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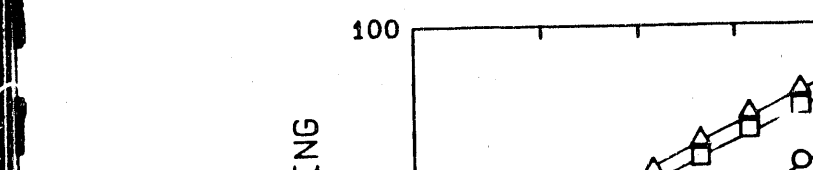

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COAL SURFACE CONTROL FOR ADVANCED FINE COAL FLOTATION

Project No. DE-AC22-88PC88878

QUARTERLY REPORT NO. 7
April 1, 1990 - June 30, 1990

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COAL SURFACE CONTROL FOR ADVANCED FINE COAL FLOTATION

DOE CONTRACT DE-AC22-88PC88878

Quarterly Report No. 7

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1.0 INTRODUCTION

Historically, coal surface characterization and control are not considered critical to coal cleaning because of prior emphasis on maintaining particle size as coarse as possible. However, the current goal of near-total removal of pyritic sulfur necessitates fine grinding of coal to liberate pyrite. At these fine sizes coal surface behavior plays an increasingly dominant role and consequently the need for surface characterization and control is critical.

In order to investigate the properties of coal surfaces and their role in coal flotation, DOE awarded a contract entitled "Coal Surface Control for Advanced Fine Coal Flotation" to the University of California at Berkeley in October 1988. The main goal of the project is to characterize the surface and control the behavior of coal during advanced flotation processing in order to achieve an overall objective of removing 90% of the pyritic sulfur at 90% Btu recovery. Also, investigation of the effects of weathering on the surface characteristics of coal is another important aspect of this project. This information will serve to support the engineering development of advanced flotation technology conducted by ICF Kaiser Engineers, Inc. as part of DOE PETC's Acid Rain Control Initiative.

The project team consists of research and engineering groups at Berkeley, Columbia University, the University of Utah and Praxis Engineers, Inc., with the University of California acting as the prime contractor. The organizational chart for this project is presented in Figure 1.1, which also identifies key project personnel.

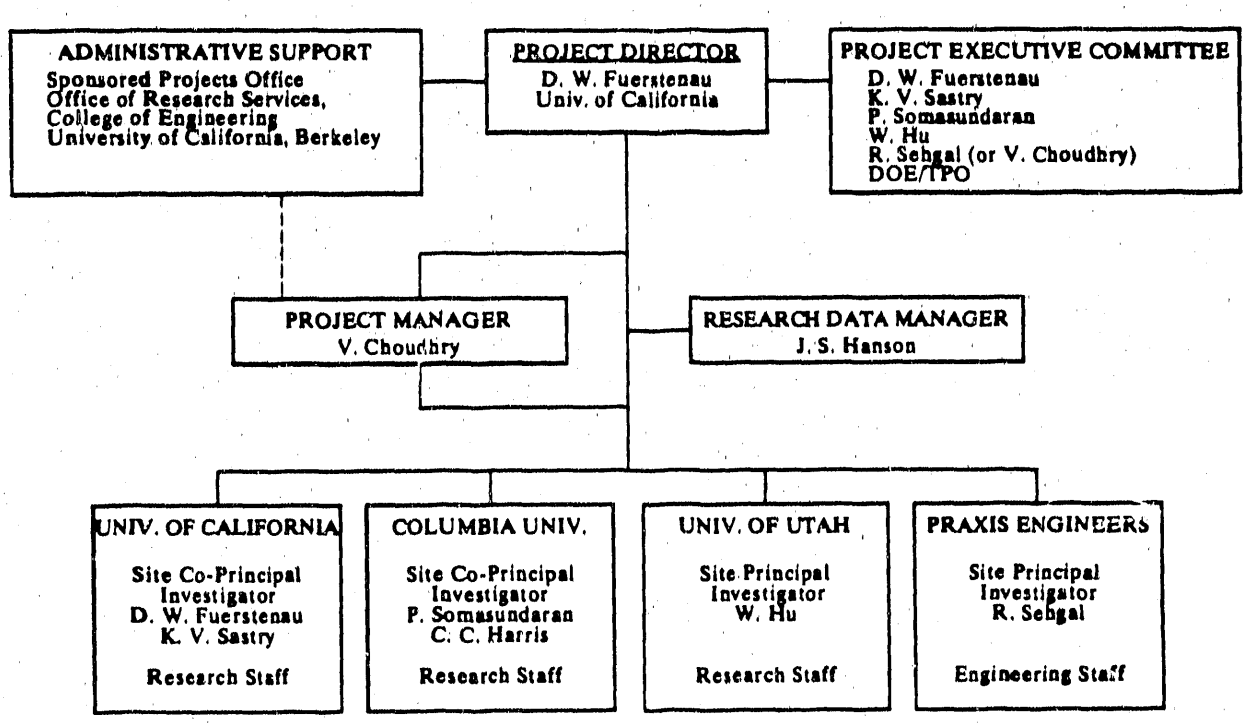

Figure 1.1 - Project organizational chart

1.1 Scope of this document

This document is the seventh quarterly report prepared in accordance with the project reporting requirements covering the performance period from April 1, 1990 to June 30, 1990. This report presents the results of the technical work undertaken as part of this project by the University of California at Berkeley, Columbia University, the University of Utah and Praxis Engineers, Inc during this period. The results of work conducted during this period are compiled together based on research topic rather than by location and reported here. Research topics covered during this quarter include the characterization of the coal samples used in the additional washability studies, further characterization of the base coals, various flotation studies on optimization and pyrite rejection, and characterization of weathering samples.

1.2 Overall Project Scope and Objectives

The primary objective in the scope of this research project is to develop advanced flotation methods for coal cleaning in order to achieve near total pyritic-sulfur removal at 90% Btu recovery, using coal samples procured from six major U.S. coal seams. Concomitantly, the ash content of these coals is to be reduced to 6% or less. Investigation of mechanisms for the control of coal and pyrite surfaces prior to fine coal flotation is an important aspect of the project objectives. The results of this research are to be made available to ICF Kaiser Engineers who are currently working on the Engineering Development of Advanced Flotation under a separate contract with DOE under the Acid Rain Control Initiative program.

As a part of this contract, large quantities of coal samples were procured from six major seams identified by DOE for use in this project for advanced flotation and weathering studies. Samples of the same coals have also been supplied to the University of Pittsburgh for selective agglomeration coal surface control research under a separate contract by DOE. In addition, coal samples of the three base coals were supplied to ICF Kaiser Engineers and Southern Company services for bench-scale test work on their project. Coal samples were also supplied to a number

of other organizations for research on DOE funded projects. Collection and preparation of a large sample for distribution for research projects served the purpose of providing uniform and identical samples. This will make comparative evaluation of the results of various processes being funded by DOE relatively easy.

A second major objective is to investigate factors involved in the progressive weathering and oxidation of coal that had been exposed to varying weathered degrees, namely, open, covered and in an "argon-inerted" atmosphere, over a period of twelve months. After regular intervals of weathering, samples of the three base coals (Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA) were collected and shipped to both the University of Pittsburgh and the University of California at Berkeley for characterization studies of the weathered material.

2.0 WASHABILITY STUDIES

During the sixth quarterly period of the contract (January 1, 1990 - March 31, 1990) an additional washability study was undertaken in order to assess the liberation behavior of the three base coals at 100, 200, 325 and 400 mesh. The coal samples used in this fine washability study were part of reserve sample of coal mined in November 1988 and stored in argon-purged 55-gallon drums. During this past quarterly period, in order to assess the fine washability data appropriately, the particle size distributions of the samples were determined with an L&N Micro-trac particle size analyzer and as part of the QA/QC program, the total sulfur, pyritic sulfur and proximate analyses of the samples were completed.

2.1 Particle size distribution of the washability samples

The particle size distribution of the 100 mesh, 200 mesh and 400 mesh grinds of the additional washability samples from the three base coals were carried out using the Micro-trac particle size analyzer. In the case of the minus 100-mesh grinds, the sample was first wet-sieved using a 200-mesh screen and the minus 200-mesh fraction was used for Micro-trac analysis. The results are presented in Figures 2.1, 2.2 and 2.3 and indicate that the particle size distributions are as expected. We were concerned that the size distributions of each of the fine washability grinds did in fact represent minus 100, 200 and 400 mesh grinds. As can be seen from the plotted size distributions, the minus 200 and 400 mesh grinds are indeed about 95% minus 75 and 37 μm, respectively, while the 100-mesh grinds appear to be about 96-98% minus 150 μm. The deviation of the particle size distributions from a straight line at extremely fine particles sizes is probably due to the measuring device since 5 μm is near the lower limit of detection.

