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THE INFLUENCE OF REAGENT DOSAGE ON THE FLOATABILITY OF PYRITE
DURING COAL FLOTATION

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ABSTRACT

In general, as the quantity of frother and/or coal collector is increased, so too, does the flotation of the undesired pyrite. The problem is particularly serious with oily reagents. For some coals, however, a collector, such as fuel oil, is required to achieve a high coal recovery. This requires a compromise between the competing desires of a high coal recovery and a high pyrite rejection. This study gives the quantitative effect of reagent dosage on coal and pyrite floatability and details several means of minimizing pyrite floatability during coal flotation. The effect of fuel oil on coal flotation is especially interesting in that the flotation process changes from froth flotation to emulsion or agglomerative flotation as the amount of oil is increased. This phenomenon provides an additional method of rejecting pyritic sulfur.

INTRODUCTION

Mitigation of the acid rain problem will require a reduction in the sulfur content of the coal used as the nation's principal fuel for electric power generation. This study is directed toward the reduction of the pyritic sulfur content of coal using the froth flotation process and a modification thereof, emulsion flotation.

High rank coals can be floated using only a frothing agent such as a short-chain, branched alcohol, a pine oil or a

polypropylene glycol methyl ether. However, the addition of oil to improve the flotation of difficult-to-float coals has long been practiced, since oil enhances the hydrophobicity of the coal and improves its flotation from the ash-forming minerals. As the amount of oil is increased, there is a continuum of froth flotation (< 1 kg/t oil), emulsion flotation (a few kg/t oil), spherical agglomeration (5-50% oil) and liquid-liquid extraction ($\sim 50\%$ oil). While the borders between these processes are ill-defined, on a practical basis only froth flotation, using 0 to ~ 1 kg/t of oil is used to any reasonable extent commercially. While the use of oil has proven invaluable to improve the froth flotation of poorly floatable coals, it is also known to promote the flotation of pyrite and the ash forming minerals (1,2). However, when a much larger amount of oil is used (say, 5-25%), spherical agglomeration occurs (3,4) with an accompanying emulsion inversion which facilitates the rejection of pyrite and ash minerals. Because this latter process requires the use of large amounts of oil to process relatively inexpensive coal ($\sim \$30$ /t), this study has focused on emulsion flotation (also called agglomeration flotation) which uses much less oil than does spherical agglomeration. The principles of emulsion flotation have been detailed elsewhere (5).

EXPERIMENTAL MATERIALS AND METHODS

The coals used and their composition are shown in Table 1. The Lower Kittanning coal is an extremely hydrophobic, low volatile bituminous coal while the Pittsburgh seam, coal is a somewhat less hydrophobic, high volatile A bituminous coal (6). Both may be floated with a frother only, though the former is more readily floatable. In order to minimize flotation complications due to ash minerals, locked particles and slime problems, coals coarse-cleaned by gravity methods were used. To 95% of these cleaned coals was added 5% of pyrite concentrate from the same seams, which had been previously purified by

TABLE 1. PROXIMATE ANALYSIS OF COALS USED (Dry Basis)

	Lower Kittanning Cambria Co., PA	Pittsburgh Seam Greene Co., PA
% Ash	6.34	7.00
% Volatile matter	19.09	38.06
% Fixed carbon	74.57	54.94
BTU/lb	14,687	14,135
% S (total)	0.94	1.76
% S (pyritic)	0.46	0.77
% S (SO ₄)	0.00	0.04
% S (organic)	0.48	0.94
ASTM rank	lvb	hvb

gravity concentration. The coal was ground to a nominal 10% 14x28 mesh, Tyler, (1168x589 μm), 45% 28x65 mesh, (589x208 μm) and 45% -65 mesh (-208 μm) shortly before use to minimize coal oxidation problems. The pyrite was ground in a mortar and pestle to 100% -28 mesh, and 90% -48 mesh immediately before use. For each test a 400g charge was used, composed of 380g coal and 20g of pyrite. The reagents used were MIBC (methyl isobutyl carbinol, or more properly 4-methyl-2-pentanol, Shell Chemical), pine oil (Yarmar F, Hercules) and No. 2 Fuel oil (Cities Service).

