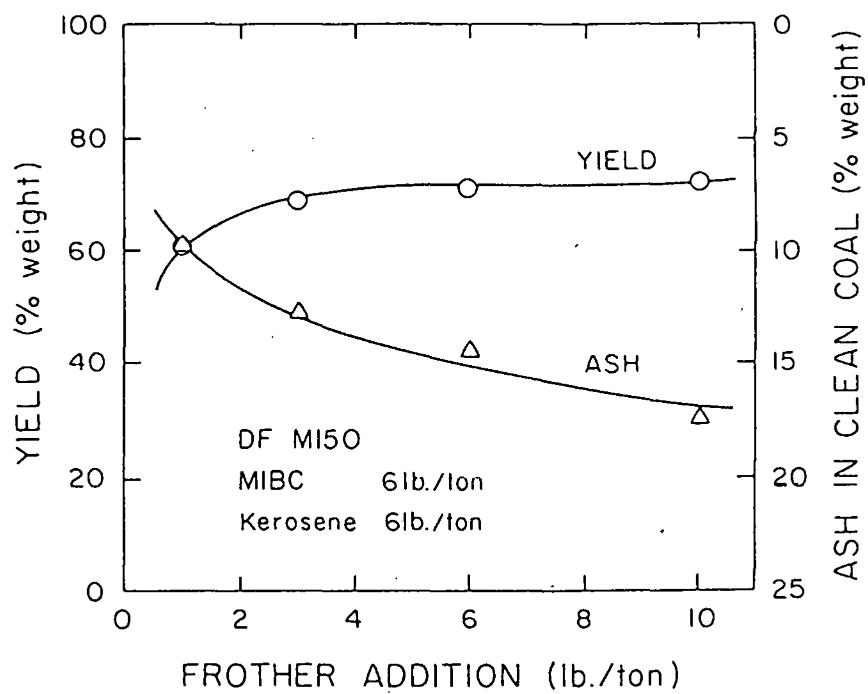


Figure 30. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 6 lb/ton of MIBC and varying amounts of Dowfroth M150. The coal was conditioned with 6 lb/ton of kerosene.

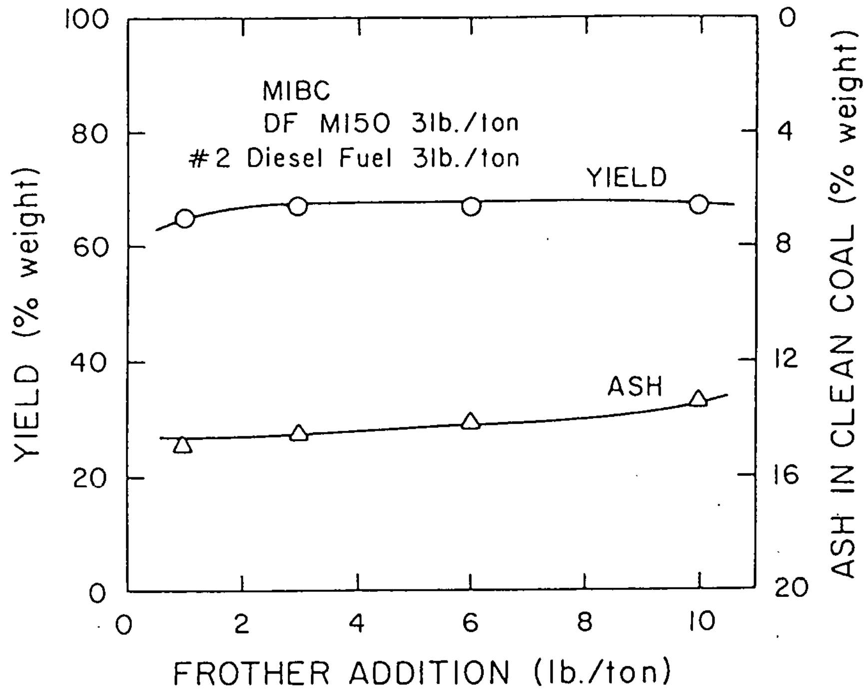


Figure 31. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 3 lb/ton of Dowfroth M150 and varying amounts of MIBC. The coal was conditioned with 3 lb/ton of No. 2 diesel fuel.