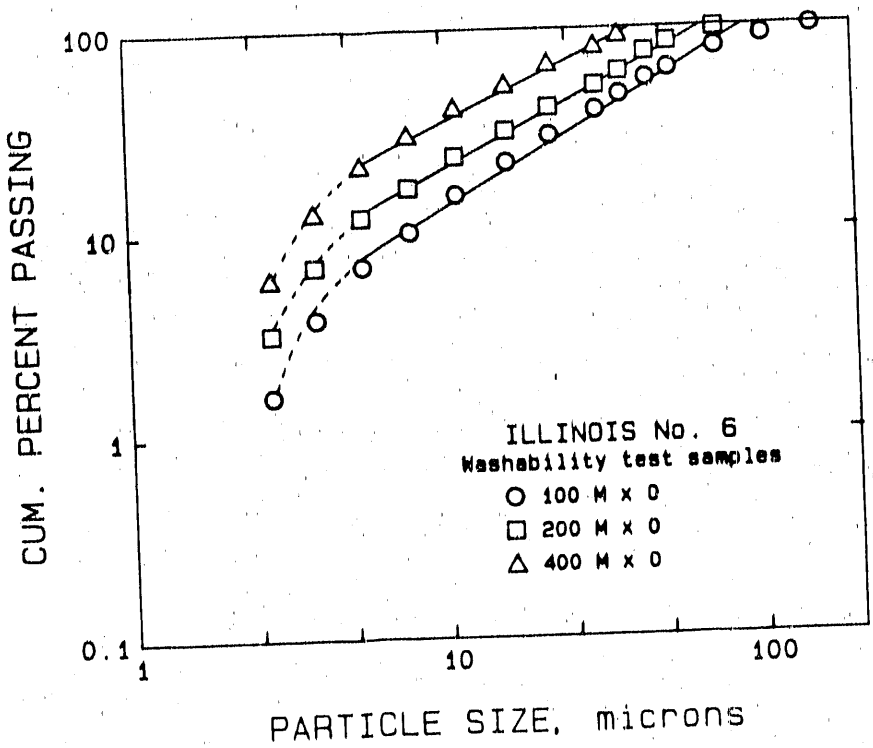

Figure 2.1 - Particle size distribution for Illinois No. 6 coal washibility test samples ground to 100, 200 and 400 mesh top size fraction.

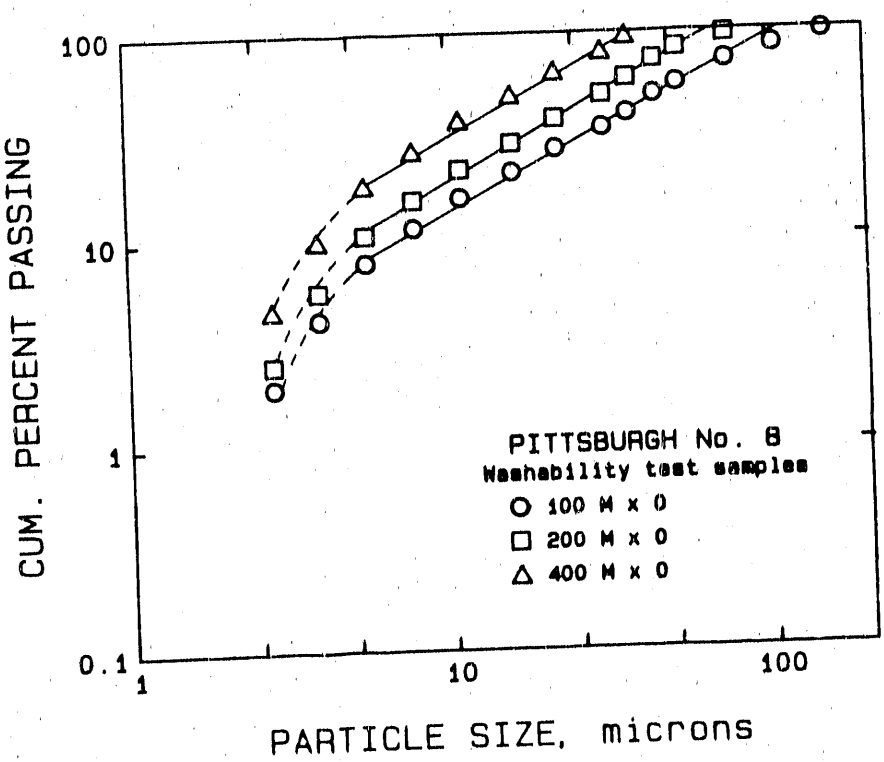

Figure 2.2 - Particle size distribution for Pittsburgh No. 8 coal washability test samples ground to 100, 200 and 400 mesh top size fraction.

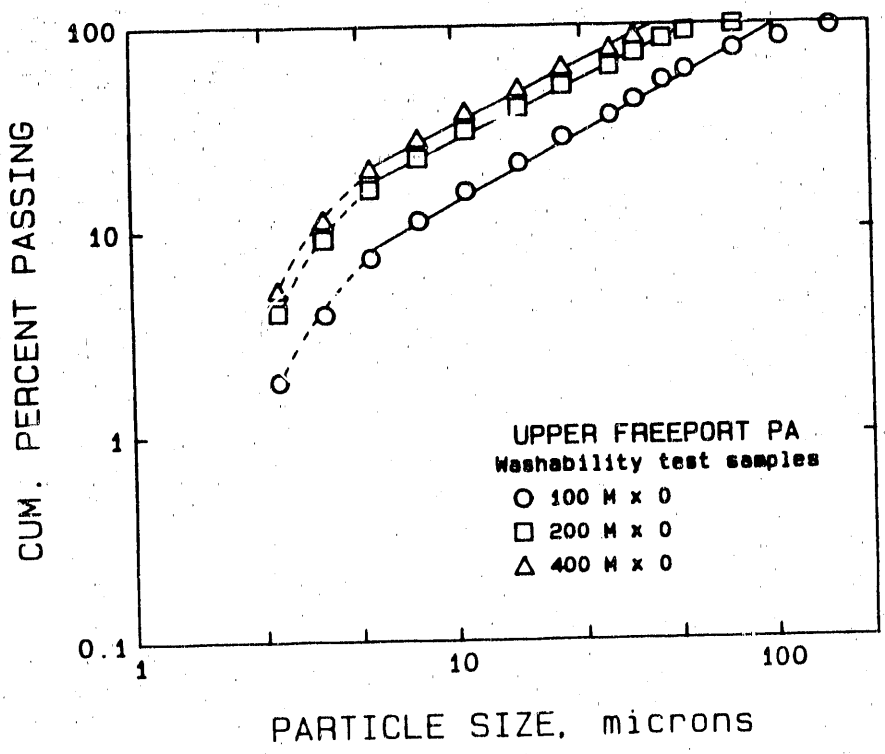

Figure 2.3 - Particle size distribution for Upper Freeport PA coal washability test samples ground to 100, 200 and 400 mesh top size fraction.

2.2 Chemical analysis of washability test samples.

During the past quarter, the coal samples used in the fine washability study (minus 100, 200 and 400 mesh) were further characterized by proximate, total-sulfur and pyritic-sulfur analyses. These samples were prepared for washability tests at the Geochemical Laboratory for each of the three base coals and a portion of each sample sent to Berkeley for various characterization tests.

Once the samples were received by Berkeley, they were homogenized and split in a rotating splitter. Half of each sample was inerted, packed and sent to Columbia University for size analysis and other QA/QC tests. The remainder of each sample was further homogenized and split in order to perform characterization studies. Approximately 10 grams of each sample was used for proximate, total-sulfur, and pyritic-sulfur analyses and the remaining sample was used for particle-size analysis.

Table 2.1 - Proximate, total sulfur and pyritic sulfur analyses for the additional fine washability test samples for the three base coals.

Coal	Size, Mesh	Moist. %	Volatile %	Ash %	Fix C. %	Tot. S %	Pyr. S %
Illinois No. 6	- 100	4.22	36.14	16.89	46.97	4.92	2.78
	- 200	3.77	36.03	16.84	47.13	5.09	2.72
	- 400	4.12	35.77	16.40	47.83	4.85	2.75
Original Washability sample				16.00		4.87	2.94
Pittsburgh No. 8	- 100	2.49	35.45	12.21	52.34	4.03	2.65
	- 200	2.57	34.86	12.52	52.62	4.26	2.72
	- 400	2.35	34.49	12.32	53.19	3.93	2.64
Original Washability sample				11.90		3.90	2.84
U Freeport PA	- 100	1.54	25.51	12.32	62.22	2.44	1.55
	- 200	1.22	25.39	12.83	61.78	2.31	1.53
	- 400	0.98	24.95	12.97	62.08	2.25	1.53
Original Washability sample				12.46		2.22	1.46

Table 2.1 presents the results of the proximate, total-sulfur and pyritic-sulfur analyses for all the washability test samples except the 325 mesh samples. These results show that the samples used in the washability tests were consistent in composition and comparable to the samples used in the original washability study. This indicates that the coals did not degrade while stored in the drums and that the results can be compared with the original washability data to yield information on both coarse and fine liberation behavior.

3.0 CHARACTERIZATION STUDIES

During this past quarterly period, characterization of the three base coals continued. The induction time of the base coals ground in the stainless steel rod mill was measured. Also, the relationship between the commonly reported value of combustible material recovery (CMR) and the more important Btu recovery was determined empirically. As part of the QA/QC program, Berkeley, Columbia, and Utah ran proximate and pyritic-sulfur analyses on identical flotation feed and product samples prepared at Berkeley. The results obtained from the QA/QC study were then compared to calculate flotation efficiency by a number of methods and the results are discussed in this chapter.

3.1 Induction time measurements

In order to complete the surface characterization studies of the three base coals, induction time measurements were performed on fresh-ground material taken from the research samples. The objective of these tests was to assess the hydrophobicity of the new surface generated after grinding. Because 100 x 150 mesh particles are needed for induction time measurements, samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coal were prepared by reducing the standard grinding time to 15 minutes in order to obtain a sufficient quantity of material to carry out the measurements. The ground products were prepared and the induction time measurements were made following the standard procedure reported in previous biweekly and quarterly reports. Induction times were measured on samples that had been deslimed as well as on freshly ground samples.