Flotation was done in the older-style WEMCO-Fagergren flotation cell with a metal rotor-stator assembly and a pyrex glass cell. The cell had an effective capacity of 2.3 l and was typically run at 1750 rpm using 7.1 lpm of air, though in a few cases other speeds and air quantities were used. The coal was pre-wet using a 5 min. conditioning period, the pH adjusted to pH 7, the reagents added and the pulp given an additional 5 min. of conditioning. Flotation froths were removed at periods of 15, 30, 45, 60, 105, 225 and 465 sec. to allow flotation rate data to be collected. The flotation products were dried, weighed and, in

some cases, screened to provide size data. The total sulfur content was determined by the LECO method (ASTM D-3174), and the liberated pyritic sulfur (SLP) was defined as the total sulfur minus the sum of the organic and sulfate sulfur and the intimately locked pyritic sulfur (as determined microscopically) as described previously (7).

Flotation rate constants were calculated for the first order rate equation proposed by Bushell (8):

$$k = 2.303 \frac{\log M_1 - \log M_2}{t_2 - t_1}$$

where k = rate constant, min^{-1}

M_1, M_2 = weight percent remaining in the cell at times t_1 and t_2 (min.), respectively

Only that portion of the data falling on the straight line portion of the semi-log plot of time vs. log percent remaining was used for the calculation. Ordinarily the data were linear for only the first one or two minutes of flotation, and in the usual case most of the material floats in about one minute. The weight percent data were used to calculate the flotation rate of the total yield (k_T), while SLP distribution data were used to calculate k_{SLP} . Further information on all of the above procedures may be found elsewhere (7).

EXPERIMENTAL RESULTS AND DISCUSSION

Influence of Frother Concentration

As a prelude to the evaluation of the addition of an oily collector and a study of the interface between froth and emulsion flotation, the effect of frother concentration was evaluated using Lower Kittanning coal. This coal has been found to be one of the most hydrophobic and readily floatable of all coals