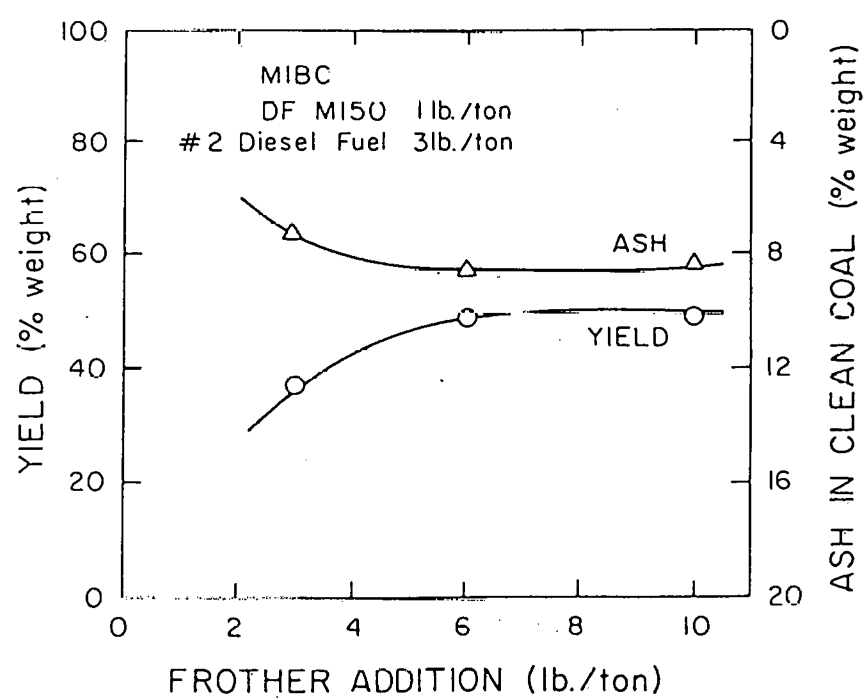


Figure 32. Results of the flotation tests conducted on the Eagle coal (-100 mesh) using CGA generated by the blender technique with 1 lb/ton of Dowfroth M150 and various amounts of MIBC. The coal was conditioned with 3 lb/ton of No. 2 diesel fuel.

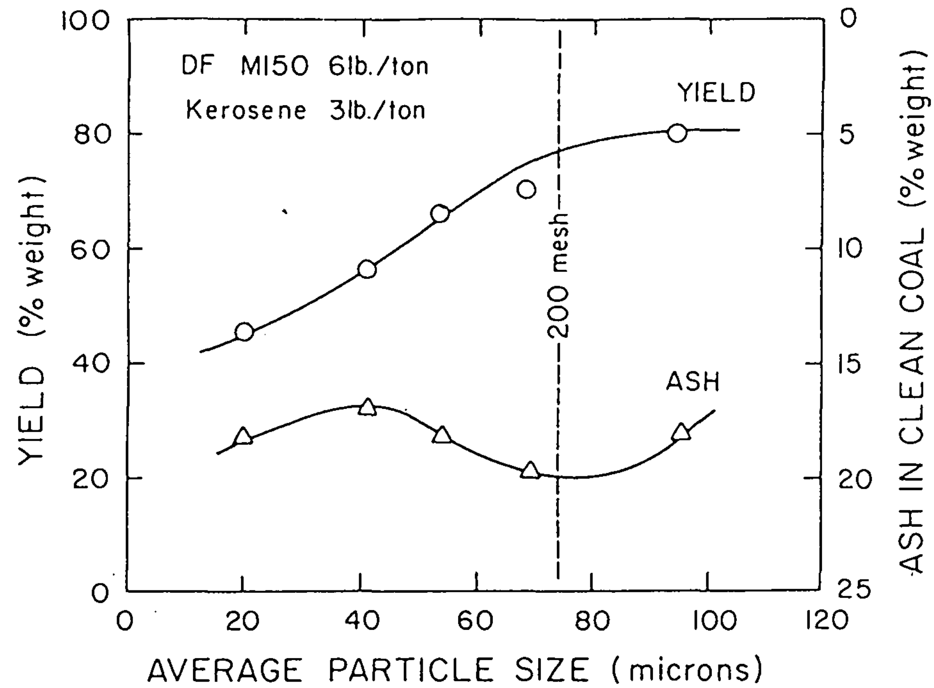


Figure 33. Results of the flotation tests conducted on various size fractions of the Eagle coal (-100 mesh) using CGA generated by the aspirator technique with 6 lb/ton of Dowfroth M150. Each coal sample was conditioned with 3 lb/ton of kerosene.