The results of the induction time measurements for fresh-ground Illinois No. 6 are presented in Figure 3.1. As expected, the deslimed sample shows a shorter induction time (470 microseconds) than that of the undeslimed sample (500 microseconds), indicating a slight increase in the hydrophobicity of the deslimed sample in comparison with the undeslimed sample. The slime-coating phenomenon, discussed in previous reports, seems to be responsible for the larger

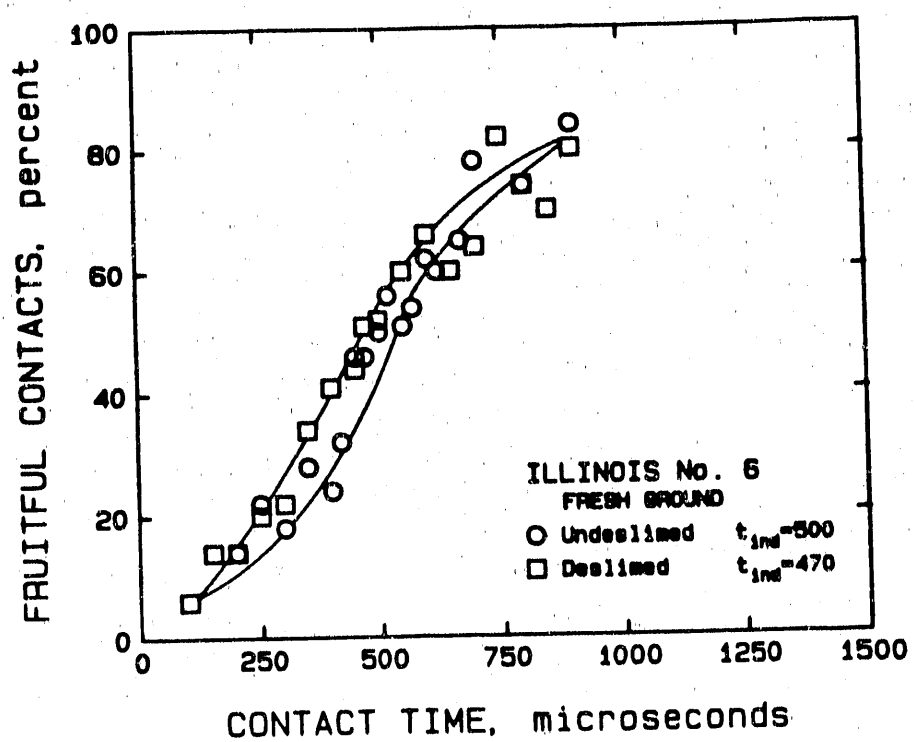

Figure 3.1 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Illinois No. 6 coal both undeslimed and deslimed fresh ground samples.

induction times of the undeslimed material. However, in the case of fresh-ground samples this difference is smaller than that observed for the natural fines of the research and weathered samples, possibly due to different size distributions of the samples.

Figure 3.2 presents the percentage of fruitful contacts as a function of contact time for fresh-ground Pittsburgh No. 8, for both the undeslimed and deslimed samples. In the case of Pittsburgh No. 8 coal, the induction time for the deslimed sample was 180 microseconds and that of the undeslimed sample was 265 microseconds. The difference between the induction time of the deslimed and the undeslimed samples is larger than that of Illinois No. 6 coal, probably due to the nature of the coals themselves and ash constituents.

In the case of Upper Freeport PA coal (Figure 3.3), similar behavior to that of Illinois No. 6 and Pittsburgh No. 8 was observed. However, the induction times for the deslimed sample (75 microseconds) and the undeslimed sample (105 microseconds) are significantly lower

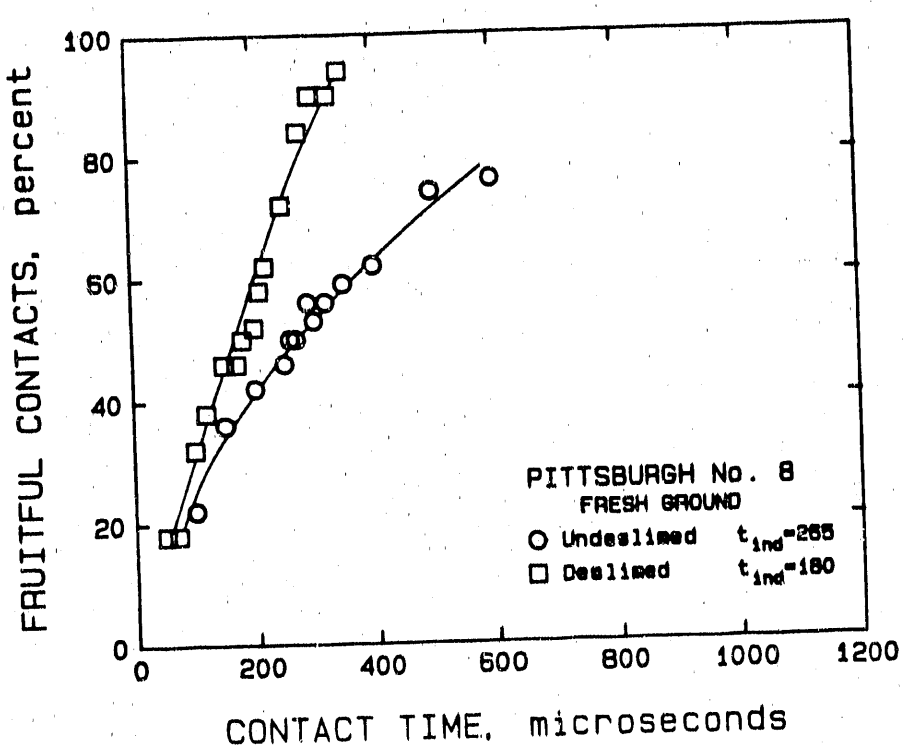

Figure 3.2 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Pittsburgh No. 8 coal both undeslimed and deslimed fresh ground samples.

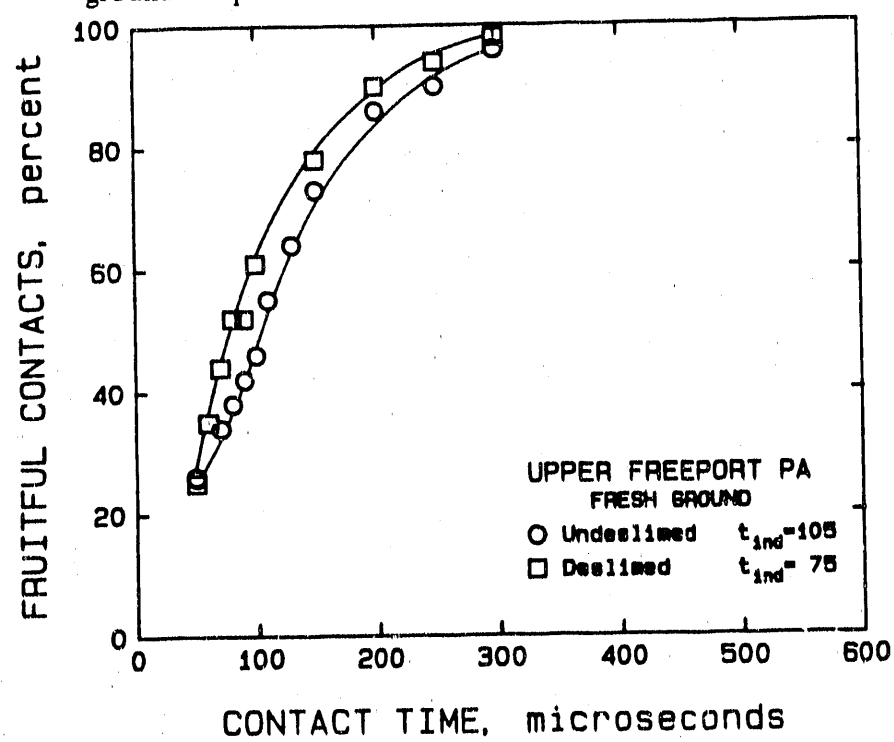

Figure 3.3 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Upper Freeport PA coal both undeslimed and deslimed fresh ground samples.

than those of Illinois No. 6 and Pittsburgh No. 8 coals confirming that Upper Freeport PA coal is by far the most hydrophilic of the three coals.

Induction-time measurements of ground coal clearly show an increase in the hydrophobicity of the three base coals, as compared to the natural fines of the unground research coals and weathered samples, probably due to the fresh surfaces generated during comminution. It seems that the slime-coating phenomenon is responsible for the decrease in the hydrophobicity of the fresh-ground material with the magnitude of this decrease being coal specific. In accordance with all previous results, the hydrophobicity of the three base coals has the following order for both the undeslimed and deslimed samples tested.

Upper Freeport PA> Pittsburgh No. 8> Illinois No. 6

3.1 Correlation between yield, combustible-material recovery (CMR), and heating-value recovery (HVR).

In all previous reports, the percent yield and percent CMR were used to evaluate process efficiency in order to avoid the more elaborate and costly heating-value (Btu) analysis. Therefore, the correlation between the yield, CMR, and HVR need to be established empirically.

The calorific value of the feed samples and selected flotation products of the three base coals were determined and the results are given in Tables 3.1, 3.2, and 3.3. The data were treated by regression and correlation analysis to give a relationship between CMR and Btu recovery which is given in Figures 3.4, 3.5 and 3.6 for Upper Freeport PA, Pittsburgh No. 8, and Illinois No. 6 coals, respectively. It can be seen that good linear relationships were obtained for each coal. These results concur with Appendix A-2 of our First Annual Report that asserts that CMR values slightly under estimate Btu recovery, and therefore, Efficiency Indices based on CMR values slightly under-report the flotation efficiency based on Btu recovery.