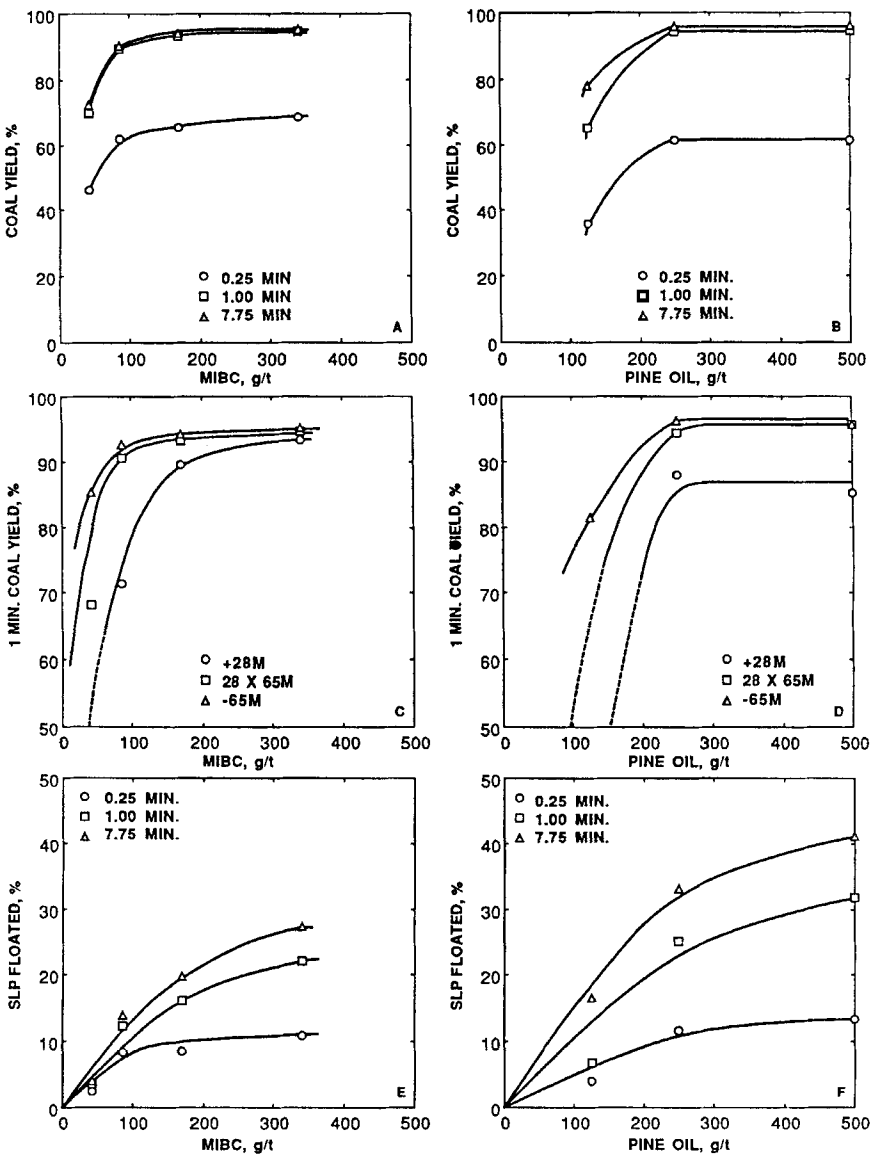


FIGURE 1. The influence of frother concentration on the flotation of Lower Kittanning coal and pyrite (SLP).

TABLE 2. FLOTATION RATE CONSTANTS USING VARIOUS FROTHERS AND FROTHER CONCENTRATIONS.
Lower Kittanning Seam

Frother	Amt. g/t	Yield @ 7.75 min	k_T , min^{-1}	k_{SLP} , min^{-1}	% SLP Floated @7.75	k_T/k_{SLP}
MIBC	40	72	1.20	0.04	4	30
MIBC	85	90	2.99	0.14	13	21
MIBC	170	94	2.69	0.18	20	15
MIBC	340	95	2.93	0.25	27	12
Pine Oil	125	78	1.05	0.07	17	15
Pine Oil	250	96	2.93	0.29	33	10
Pine Oil	500	96	2.97	0.39	41	8

evaluated in our laboratory, and it can be floated with a minimal amount of a frother only (6,9). Figures 1A and 1B show that this coal can be floated in near-ultimate yields (~95%) using ~100 g/t MIBC or ~250 g/t pine oil. Though MIBC is a superior frother in this case, this observation cannot be generalized. For lower rank coals, the authors have found that pine oil often gives similar results at a comparable dosage level. From Table 2, it will be noted that the coal flotation rate constant (k_T) achieves its maximum value near 100 g/t MIBC and 250 g/t pine oil and then remains roughly constant at higher frother dosages. Note in both cases that ~60 % of the coal floats in the first 15 sec. (Figures 1A and 1B).

Figures 1C and 1D give the relative floatability of various particle sizes of coal as a function of frother concentration. For a frother concentration of ~80 g/t, the 28x65 and -65 mesh particles float in 90+ % yields, while the coarser 14x28 mesh particles require up to 300 g/t MIBC for their flotation. Pine oil shows a similar pattern with the two finer particle size ranges requiring ~250 g/t pine oil, with the coarsest (14x28

mesh) particles achieving a lower ultimate flotation of ~85% at this and higher frother dosages.

While the hierarchy for the flotation of the various particle sizes decreases with increasing size in the order -65, 28x65 and 14x28 mesh, this observation, also documented elsewhere (1-7, 9-11), applies to those situations where all particles in a nominal -28 mesh assemblage must compete with one another for a position on the bubble surface. If, however, a sufficient amount of oil is present, all particles may become strongly hydrophobic, in which case they all tend to float at a similar rate (7). When particles with a smaller top size are floated, a different pattern emerges. Raleigh and Aplan (12) have demonstrated that, for several coals, decreasing the top size from -28 mesh to -100 mesh (150 μm) leads to increased floatability, while a further reduction to -400 mesh (37 μm) results in a decreased floatability. This reduced floatability has been attributed to an inadequate bubble surface area for the plethora of very fine particles and to the adsorption of frother by the large surface area of the fine coal, leading to a decrease in frothing (12).