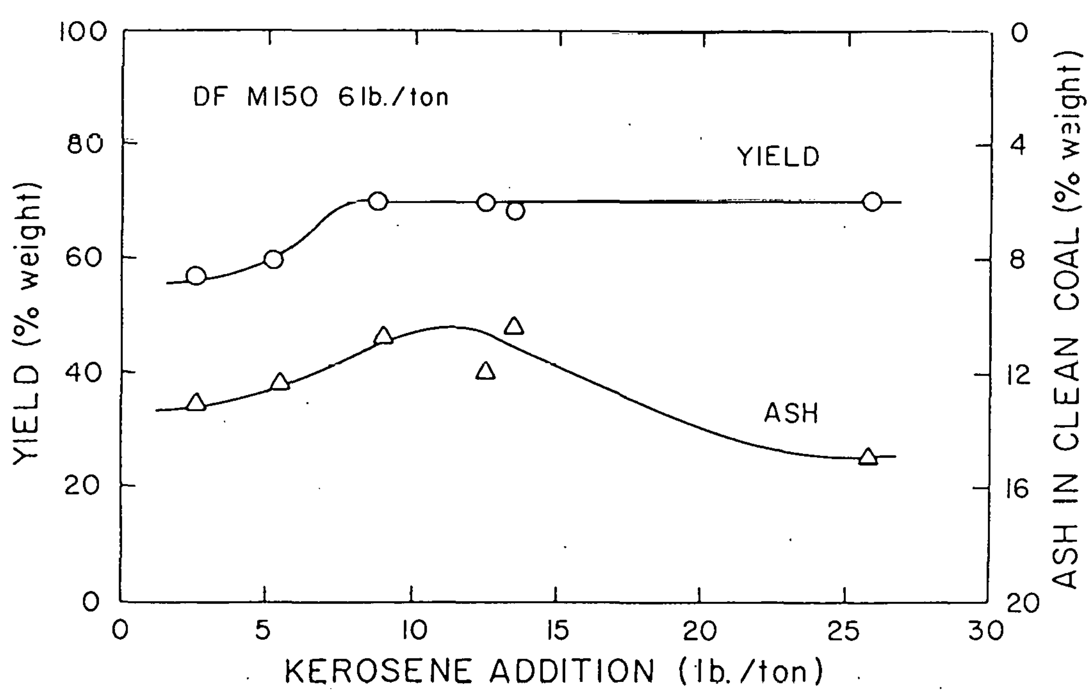


Figure 34. Results of the flotation tests conducted on the Eagle coal wet-ground for 4 hours (99% -400 mesh) using CGA generated by the aspirator technique with 6 lb/ton of Dowfroth M150. The coal samples were conditioned with varying amounts of kerosene.

e.g., those containing less ash, are floated. The increasing ash content below 40 μm may be ascribed to the fact that the feed ash increased with decreasing particle size.

In order to demonstrate the beneficial effect of using CGA for ultrafine particle flotation, tests were made with Eagle coal samples wet-ground for 4 hours in a pebble mill. Wet-screen analysis revealed that 99% of the sample, thus prepared, passed a 400 mesh screen. The flotation tests were made using CGA generated by the aspirator technique. Each test was made using 6 lb/ton of Dowfroth M150 and a varying amount of kerosene. A maximum yield of approximately 70% was obtained when using more than 8 lb/ton of kerosene. The best results were obtained when 8 to 14 lb/ton of kerosene were used. Under these conditions, the ash content of the clean coal was as low as 11%.

For comparative purposes, conventional flotation tests were conducted on the 4-hour ground sample using a Denver laboratory flotation machine. Two tests were made using 8 and 12 lb/ton of kerosene, respectively. Only 1 lb/ton of Dowfroth M150 was used in each test, as this amount has been determined to be adequate for conventional flotation tests. The results given in Tables II and III demonstrate that the froth product of the conventional flotation tests contained almost twice as much ash as those of the CGA flotation tests. Note also that in the conventional tests approximately 60% of the feed ash is rejected, while almost 80% of the ash is rejected with CGA flotation.