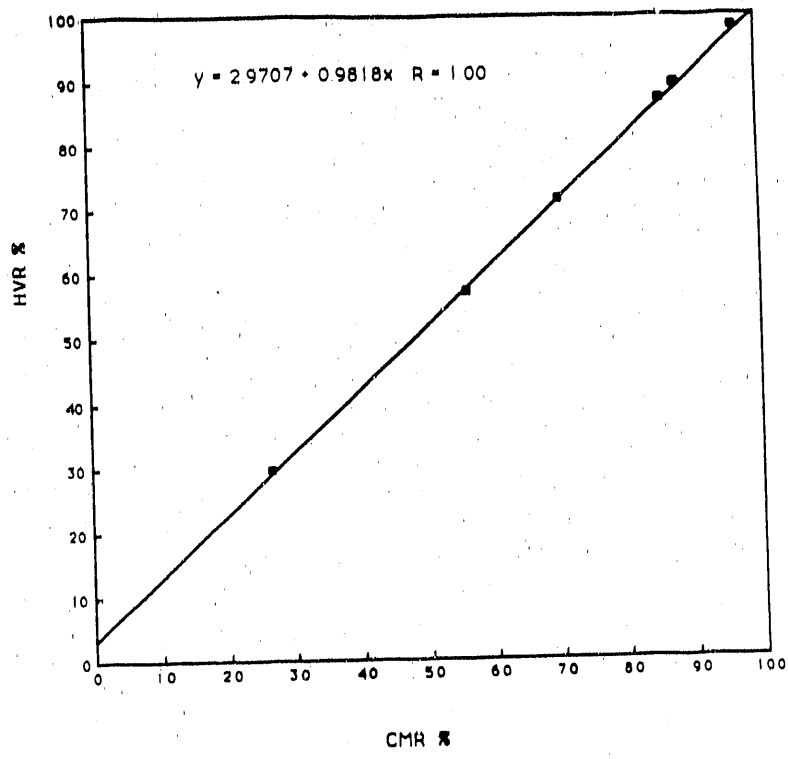

Figure 3.4 - Relation between CMR and HVR for Upper Freeport PA coal.

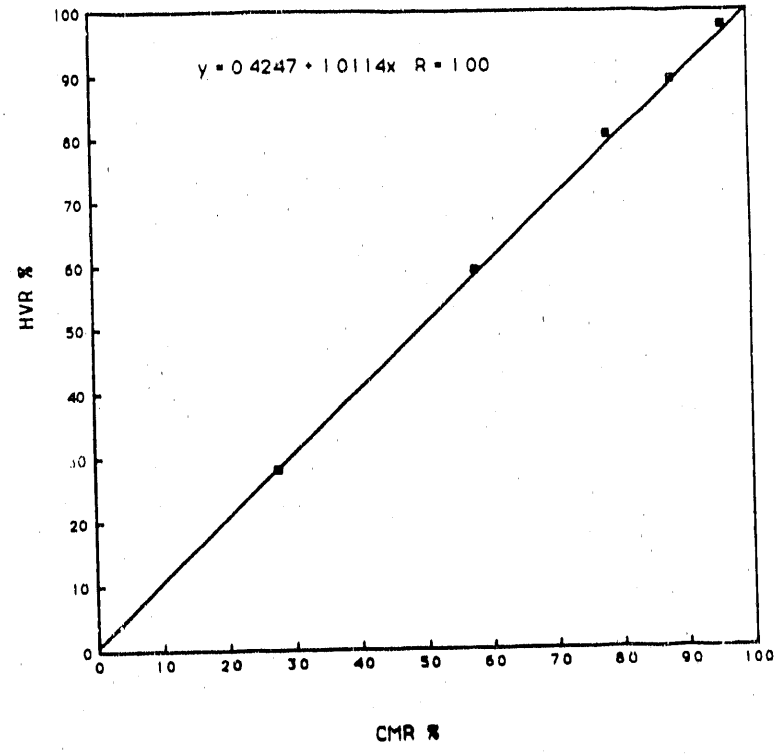

Figure 3.5 - Relation between CMR and HVR for Pittsburgh No. 8 coal.

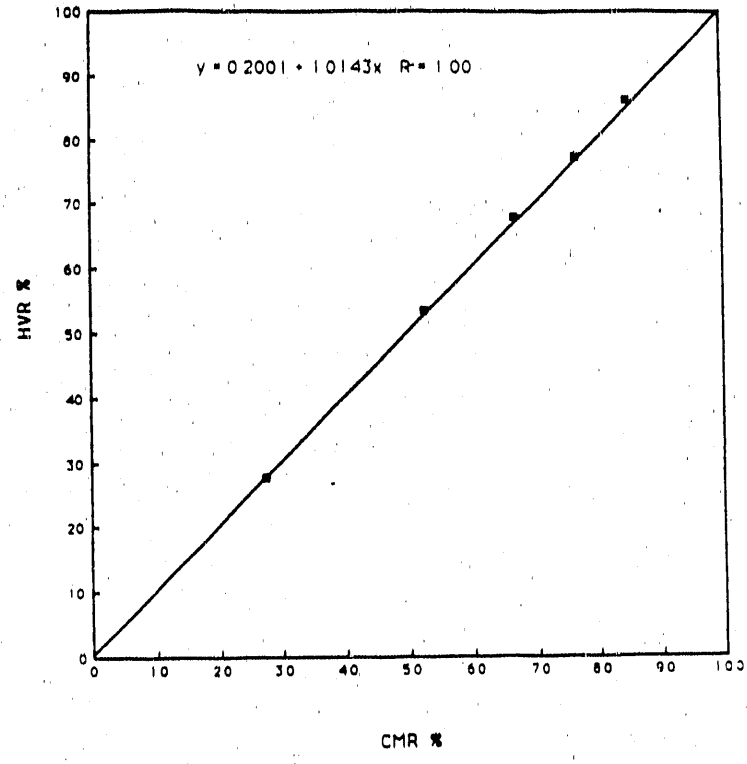

Figure 3.6 - Relation between CMR and HVR for Illinois No. 6 coal.

3.3 QA/QC for Standard Flotation Tests and Coal Analyses

In order to check the reproducibility and repeatability of the standard flotation tests and coal analyses, a QA/QC program was conducted during this reporting period. In this study, 500-gram samples (minus 1/4-inch in size) of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals were wet-ground to 95% minus 200 mesh using our standard grinding procedures. The ground samples were then split into four parts, designated as A, B, C and D, using a stainless steel riffle (8-1/4 inch x 1 inch in size with 32-1/4 inch chutes). Parts A, B, and C were used to conduct three standard flotation tests, and part D was reserved for analysis. Subsequently, each sample of flotation feed (part D), concentrate, and tailings was analyzed for ash, total sulfur, and pyritic sulfur. In order to provide enough material for QA/QC analyses, the three flotation concentrates and tailings for each coal were combined and homogenized. The feed and the combined concentrate and tailing for each coal were then split into four parts. One

Table 3.1 - Relationship between HVR, Yield, and CMR for Upper Freeport PA coal.

Sample No.	Yield %	Ash %	Btu/ lb	CMR %	HVR %
1	24.2	3.54	15087	26.6	29.7
2	51.7	5.25	14943	54.7	57.2
3	64.8	50.30	14898	70.3	71.5
4	79.9	6.34	14706	85.7	87.0
5	81.3	4.45	14053	87.7	89.4
6	81.6	4.77	14060	87.9	89.6
7	92.6	8.16	14327	96.8	98.2
Feed			13507		

Table 3.2 - Relationship between HVR, Yield, and CMR for Pittsburgh No. 8 coal.

Sample No.	Yield %	Ash %	Btu/ lb	CMR %	HVR %
1	25.6	4.31	13960	27.5	28.1
2	54.0	4.53	13965	58.1	59.3
3	73.8	4.74	13895	78.7	80.6
4	82.2	5.08	13809	88.4	89.2
5	91.7	7.42	13509	96.1	97.7
Feed			12726		

Table 3.3 - Relationship between HVR, Yield, and CMR for Illinois No. 6 coal.

Sample No.	Yield %	Ash %	Btu/ lb	CMR %	HVR %
1	25.0	7.34	12888	52.4	53.5
2	48.0	7.84	12888	52.4	53.5
3	60.7	6.85	12951	66.6	68.0
4	70.2	4.85	12712	76.4	77.2
5	78.7	9.18	12665	84.7	86.3
Feed			11554		

part was sent to Columbia University and one to the University of Utah to conduct proximate, total-sulfur, and pyritic-sulfur analyses.

Tables 3.4 to 3.6 give the flotation yield, ash and total-sulfur contents of the flotation products of the three base coals. The pyritic-sulfur content of the flotation products of Illinois No. 6 coal and the efficiency index for each separation are also presented in Table 3.4. The results given in these tables clearly show that the standard flotation tests are quite reproducible. The yields obtained for each coal differ by no more than 4.4 percentage points at most (Upper Freeport PA) and only 3.2 percentage points for Illinois No. 6 coal. The differences in the efficiency index for Illinois No. 6 coal is less than 2 points. The ash and total sulfur contents of the flotation products are also quite consistent in the different tests. Furthermore, the average flotation yields obtained in this study, 76.8, 75.9 and 75.3% for Illinois No. 6, Pittsburgh No. 8, and Upper Freeport PA coals, respectively, agree very well with the average values reported in our First Annual Report (77.4% for Illinois No. 6, 76.2% for Pittsburgh No. 8 and 73.0% for Upper Freeport PA coals).

Table 3.7 compares the weight and composition of the split samples from the 200 mesh wet grinds of the three base coals. The ash, total-sulfur, and pyritic-sulfur contents of parts A, B and C in this table are calculated from the analyses of the flotation concentrate and tailings. The results show that the split samples are quite uniform with respect to the ash content, total-sulfur, and pyritic-sulfur contents. The maximum difference in the weight of the split samples is 12 grams, which did not result in any significant effect on the flotation as can be seen from the results given in Tables 3.4 to 3.6.

Table 3.8 compares the results of ash, total-sulfur and pyritic-sulfur analyses conducted at the various laboratories on the flotation feed, combined concentrate and tailings of the three base coals. The results in this table show that the values are consistent and reproducible at each of

Table 3.4 - Reproducibility of the standard flotation tests (dodecane 5.75 lb/T, MIBC 1.16 lb/T, pH 6.6) of Illinois No. 6 coal wet ground to 95% minus 200 mesh.