The most striking feature of Figure 1 is the response of liberated pyritic sulfur (SLP) to an increase in frother concentration. As seen in Figures 1E and 1F, the amount of SLP that floats is a function of both the time of flotation and the frother dosage. A prolonged time of flotation and a high amount of frother both lead to a great increase in the pyrite content of the froth. At the longest time of flotation (7.75 min.) and at the highest frother level tested, 27% of the SLP is floated with MIBC and 41% with pine oil. Note that pine oil results in greater pyrite flotation than does MIBC in this case.

Another way of evaluating the test data is to evaluate the coal (k_T) and pyrite (k_{SLP}) flotation rate constants given in Table 2. While the flotation rate of the coal increases to some value and then remains constant, the flotation rate of the SLP continually increases with increasing frother concentration. A clearer way of evaluating these data is to make use of the

selectivity criterion expressed by the ratio of the rate constants of the total material floated (essentially the coal) and the pyrite, k_T/k_{SLP} . The higher the ratio, the better the selectivity between coal and pyrite. Table 2 shows that this ratio decreases as the frother concentration is increased with both MIBC and pine oil. The very high ratio of 30:1 for 40 g/t of MIBC is probably unrealistic because of the relatively poor coal yield. However, as the yield is increased toward the ultimate, the selectivity between coal and pyrite decreases. It should be noted that better selectivity is achieved with MIBC than with pine oil for this coal.

Influence of Fuel Oil Collector.

While fuel oil is known to be valuable for improving the flotation rate and the floatability of difficult-to-float particles, it is also known to increase the floatability of pyrite and the ash-forming minerals (1,2). The influence of oil on coal floatability was studied using a base amount of 85 g/t of MIBC and 125 g/t pine oil with the Lower Kittanning coal. These amounts had previously been found to be the incipient amount to give 90-95% recovery when used as the only flotation reagent. To this amount of frother was added varying amounts of fuel oil up to about 50 times as much. The experimental data are given in Table 3. Figure 2 shows that as the amount of oil is progressively increased, the yield increases slightly until essentially all of the 95% coal initially added has floated. Any yield over 95% indicates the flotation of some of the 5% added pyrite. Other experiments (not shown here) indicate that at a ratio of about 5:1 (fuel oil:MIBC) all of the coarse coal particles have floated. The most important feature of Figure 2 and Table 3 is that as the oil is increased above the ~5:1 ratio, the amount of pyrite (SLP) that floats decreases while the yield remains near perfect. The worst condition leading to strong pyrite flotation occurs at a ~5:1 fuel oil:MIBC ratio (~1 kg/t

TABLE 3. EFFECT OF FUEL OIL-FROTHER MIXTURES ON THE FLOTATION OF COAL AND PYRITE.

Base amount of frother used for:					
Lower Kittanning: 85 g/t MIBC, 125 g/t pine oil					
Pittsburgh Seam : 170 g/t MIBC, 250 g/t pine oil					
Frother	Fuel oil/ Frother Ratio	Yield @ 7.75 min.	k_T , min^{-1}	k_{SLP} , min^{-1}	% SLP floated @ 7.75 min
<u>Lower Kittanning Seam</u>					
MIBC	0	90	2.25	0.14	13
MIBC	1.1	95	2.48	0.33	34
MIBC	2.2	95	2.85	-	-
MIBC	5.5	97	3.30	0.74	56
MIBC	22	97	3.25	0.46	44
MIBC	55	98	2.82	0.35	70
Pine Oil	0	78	1.05	0.07	17
Pine Oil	0.74	89	1.86	0.18	19
Pine Oil	1.5	89	1.80	-	-
Pine Oil	3.7	97	2.54	0.59	53
Pine Oil	15	97	2.54	0.28	45
Pine Oil	37	98	2.86	0.36	57
<u>Pittsburgh Seam</u>					
MIBC	0	84	1.55	0.14	16
MIBC	1.1	92	2.35	0.23	24
MIBC	2.2	95	2.85	-	-
MIBC	5.5	97	3.22	0.55	45
MIBC	22	97	2.76	0.28	44
MIBC	55	97	2.86	-	47
Pine Oil	0	89	1.97	0.17	21
Pine Oil	0.7	92	2.32	0.36	34
Pine Oil	1.5	96	3.05	-	-
Pine Oil	3.7	97	3.26	0.53	46
Pine Oil	15	97	2.45	0.30	49
Pine Oil	37	98	2.56	0.29	61