Table II. Results of a Conventional Flotation Test Using 1 lb/ton Dowfroth M-150 and 8 lb/ton Kerosene Conducted on an Eagle Coal Sample Wet-Ground for Four Hours.

Products	Weight (%)	Analysis (%) Ash	Analysis (%) Coal	Distribution (%) Ash	Distribution (%) Coal
Clean Coal	70.8	21.1	78.9	39.7	89.6
Refuse	29.2	77.8	22.2	60.3	10.4
Feed	100.0	37.7	62.3	100.0	100.0

Table III. Results of a Conventional Flotation Test Using 1 lb/ton Dowfroth M-150 and 12 lb/ton Kerosene Conducted on an Eagle Coal Sample Wet-Ground for Four Hours.

Products	Weight (%)	Analysis (%) Ash	Analysis (%) Coal	Distribution (%) Ash	Distribution (%) Coal
Clean Coal	72.3	20.6	79.4	40.7	90.6
Refuse	27.7	78.4	21.6	59.3	9.4
Feed	100.0	36.6	63.4	100.0	100.0

DISCUSSION

The results of the flotation tests conducted during this reporting period demonstrate that the use of fine CGA bubbles is beneficial for fine coal flotation. Contrary to the general conception that flotation processes using fine bubbles, e.g., vacuum or pressure release flotation, are not selective, CGA flotation has been shown to be remarkably selective. As demonstrated with the ultrafine coal sample, the froth products of CGA flotation are almost twice as clean as those of the conventional flotation tests at 70% yield (Figure 34, Tables II and III). The kerosene consumption was considerably higher, however, both in conventional and in CGA flotation.

Attempts were made to coat the CGA bubbles with a film of kerosene and use them for flotation, hoping that this would reduce the oil consumption. However, no positive results have yet been obtained with this process. Perhaps the strenuous agitation employed in the oil-coating process may have been too drastic.

Other approaches, such as the addition of oils as emulsions and poly-aphrons, are being attempted in order to minimize the oil consumption. The latter involves encapsulating oil droplets in a thin film of water with a surfactant. The film breaks open when contacting a hydrophobic particle such as coal.

Another problem associated with CGA flotation is that the ash content of the froth products is relatively high when using a stable CGA, such as that prepared with Dowfroth M150. On the other hand, when using an unstable CGA, as is the case with MIBC, low ash clean coal products can be obtained, but at the expense of the yield.

Two approaches have been taken to correct this problem. Firstly, when a stable CGA is used, it is introduced into a flotation cell containing a lower pulp density coal. This reduces the number of CGA bubbles per unit volume of the coal suspension during flotation, so that the mechanical entrapment of ash particles can be reduced. Secondly, combined use of a weak frother, such as MIBC, and a more powerful frother, such as Dowfroth M150 has exhibited a synergistic effect, i.e., relatively low ash froth products are obtained while maintaining respectable yields. Further investigation is underway.

Electrophoretic mobility measurements conducted on coal and quartz particles in the presence of frother solutions suggest that Dowfroth M150 adsorbs indiscriminately on these particles while MIBC does not (Figures 5 and 6). This may partially account for the flotation results showing that MIBC is a more selective frother. Electrophoretic mobility measurements are also being conducted on clay minerals and coal pyrite.

A considerable amount of effort has been made to determine the surface charge of the CGA. The method employed was the one developed by Dibbs et al. (1974). Some problems have been encountered with this technique, and, therefore, a new method has been developed and is currently being tested with various ionic and nonionic frothers.

As has already been noted, there is no question that CGA flotation is advantageous over conventional flotation techniques. However, the advantages are evident only when the particles are fine. Our future work is, therefore, being directed toward the flotation of ultrafine coal samples. Micron-sized coal samples are being prepared with a micronizer.

One of the most exciting developments made during this reporting period is that a new method of generating CGA has been established. Essentially, the blender technique involves a simple mechanism of shredding the large bubbles formed around the vortex into smaller ones by high shear agitation. The stabilities of the CGA generated by this technique are comparable to those generated with the aspirator technique. The blender technique is perhaps more versatile, however, and can be more easily incorporated into large scale operations.

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