Split Sample	Product	Yield, %	Ash, %	Tot S, %	Pyr. S %	EI_H
Part A	Conc.	78.3	8.4	3.54	1.15 7.20	47.7
	Tail	21.7	38.2	8.72		
Part B	Conc.	77.0	8.1	3.51	1.04	49.7
	Tail	23.0	37.0	8.50	6.96	
Part C	Conc.	75.1	8.0	3.50	1.10	49.4
	Tail	24.9	35.7	8.53	7.12	
Part D	Feed		14.9	4.66	2.37	

Table 3.5 - Reproducibility of the standard flotation tests (dodecane 1.92 lb/T, MIBC 0.30 lb/T, pH 3.8) of Pittsburgh No. 8 coal wet-ground to 95% minus 200 mesh.

Split Sample	Product	Yield, %	Ash, %	Tot S, %
Part A	Conc.	73.8	5.2	2.73
	Tail	26.2	29.1	8.61
Part B	Conc.	76.4	5.5	2.79
	Tail	23.6	31.3	9.13
Part C	Conc.	77.7	5.4	2.72
	Tail	22.3	31.9	9.02
Part D	Feed		11.6	4.29

Table 3.6 - Reproducibility of standard flotation tests (dodecane 0.48 lb/T, MIBC 0.21 lb/T, pH 3.3) of Upper Freeport PA coal wet ground to 95% minus 200 mesh.

Split Sample	Product	Yield, %	Ash, %	Tot S, %
Part A	Conc.	73.7	6.3	1.27
	Tail	26.3	34.0	4.61
Part B	Conc.	74.1	6.4	1.26
	Tail	25.9	34.3	4.78
Part C	Conc.	78.1	6.8	1.29
	Tail	21.9	38.5	5.57
Part D	Feed		13.9	2.28

Table 3.7 - Comparison of split samples from the 200-mesh wet grind of the three base coals.

Coal	Part	Weight grams	Ash %	Tot. S %	Pyr. S %
Illinois No. 6	A*	111.38	14.8	4.66	2.46
	B	112.31	14.7	4.66	2.40
	C	104.24	14.9	4.75	2.60
	D	115.57	14.9	4.66	2.37
Pittsburgh No. 8	A	121.39	11.5	4.27	
	B	122.06	11.6	4.29	
	C	115.09	11.3	4.12	
	D	117.34	11.6	4.29	
Upper Freeport PA	A	120.64	13.6	2.15	
	B	118.36	13.7	2.17	
	C	114.42	13.7	2.23	
	D	123.03	13.9	2.21	

*Results of split part A, B and C are calculated from the analyses of the flotation concentrates and tailings.

the three test locations, especially the ash and total-sulfur contents. The difference in the pyritic-sulfur contents is also within the range of experimental error.

Table 3.9 shows the combustible material recovery (CMR), ash, and pyritic-sulfur rejection, and the efficiency index resulting from standard flotation as calculated from the analyses obtained by the different research groups using two different methods. In the first method, the results were calculated from the analyses of the flotation feed and concentrate. In the second method, the results are calculated from the analyses of the flotation concentrate and tailing. It can be seen from this table that the results obtained using these two calculation methods with experimental results from three different test locations are quite consistent. The maximum difference in the efficiency index is only 4.3 points between the two different methods and 6.4 points when the data obtained from different test locations are used. The difference in the efficiency index is mainly due to variation in pyritic-sulfur rejections.

Table 3.8 - Comparison of ash, total-sulfur, and pyritic-sulfur analyses conducted at different universities.

Coal Sample	Product	Location	Yield, %	Ash, %	Tot. S, %	Pyr. S, %
Illinois No. 6	D Feed	Berkeley		14.9	4.61	2.25
		Columbia		14.5		2.16
		Utah		14.9	4.73	2.48
	A, B, C Con	Berkeley	76.8	7.9	3.50	1.11
		Columbia		8.1		0.96
		Utah		8.1	3.57	1.01
	A, B, C Tail	Berkeley	23.2	36.5	8.39	6.92
		Columbia		35.8		7.23
		Utah		36.7	8.86	7.64
Pittsburgh No. 8	D Feed	Berkeley		11.5	4.27	2.91
		Columbia		11.3		2.59
		Utah		11.4	4.10	2.82
	A, B, C Con	Berkeley	75.9	5.5	2.70	1.38
		Columbia		5.5		1.18
		Utah		5.4	2.68	1.26
	A, B, C Tail	Berkeley	24.1	30.7	8.97	8.18
		Columbia		30.0		7.88
		Utah		30.5	9.33	9.01
Upper Freeport PA	D Feed	Berkeley		13.9	2.30	1.68
		Columbia		13.9		1.37
		Utah		13.9	2.21	1.56
	A, B, C Con	Berkeley	75.3	6.5	1.27	0.59
		Columbia		6.7		0.50
		Utah		6.5	1.25	0.51
	A, B, C Tail	Berkeley	24.7	35.4	4.88	4.85
		Columbia		35.2		5.10
		Utah		35.3	5.08	4.86

Table 3.9 - Comparison of CMR, ash rejection, pyritic-sulfur rejection, and efficiency index of three base coal calculated using different methods based on the results obtained by different universities.

Coal Sample	Location	CMR %		Ash Rej. %		Pyr. S Rej. %		EI_H	
		M1	M2	M1	M2	M1	M2	M1	M2
Illinois No. 6	Berkeley	83.1	82.8	59.2	58.2	62.1	65.3	45.2	481
	Columbia	82.5	82.6	57.1	57.2	65.9	69.5	48.4	521
	Utah	82.9	82.8	58.2	57.8	68.7	69.6	51.6	524
Pittsburgh No. 8	Berkeley	81.8	81.1	63.8	64.0	64.0	[illegible]	45.1	464
	Columbia	80.9	81.0	63.1	63.4	65.4	[illegible]	46.3	490
	Utah	81.1	81.1	64.1	64.2	66.1	[illegible]	47.2	505
Upper Freeport PA	Berkeley	81.7	81.5	64.6	64.0	73.6	72.9	55.3	544
	Columbia	81.6	81.4	63.7	63.3	72.5	77.0	54.1	584
	Utah	81.7	81.5	64.5	64.0	75.4	75.8	57.1	573

M1 - Results based on the analyses of the flotation feed and concentrate.
M2 - Results based on the analyses of the flotation concentrate and tailings.

4.0 SURFACE MODIFICATION AND CONTROL

Surface modification in fine coal flotation can be directed towards the surface of the pyrite, ash or coal itself. In any case, the ultimate goal is to help improve the separation efficiency of the flotation process on coals that may have been subjected to a range of weathering conditions. During the past quarter work has been at modifying the coal surface to restore hydrophobicity to oxidized coal. The effect of pH has also been investigated in more detail and extensive work has been directed at the effect of various pyrite depressants on the efficiency of coal flotation.

4.1 Testing of surface-modifying reagents

In previous biweekly reports, it was mentioned that the phenol group of the GH series of surface modifiers (whose molecular structures will be discussed later) might play an important role in the action of the reagents with the surface of coal. In order to verify this hypothesis, GHB which has a similar structure to GH0, but lacks the phenolic group in place of a phenolic group in its structure, was tested. Flotation tests were carried out on Illinois No. 6 coal with GHB and GH0, respectively. The results (Table 4.1) show that the yields obtained with GH0 are significantly higher than that obtained with GHB. However, the ash in the clean coal is also increased by about 28% with GH0. The higher yields obtained with GH0 may be due to the interaction of the phenol group of the GH0 with the hydrophilic sites of the coal, while the increase of the ash may be due to both entrapment and lower selectivity of the reagent.

Table 4.1 - The effect of GH0 and GHB on the flotation of 200-mesh wet-ground Illinois No. 6 coal.

	Dodecane lb/T	Frother lb/T	Modifier lb/T	Yield %	Ash %	CMR %
GH0	0	1.16	0.52	75.3	9.03	79.3
	0	1.16	0.78	82.8	9.43	86.9
	0	1.16	1.17	91.8	10.18	95.5
GHB	0	1.16	0.52	55.1	7.51	59.0
	0	1.16	0.78	64.2	7.73	68.6
	0	1.16	1.17	77.8	8.04	82.9

4.2 Restoration of hydrophobicity to lab-oxidized coals.

The effect of the GH series of reagents on laboratory-oxidized coal was investigated. Laboratory-oxidized coal was prepared by first grinding Pittsburgh No. 8 coal to minus-200 mesh following our standard grinding procedures. The ground coal was then stored in slurry form for 120 hours at 60°C and additional distilled water was added periodically to keep the sample from drying out. This sample was then used for flotation tests with dodecane, GH0, and GH4 added to the flotation cell. From the results presented in Table 4.2, it can be seen that the yield is very low when dodecane is used as collector. When either GH0 or GH4 is used, however, the yield returned to levels equivalent to those obtained with unoxidized coals. These initial results indicate that both GH0 and GH4 appear to have the ability to restore the flotability of oxidized coal.

4.3 pH Effects on Coal Flotation

Tests conducted during the course of the project showed that increasing the flotation pH depresses the flotability of both coal and pyrite. However, pyrite flotation was depressed more than that of coal and, therefore, higher selectivity was obtained at moderately higher pH values (in the range 4 - 8). The pH effect on the flotabilities of coal and pyrite may be due to either an increase in the concentration of hydroxide ions or factors such as the precipitation of

Table 4.2 - The effect of dodecane, GH0 and GH4 reagents on the flotation of lab-oxidized 200-mesh wet-ground Pittsburgh No. 8 coal.

	Collector lb/T	Frother lb/T	Modifier lb/T	Yield %	Ash %	CMR %	Ash Rej. %
Dodecane							
	1.92	0.30	0.0	16.9	4.44	18.3	93.6
	3.84	0.30	0.0	47.1	4.50	50.9	81.9
GH0							
	0.0	0.30	0.13	47.0	4.83	50.7	80.6
	0.0	0.30	0.78	80.7	6.17	85.8	57.4
GH4							
	0.0	0.30	1.02	79.4	6.98	83.6	52.6

hydroxide complexes of dissolved metal ions at increased pH's. In order to delineate the two effects, flotation tests were conducted at conditions under which the precipitation of dissolved metal ions can be either minimized or be avoided.