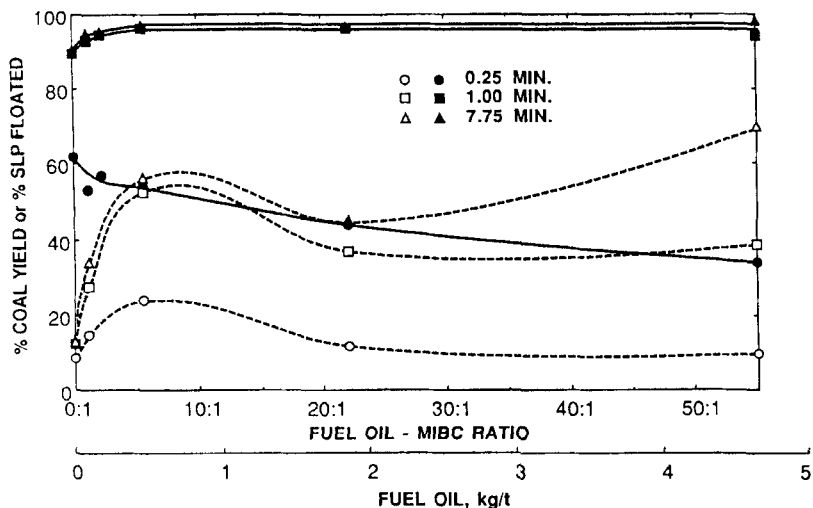


FIGURE 2. The influence of fuel oil collector on the flotation of Lower Kittanning coal and liberated pyritic sulfur (SLP). Base condition: 85 g/t MIBC frother. Filled symbols: coal yield. Open symbols: SLP flotation.

oil) or when large amounts of oil (~55:1) and prolonged flotation times (e.g., 7.75 min) are used. Fuel oil-pine oil mixtures responded in the same manner (Table 3).

To see if this condition also pertains to another coal, tests were also run using Pittsburgh seam coal (Figure 3). Table 3 shows that twice the amount of frother (170 g/t MIBC or 250 g/t pine oil) gave relatively high recoveries (84-89%) for this somewhat less hydrophobic coal. The results were nearly the same as before, except that more oil was used. As before, auxiliary tests showed that essentially all of the coarse particles were floated at a 5:1 fuel oil:frother ratio. Furthermore, the same pattern was repeated when pine oil was used as the frother with this coal (Table 3). When larger amounts of fuel oil are used, the flotation of pyrite is decreased. It seems apparent that if oil must be used for coal flotation, the ~5:1 ratio should be

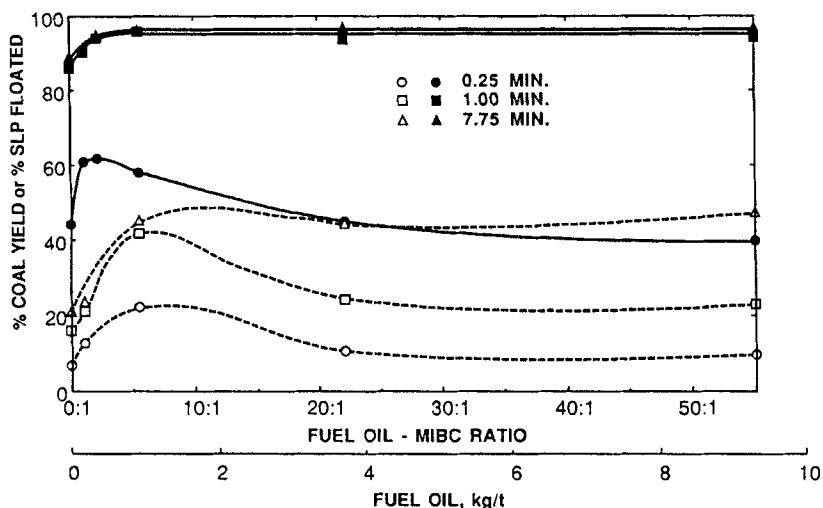


FIGURE 3. The influence of fuel oil collector on the flotation of Pittsburgh Seam coal and liberated pyritic sulfur (SLP). Base condition: 170 g/t MIBC Frother. Filled symbols: coal yield. Open symbols: SLP flotation.