Pre-washing of coal with distilled water: Washing prior to grinding was carried out to remove as much soluble material as possible. The washing procedure consisted of gently stirring 500 grams of 1/4 inch x 0 coal samples in 15 liters of distilled water at pH 4 for 5 minutes and then allowing the solids to condition for one additional hour. The supernatant was decanted and fresh distilled water was added to the solids. The coal samples were washed three times using this procedure. The pH of the water was maintained at 4 (which is close to the natural pH of the slurry) in order to avoid possible precipitation. The washed coal was then reslurried with distilled water and charged into the rod mill for grinding.

Effect of pH in the grinding mill: In all the flotation tests conducted thus far to study the effect of pH, the pH of the slurry was varied in the flotation cell. The pH of the slurry in the rod mill was never adjusted and hence was the natural pH of the slurry. In the case of Pittsburgh No. 8 coal, the natural pH was always around 4. At low pH values the solubility of the majority of the ash constituents is high so that the dissolved ion content of the supernatant of the slurry from the rod mill can be expected to be high. When the pH of this solution is increased during flotation, the dissolved ions may precipitate on the coal and/or pyrite surfaces. Such precipitation can be avoided by conducting the flotation tests at the same pH as that of the slurry from the grinding mill. Since it is not possible to control the pH during batch grinding, slurries of varying pH can be prepared by adjusting the initial pH of the slurry to different values.

Comparison of dissolved ion concentrations in different supernatants: The concentration of dissolved ions in the supernatants of the slurry from the grinding mill is summarized in Table 4.3. The supernatants were obtained under a variety of procedures: i) grinding the coal at its natural

Table 4.3 - Comparison of ionic make-up of supernatants from the grinding mill for Pittsburgh No. 8 coal.

Sample	pH	CONCENTRATION, ppm Ca	Mg	Fe	Al
Unwashed coal (Ground at natural pH)	4.2	72.0	24.0	168.0	0.0
Unwashed coal (ground at varying initial pH values)	4.1	51.0	25.0	171.0	34.0
	4.8	47.0	16.0	113.0	37.0
	5.2	47.0	17.0	5.0	42.0
	7.5	43.0	16.0	1.0	39.0
	9.2	37.0	10.0	5.0	31.0
Pre-washed coal (ground at pH 4)	4.0	24.0	0.0	0.0	21.0

pH, ii) pre-washing the coal prior to grinding to remove as much soluble material as possible and iii) grinding the coal at various pHs, adjusted using sodium hydroxide (the final pH of the slurry will depend on the initial pH). It is important to note that the dissolved ion concentrations shown in Table 4.3 represent equilibrium values at the indicated pH. Our earlier leaching tests indicated that equilibrium is reached within 10 minutes, and since grinding is carried out for 20 minutes, it is reasonable to assume that the concentration of the dissolved ions in the supernatant of the slurry from the grinding mill will be the equilibrium value. It can be seen from this table that the concentration of all the ions identified decreases with increasing pH. The decrease is particularly sharp in the case of iron. These results indicate that, if the pH of the grinding suspension is pH 4.1 and is increased during flotation to 9.2, precipitation of iron equivalent to 166 ppm (that is, 171 ppm less 5ppm, Table 4.3) will occur. In addition, the precipitation of other ions will also occur. If this precipitate coats the surface of coal or pyrite, the flotability of these components may be decreased. However, by carrying out the flotation at the same pH as that of the slurry from the grinding mill, such precipitation can be avoided. The results also show that the concentration of dissolved ions in the pre-washed coal supernatant is much less than that in the supernatant of the unwashed coal. For example, the concentration of iron in the supernatant

of unwashed coal slurry ground at natural pH (4.2) is 168 ppm compared to 0 ppm in the case of pre-washed coal ground at pH 4 (Table 4.3). Also, the concentration of all the ions in the supernatant of washed coal is either equal to or less than the equilibrium concentration in the pH range 4.1 to 9.2. Therefore, in the case of prewashed coal, even if the grinding is carried out at low pH values and increased during flotation, precipitation should not occur.

The results presented in Figure 4.1 indicate that in flotation tests carried out with pre-washed coal or with unwashed coal for which the flotation pH is maintained the same as that of the pH of the slurry from the grinding mill, the flotation recovery is independent of flotation pH in the range of 4 to 9. The flotation recovery of unwashed coal which had been ground at pH's lower than the natural pH (4.2) decreases sharply with increasing flotation pH. These results suggest that the coating of the coal surface by metal hydroxide precipitates is the predominant reason for flotation depression at higher pHs.

To determine whether or not precipitation occurs preferentially on the coal or the pyrite surface, flotation results were plotted in terms of selectivity curves (Figure 4.2). In this figure combustible material recovery is plotted against the pyritic sulfur rejection efficiency. It can be seen from these results that higher selectivity is obtained when flotation is carried out at the same pH as that of the slurry from the grinding mill (as indicated by the shift of the curve towards the upper right hand corner). It should be recalled that under these conditions precipitation of metal hydroxide complexes will not occur. Therefore, it appears that precipitation reduces the floatability of coal more than that of pyrite, though only marginally, resulting in reduced selectivity under precipitation conditions. It is important to note, however, that the selectivity obtained with pre-washed coal is similar to that of the unwashed coal floated under precipitation conditions, that is grinding at lower pH and flotation at higher pH values. This is probably because the pyrite surfaces are oxidized, at least partially, as can be seen from the high concentrations of iron

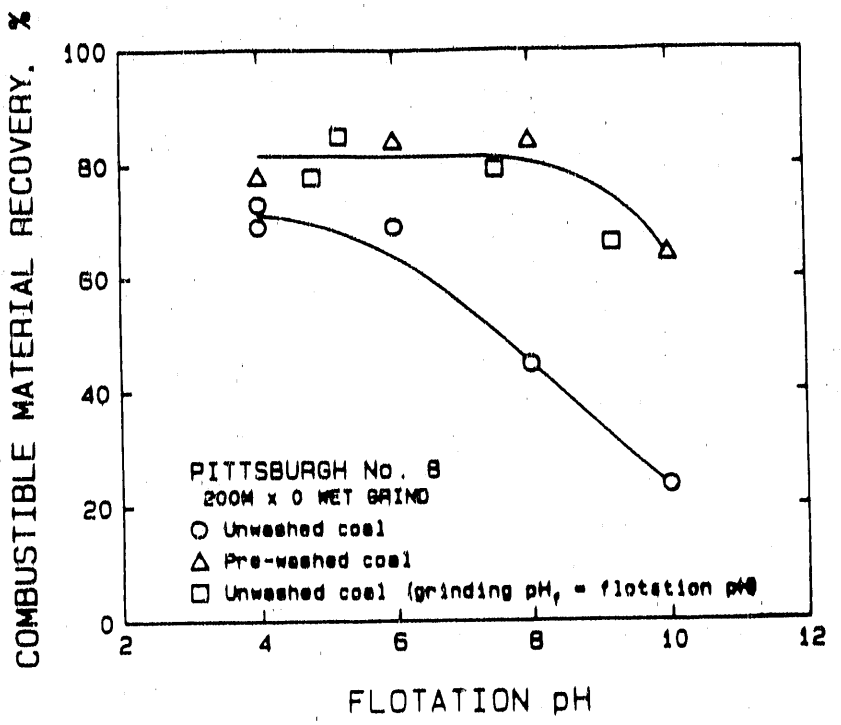

Figure 4.1 - Combustible material recovery vs. pH curves; determination of effect of pre-washing the coal and of maintaining the same pH for flotation as that of the slurry from the grinding mill.

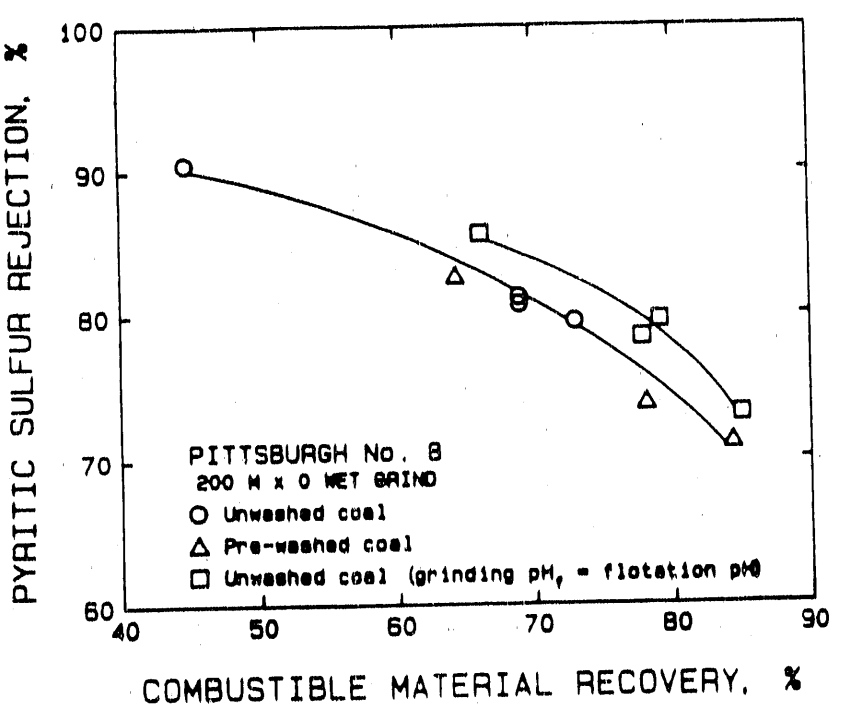

Figure 4.2 - Pyrite rejection efficiency vs. combustible material recovery curves; determination of effect of pre-washing the coal and maintaining the same pH for flotation as that of the slurry from the grinding mill.

in the leach liquor at low pH (4 or lower) values. The high iron concentration can be attributed to the high solubility of iron sulfate in acidic solutions and therefore, pre-washing the coal at pH 4 is likely to result in the dissolution of the iron sulfate layer from the pyrite surfaces. Since the sulfate layer is hydrophilic, the washing process will expose fresh pyrite surfaces which may result in decreased selectivity during coal flotation. This hypothesis can also explain the reason for the previously observed beneficial effect of increasing pH on the pyrite rejection efficiency. Since the solubility of iron sulfate decreases with increasing pH, it is likely that the hydrophilic iron sulfate layer will be removed at lower pH values but will be retained at higher pHs.