avoided and short flotation times should be used if the object is to minimize the flotation of pyrite. Other tests in our laboratory have demonstrated that this same response is repeated for those relatively high-rank coals that can be floated in a reasonable yield using a frother only.

Figure 4 shows the response of the flotation rate constants of both coal and pyrite to increasing amounts of coal. Note that at a ratio of 5:1 (fuel oil:frother) there is a change in the nature of the flotation with the flotation rates apparently decreasing, that of the pyrite (k_{SLP}) more so than the coal (k_T). The flotation of these two coals with fuel oil:pine oil mixtures showed the same pattern, with the advantage of using short flotation times and either no fuel oil or an amount somewhat greater than the 5:1 ratio.

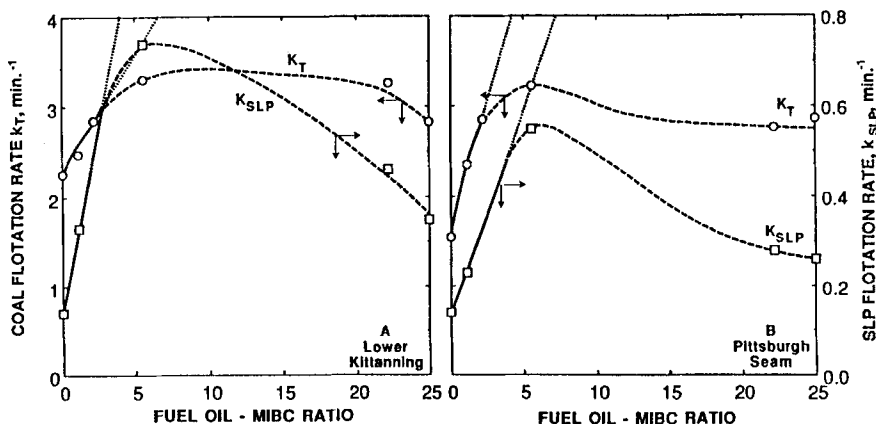


FIGURE 4. The influence of fuel oil on the flotation rates of coal and liberated pyritic sulfur. Base condition: Lower Kittanning 85 g/t MIBC, Pittsburgh Seam 170 g/t MIBC.

This finding prompted a more detailed evaluation of the system. At a fuel oil:frother ratio of ~2:1 (~185 g/t fuel oil for the Lower Kittanning Coal and ~370 g/t for the Pittsburgh seam coal) a change in the nature of the froth was noted. The coal floated more quickly, and the froth was not quite as free-flowing as in the collectorless froth flotation of the same coal. At a value of ~5:1 (~465 g/t oil for the Lower Kittanning and ~930 g/t oil for the Pittsburgh seam coal), this phenomenon was much more pronounced and the froth was thick, matted, not free-flowing and was difficult to remove from the cell. The froth was dry in appearance and when bubbles broke and released their attached coal particles, the particles appeared perfectly dry. The floated coal, when filtered, contained ~5% less moisture than the filter cake from froth flotation. The apparent decrease in flotation rates at about ~5:1 fuel oil:frother level was found to be an artifact. The flotation rate determining step has changed from bubble mineralization to froth removal. Exam-

ation of the particles through the glass walls of the flotation cell showed that the particles went to the froth almost immediately at a rate too fast to be readily quantifiable. The apparent reduction in rate can be attributed to an inability to remove the thickly matted particles in the froth as quickly as the particles report to the froth. The rate curves should thus be drawn roughly as the dotted line rather than as the dashed segments of the rate curves shown for froth removal-controlled segments of the curves roughly above the 5:1 ratio. This phenomena is a manifestation of emulsion inversion due to a change from one of oil droplets in water (O/W emulsion) under froth flotation conditions, to one of a water-in-oil (W/O) emulsion in the froth. In the latter case the continuous phase is coal particles with an oiled surface and containing a modest amount of water droplets. The emulsion inversion has rejected pyrite particles in a manner similar to that found in spherical agglomeration, but at a substantially lower oil requirement. Current tests in our laboratories have indicated the pyrite rejection in emulsion flotation is not as effective as it is in spherical agglomeration, and this aspect will require more work. Additional studies will be reported soon (13).

Figure 5 shows the effect of oil on coal flotation where the data are expressed as grade-yield curves. For these curves, the best tests are those showing the lowest sulfur content in the floated coal at the greatest yield of coal. Each of the data points in a set (say 0:1, 1.1:1 etc.) represent the values obtained at 15, 30, 45, 60, 105, 225 and 465 sec. The 15 sec. point shows the lowest sulfur and lowest yield with the various other timed, cumulative froth fractions progressing upward and to the right in each set. Note that as yield exceeds ~85%, the amount of sulfur in the froth increases substantially. Similar curves were obtained with both coals using pine oil as the frother. For both coals, the lowest sulfur content for a given yield is usually found when no oil is used (though for yields approaching 90%, other ratios may also be good for the Pittsburgh

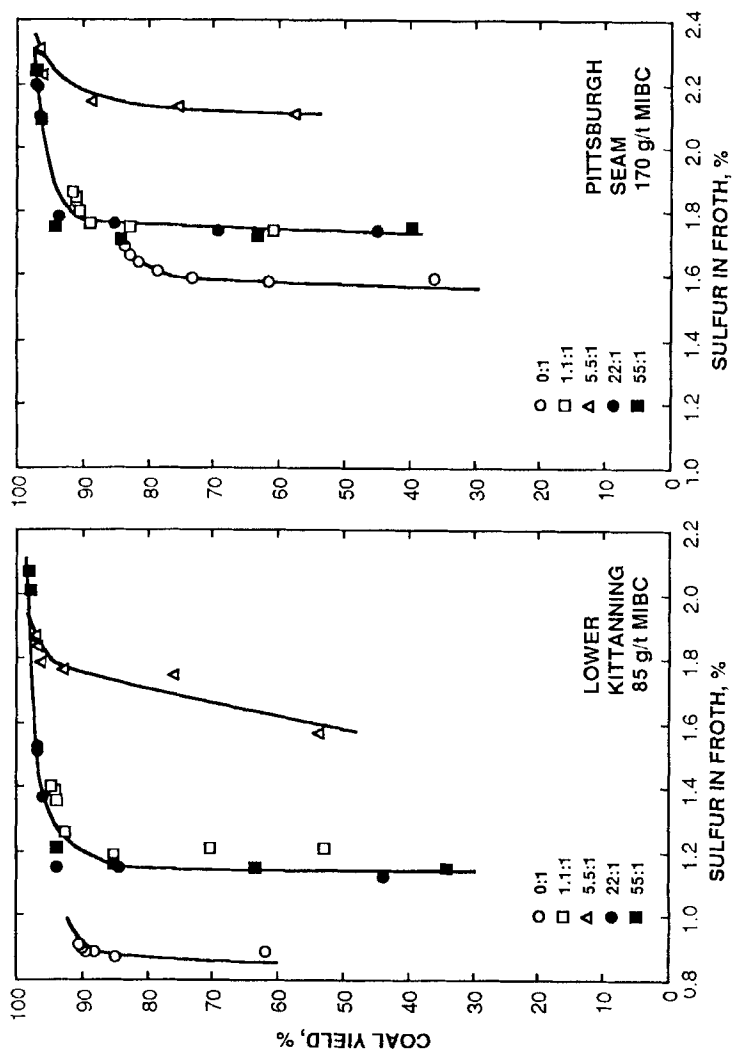


FIGURE 5. Grade-yield curves for the flotation of Lower Kittanning and Pittsburgh Seam coals using MIBC (85 g/t) and varying amounts of fuel oil.

TABLE 4. SPLIT-FEED FLOTATION OF COAL SIZED AT 65 MESH.