4.4 Depression of Pyritic Sulfur

4.4.1 Pyrite depression with Calcium cyanide

According to the conclusions of the Progress Review Meeting No. 6, March 14, 1990, the depression of pyrite during flotation should be studied in order to achieve the project goal of 90% combustible material recovery (CMR) and 90% pyritic-sulfur rejection. For this purpose, the effect of lime and calcium cyanide on the depression of pyrite in Pittsburgh No. 8 coal was tested in our laboratories. The tailings from initial tests with cyanide appeared to be rich in pyrite and encouraged further investigation. The coal samples used in this study were wet ground to minus 200 mesh using the standard grinding procedure. Flotation was performed following the standard procedure except when it was necessary to compensate for any effects of the cyanide or lime.

Table 4.4 summarizes the results of a series of preliminary flotation tests along with the efficiency index, EI_H, for each test. When 9.6 lb/T of lime and 0.14 lb/T calcium cyanide were added, the pH increased to 7.5 and the flotation yield decreased to 70.4% (CMR = 75.5%). In addition, it was observed in the flotation tests that the froth was weak and the flotation response of the coal was slowed down considerably. A further increase in lime addition (12.8 lb/T), with the same calcium cyanide addition, increased the pH to 9.8 and the pyritic-sulfur rejection, but

Table 4.4 - Preliminary flotation test results for Pittsburgh No. 8 coal.

REAGENT DOSAGE				PRODUCT ANALYSIS					REJECTION		
pH	Frot. lb/T	Lime lb/T	CaCN lb/T	Yield %	CMR %	Ash %	Tot.S %	Pyr S %	Ash %	Pyr S %	EI_H —
3.6	0.30	--	--	75.2	80.4	5.4	2.54	1.15	64.8	64.0	44.4
7.5	0.30	9.6	0.13	70.4	75.5	5.6	2.51	--	67.1	54.9*	30.5
9.8	0.30	12.8	0.13	15.8	16.9	6.4	2.37	--	91.8	90.8*	7.7
8.5	0.65	9.6	--**	85.8	90.8	6.4	2.64	1.38	52.7	52.9	43.6
8.7	0.65	9.6	0.14**	86.6	91.7	6.4	2.69	1.39	50.1	53.5	45.1
8.4	0.65	9.6	0.13	78.6	84.1	6.3	2.75	--	59.7	47.9*	31.9
7.8	0.65	9.6	0.13**	86.0	91.6	6.4	2.78	1.38	54.5	56.5	48.1

* Pyritic sulfur rejection and EI calculated from total sulfur analysis data.
** Conditioning time increased by one minute after the addition of calcium cyanide.

decreased the flotation yield to 15.8% (CMR = 16.9%). Increasing the dosage of MIBC from 0.3 lb/T to 0.65 lb/T increased the flotation yield to 78.6% (CMR = 84.1%) but had little effect on the efficiency index. At constant lime addition, calcium cyanide had little effect on the efficiency index of the flotation process.

These preliminary tests show that Pittsburgh No. 8 coal is very sensitive to changes in the pH in the range studied. An increase in pH to 9.8 or so completely depresses the coal. On the other hand, it appears that the addition of lime at moderate levels enhances the CMR, but makes the froth weaker. Therefore, a higher frother dosage than that of the standard may be beneficial. Similar results were reported by the Columbia University research team in previous reports.

Table 4.4 also presents the results of flotation tests in which the flotation pulp was conditioned for one minute with and without calcium cyanide before the addition of the collector. This extra conditioning resulted in an increase in the flotation yield ~(86.0%) and CMR (91-92%) with a pyritic-sulfur rejection of 53-56%. These results suggest that the conditioning time could be an important parameter, however, the addition of calcium cyanide did not seem to have any effect on the efficiency of the separation.

Table 4.5 - Effect of pH on pyrite depression for Pittsburgh No. 8 coal in the absence of calcium cyanide.

FLOTATION CONDITIONS				PRODUCT ANALYSIS					REJECTION		
pH	Cond. t minutes	Lime lb/T	CaCN lb/T	Yield %	CMR %	Ash %	Tot. S %	Pyr. S %	Ash %	Pyr. S %	EI_H
7.8	2	9.9	--	87.9	92.9	6.5	2.7	1.4	50.6	56.8	49.7
8.4	2	9.7	--	85.1	90.3	6.1	2.8	1.4	45.4	59.9	50.2
8.7	2	12.2	--	84.7	89.9	6.1	2.7	1.3	55.3	61.9	51.8
9.3	2	11.4	--	79.8	84.9	5.8	2.6	1.2	59.6	66.0	50.9

As determined earlier, the flotation of Pittsburgh No. 8 coal is very sensitive to pH in both the absence and presence of calcium cyanide and the conditioning time. To delineate the effects of the conditioning time, calcium cyanide addition, and lime addition, the pH of the flotation slurry was controlled with the addition of lime and the conditioning time was increased by two minutes over that of the standard test. The conditioning time was increased so that in those tests in which calcium cyanide was used, the coal would have time to interact with the reagent. Table 4.5 presents the flotation results for Pittsburgh No. 8 coal as a function of pH in the absence of calcium cyanide. The CMR decreases from 92.9% at pH 7.8 to 84.9% at pH 9.3, whereas the pyritic-sulfur rejection increases from 56.8% at pH 7.8 to 66.0% at pH 9.3. The overall efficiency index (EI_H) increases insignificantly, from 49.7 at pH 6.7 to only 50.9 at pH 9.3 which is well within the statistical valuability.

The flotation results for Pittsburgh No. 8 coal as a function of pH conditioned for two minutes with calcium cyanide are presented in Table 4.6. As can be seen, the CMR decreases from 91.8% at pH 6.7 to 84.8% at pH 9.5 and the pyritic-sulfur rejection increases from 56.4 % at pH 6.7 to 66.5% at pH 9.5. Even though there is a significant increase in the pyritic-sulfur rejection, the efficiency index only increases from 48.2 at pH 6.7 to 51.3 at pH 9.5 and these results are nearly identical to those in which no calcium cyanide was added.

In order to complete this study, the effect of conditioning time was determined. Since the efficiency index seems to level off at pH 8.5 or so, experiments to evaluate the effect of

Table 4.6 - Effect of pH on pyrite depression for Pittsburgh No. 8 coal in the presence of 0.14 lb/T calcium cyanide.

FLOTATION CONDITIONS				PRODUCT ANALYSIS					REJECTION		
pH	Cond. t minutes	Lime lb/T	CaCN lb/T	Yield %	CMR %	Ash %	Tot. S %	Pyr. S %	Ash %	Pyr. S %	EI_H
6.7	2	8.9	0.14	86.9	91.8	6.5	2.8	1.5	51.0	56.4	48.2
8.2	2	10.7	0.14	86.8	92.0	6.2	2.7	1.3	53.4	61.2	53.4
8.8	2	9.8	0.14	85.1	90.2	6.2	2.7	1.4	54.1	59.6	49.8
9.5	2	11.5	0.14	79.8	84.8	6.0	2.5	1.2	58.6	66.5	51.3

conditioning time on pyrite depression were carried out at pH 8.5 ± 0.3. The effect of conditioning time on the flotation response of Pittsburgh No. 8 at pH 8.5 in the absence of calcium cyanide is presented in Table 4.7. It can be seen that the CMR remains almost constant, whereas, the pyritic-sulfur rejection increases from 52.8% at one additional minute of conditioning to 61.9% at two minutes, reaching a maximum. Further increasing in the conditioning time to four minutes results in a lower pyritic-sulfur rejection (57.7%). Because the CMR values are nearly identical, the EI_H values follow the same trend as the PSR values. Initially, it increases from 43.6 at one minute of conditioning time to 51.8 at two minutes of conditioning time, decreasing to 47.7 at four minutes of conditioning time. Further, the differences in pyritic-sulfur content of the coal are minor and therefore these differences in the pyritic-sulfur rejections should be evaluated accordingly.

Calcium cyanide did not have any significant effect on the flotation of Pittsburgh No. 8 coal, as can be seen by comparing Tables 4.7 and 4.8. A few additional tests will be made to determine

Table 4.7 - Effect of conditioning time on pyrite depression for Pittsburgh No. 8 coal in the absence of calcium cyanide.

FLOTATION CONDITIONS				PRODUCT ANALYSIS					REJECTION		
pH	Cond. t minutes	Lime lb/T	CaCN lb/T	Yield %	CMR %	Ash %	Tot. S %	Pyr. S %	Ash %	Pyr. S %	EI_H
8.5	1	10.0	--	85.8	90.8	6.4	2.7	1.4	52.7	52.8	43.6
8.7	2	12.2	--	84.7	89.9	6.1	2.7	1.3	55.3	61.9	51.8
8.8	4	11.9	--	84.6	90.0	6.6	2.7	1.4	54.2	57.7	47.7

Table 4.8 - Effect of conditioning time on pyrite depression for Pittsburgh No. 8 coal in the presence of 0.14 lb/T calcium cyanide.