Mesh Size	MIBC g/t	Speed RPM	Air lpm	Yield @ 7.75 min	k_T min^{-1}	k_{SLP} min^{-1}	$\frac{k_T}{k_{SLP}}$
<u>Lower Kittanning Seam</u>							
-65	85	1750	7.1	93	1.55	0.15	10
-65	85	1350	5.2	91	1.36	0.18	8
+65	170	1750	7.1	92	2.45	0.14	18
+65	170	1350	5.2	90	2.02	0.08	25
<u>Pittsburgh Seam</u>							
-65	125	1750	7.1	87	1.49	0.11	14
+65	250	1750	7.1	78	1.24	0.03	41

Seam coal. Similar data were obtained with fuel oil-pine oil mixtures. In all cases (both coals, both frothers), the worst situation is with the ~5:1 fuel oil:frother ratio. This analysis of the data strongly reinforces the findings in Figures 2 and 3.

Split-Feed Flotation

The response of various particle sizes in the series of tests dealing with frother concentration prompted an investigation of split-feed flotation. In this process (14), the feed coal is sized into two fractions, and each size fraction is floated separately. The concept behind this process is that the finer fraction should not require as much reagent for its flotation as does the coarser fraction. Test results shown in Table 4 demonstrates that, in fact, the -65 mesh ($-208 \mu\text{m}$) fraction requires less frother for high-yield flotation, than does the +65 mesh fraction. Comparing the flotation of both the Lower Kittanning and the Pittsburgh Seam coals under standard conditions (1750 rpm, 7.1 lpm air) shows that much better selectivity between coal and pyrite is achieved with the coarser

fraction than with the finer one. Note k_T/k_{SLP} ratios greater than 18:1 are possible with the coarser particles. It is apparent that under these test conditions, selectivity in the flotation of coal from pyrite is relatively poor for the -65 mesh particles. The very high k_T/k_{SLP} ratio of 41 for the +65 mesh Pittsburgh seam coal is partially a reflection of the low coal yield since selectivity decreases as yield decreases.

Since gentle operating conditions (starvation frother, low rpm and low air rates) are known to minimize pyrite flotation (15) a few tests were made at 1350 rpm and 5.2 lpm air rate. Table 4 shows an insignificant change in the k_T/k_{SLP} ratio for the -65 mesh material, but a significant improvement on the rejection of pyrite in the coarser fraction.

SUMMARY AND CONCLUSIONS

For the coals and operating conditions evaluated here:

1. Coal flotation recovery and rate increase as the amount of frother is increased, approaching complete coal recovery asymptotically.
2. The flotation of coarser particles requires more frother for their recovery than do the finer particles.
3. As the amount of frother increases, particles of all sizes (up to ~28 mesh) tend to float in similar yields.
4. As the time of flotation and the amount of frother increases, so, too, does the amount of pyrite that floats. At prolonged flotation times and excessive frother dosage, the increased floatability of pyrite is massive.
5. The use of fuel oil in conjunction with a frother increases the yield (recovery) and flotation rate of coal particles.
6. As the amount of oil used in flotation is increased, pyrite flotation also increases up to a fuel oil:frother ratio of about 5:1. Beyond this value, pyrite tends to float more slowly, especially for short flotation times and assuming grossly excessive amounts of oil are not used.
7. At this 'magic' ratio of about 5:1 fuel oil:frother, the froth changes from free flowing to viscous and the flotation rate-determining step changes from bubble mineralization to froth removal.

8. Also at this point, the flotation has changed from froth flotation to emulsion flotation and the system has inverted from an O/W emulsion to a W/O emulsion with oiled coal particles being the continuous phase.
9. Analyses of the data by use of grade-yield curves shows that the lowest amount of pyrite in the floated coal is obtained using no oil, while at a 5:1 fuel oil:frother level the pyrite in the floated coal is the greatest. Other ratios from 1:1 to 50:1 generally are intermediate between the limiting cases.
10. Split-feed flotation at ± 65 mesh using a frother-only system, shows that coal-pyrite selectivity in the coarser (+65 mesh) fraction is greater than that in the finer (-65 mesh) fraction.

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