FLOTATION CONDITIONS				PRODUCT ANALYSIS					REJECTION		
pH	Cond. t minutes	Lime lb/T	CaCN lb/T	Yield %	CMR %	Ash %	Tot. S %	Pyr. S %	Ash %	Pyr. S %	EI_H
8.7	1	10.2	0.14	86.6	91.7	6.4	2.8	1.4	50.1	53.5	45.2
8.2	2	10.7	0.14	86.8	92.0	6.2	2.7	1.3	53.4	61.2	53.2
8.6	4	11.5	0.14	86.9	92.4	6.3	2.7	1.3	54.0	56.6	49.0

the effect of calcium cyanide and lime directly to the grinding mill, using conditions selected from previous tests. (Note: Our plans are now to drop further work with CaCN since its effectiveness appears limited.)

4.4.2 Pyrite depression with xanthated reagents

Flotation tests were conducted with a minus 200 mesh grind from Pittsburgh No. 8 coal to test the effectiveness of xanthated reagents as pyrite depressants. The reagents tested were citric-xanthate and sucrose-xanthate. These reagents were synthesized in the laboratory using the procedure briefly described below. This procedure is similar to that used by Attia[1] and co-workers (1988) for preparing polyxanthate dispersants based on polyacrylic acid.

1) 4.8 grams of citric acid (or 4.3 grams of sucrose) was added to 25 ml of distilled water in a 150 ml cylindrical glass vessel provided with a jacket. Cold water was circulated through the jacket in order to maintain the contents at $8^{o} \pm 1\ ^{o}C$.

2) Six grams of sodium hydroxide pellets were added slowly into the vessel and the contents stirred till the solution became clear.

3) 11.4 grams of carbon disulfide (CS_2) was then added to the solution in several increments, and continuously stirred between additions. The incremental dosage was chosen in such a way that the temperature of the solution could be maintained within $\pm 1\ ^{o}C$.

4) After completion of carbon disulfide addition, the solution was further stirred for 5 hours at $8^{o} \pm 1\ ^{o}C$.

[1]Attia, Y.A., Bavarian F., and Driscoll, K.H., "Use of polyxanthate dispersant for ultrafine pyrite removal from high sulfur coal by selective flocculation" Coal Preparation vol. 6 (1988) 35-51.

5) The contents of the vessel were then transferred into a 250 ml Erlenmeyer flask and stored in the refrigerator.

6) Unreacted CS_2 was removed at the end of reaction period by using n-heptane and decanting the organic layer with a separatory funnel.

Since we used a different base (sucrose or citric acid) it is possible that the reaction did not proceed to completion. The degree of completion of the reaction can be checked by analyzing the purity of the product by UV-spectrophotometer. However, since our tests were preliminary in nature the purity of the products were not considered to be critical at this stage.

In flotation tests, the depressant was added to the cell prior to the addition of dodecane. The conditioning time was set at five minutes, and the remainder of the standard flotation procedure was followed. The variables tested were i) depressant dosage, ii) pH and iii) frother dosage.

The results of the flotation tests carried out using citric-xanthate and sucrose-xanthate are summarized in Tables 4.9 and 4.10, respectively. It can be seen from these results that, under the pH and the depressant dosage range tested, the addition of the xanthated reagents above depressants did not cause any significant improvement in the flotation performance.

It was thought that dissolved ions may have been interacting with the xanthate functional groups and, therefore, it was decided to evaluate the performance of the two xanthated reagents, which were not effective in the presence of dissolved ions, at conditions under which dissolved ions are present at very low levels. Flotation tests were conducted to determine the effect of citric-xanthate and polyacrylate-acryiodithiocarbonate (polyxanthated acrylic acid) on the flotation performance of 200 mesh wet-ground, pre-washed (with distilled water) Pittsburgh No. 8 coal.

Results of flotation tests carried out using citric-xanthate and polyacrylate-acrylodithiocarbonate (polyxanthated acrylic acid) on prewashed Pittsburgh No. 8 coal are summarized in Tables 4.11 and 4.12, respectively. It can be seen from these results that there may be a small improvement in the efficiency index value at pHs 6 and 8 upon the addition of 4lb/T of citric xanthate. However, the maximum value of efficiency index obtained, 65.2, is marginally less than

Table 4.9 - Effect of citric-xanthate addition on the flotation performance of wet-ground 200 mesh Pittsburgh No. 8 coal (standard collector dosage was used in all tests).

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		
Flot. lb/T	Depr. lb/T	pH	Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	EI_H
0.88	0.00	4	87.3	94.2	5.6	1.01	60.9	67.5	61.7
0.88	0.40	4	87.9	94.8	5.6	1.14	60.6	64.2	59.0
0.88	2.00	4	88.7	95.9	5.4	1.12	61.7	64.5	60.4
0.88	4.00	4	81.5	87.7	5.8	1.16	62.2	66.2	53.9
0.44	2.00	4	70.2	76.3	4.9	1.00	72.5	74.9	51.2
0.88	0.00	6	83.6	90.1	5.7	0.94	61.9	71.9	62.0
0.88	0.40	6	77.5	84.2	4.9	0.85	69.6	74.4	58.6
0.88	2.00	6	80.9	88.0	4.8	0.87	68.9	72.7	60.7
0.88	4.00	6	77.9	84.4	5.2	0.79	67.6	78.0	62.4
0.88	0.00	8	84.6	90.9	6.0	0.87	59.4	73.7	64.6
0.88	2.00	8	66.3	72.1	4.8	0.63	74.5	85.1	57.2
1.16	0.40	8	77.8	84.7	4.7	0.69	70.7	80.8	65.5
1.16	2.00	8	79.1	86.0	4.9	0.80	69.0	77.4	63.4
1.16	4.00	8	81.5	88.8	4.7	0.74	69.4	78.5	67.3

Table 4.10 - Effect of sucrose-xanthate addition on the flotation performance of wet-ground 200 mesh Pittsburgh No. 8 coal (standard collector dosage was used in all tests).

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		
Frot. lb/T	Depr. lb/T	pH	Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	EI_H
0.88	0.00	4	87.3	94.2	5.6	1.01	60.9	67.5	61.7
0.88	0.04	4	85.0	92.0	5.3	1.05	64.0	68.1	60.1
0.88	0.08	4	81.2	87.8	5.4	0.94	64.9	72.7	60.5
0.88	0.00	6	83.6	90.1	5.7	0.94	61.9	71.9	62.0
0.88	0.04	6	79.2	85.7	5.3	0.87	66.4	75.4	61.1
0.88	0.08	6	71.7	77.8	5.0	0.76	71.3	80.5	58.3
0.88	0.40	6	8.7	9.3	6.5	0.79	95.5	97.5	6.8
0.88	0.00	8	84.6	90.9	6.0	0.87	59.4	73.7	64.6
0.88	0.04	8	81.5	87.9	5.6	0.83	63.5	75.8	63.7
0.88	0.08	8	80.2	86.6	5.5	0.77	64.7	77.9	64.5
0.88	0.00	10	79.1	85.4	5.5	0.82	65.2	76.8	62.2
0.88	0.08	10	76.5	83.1	5.0	0.79	69.4	78.4	61.5

Table 4.11 - Effect of citric-xanthate addition on the flotation performance of wet-ground 200-mesh prewashed Pittsburgh No. 8 coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		
Froth. lb/T	Depr. lb/T	pH	Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	EI_H
0.59	0.00	4	82.2	89.0	5.3	1.01	65.1	70.3	59.3
0.59	0.80	4	81.0	87.6	5.4	1.07	69.0	65.0	56.6
0.59	1.60	4	80.4	87.1	5.2	1.02	66.6	70.7	57.8
0.59	4.00	4	77.0	83.7	4.9	0.95	69.8	73.9	57.6
0.59	0.00	6	87.5	94.6	5.4	1.05	62.2	67.2	61.8
0.59	0.40	6	87.4	94.7	5.2	1.11	63.6	65.4	60.0
0.44	0.00	6	82.6	89.8	4.9	0.99	67.6	70.8	60.6
0.44	0.80	6	81.2	88.4	4.7	0.99	69.5	71.3	59.7
0.44	4.00	6	89.6	97.5	4.8	1.02	65.6	67.4	64.8
0.44	0.00	8	77.9	84.8	4.7	0.90	70.7	75.0	59.8
0.44	0.80	8	76.8	83.8	4.5	0.94	72.4	74.2	58.0
0.44	1.60	8	80.5	87.9	4.5	0.95	71.0	72.7	60.5
0.44	4.00	8	85.5	93.3	4.5	0.92	69.2	71.9	65.2
0.59	0.00	10	73.5	80.4	4.3	0.81	74.7	78.7	59.1
0.59	0.80	10	76.6	83.5	4.6	0.92	71.8	74.8	58.3
0.59	1.60	10	79.1	86.3	4.5	0.88	71.5	75.1	61.5

the value obtained using citric-xanthate on unwashed coal (EI = 67.3). In the case of xanthated polyacrylamide, no improvement was obtained upon addition of this reagent, which indicates that dissolved ions are apparently not the cause for the relative ineffectiveness of the xanthated reagents used.

Table 4.12 - Effect of polyacrylate-acrylodithiocarbonate addition on the flotation performance of wet-ground 200-mesh prewashed Pittsburgh No. 8 coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		
Froth. lb/T	Depr. lb/T	pH	Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	EI_H
0.88	0.00	4	87.3	94.2	5.6	1.01	60.9	67.5	61.7
0.88	0.03	4	82.2	89.2	5.1	0.96	66.5	71.8	61.0
0.88	0.06	4	78.6	85.2	5.1	1.00	67.9	71.9	57.2
0.88	0.12	4	66.3	72.1	4.8	0.88	74.5	79.2	51.3

5.0 FLOTATION OPTIMIZATION AND CIRCUITRY

5.1 Staged Reagent Addition

Results of flotation reagent tests on Upper Freeport PA coal (carried out at the University of Utah) have shown that the addition of either ethanol or butanol as surface modifiers has a beneficial effect on the selectivity that can be achieved by flotation. High-levels of collector (n-dodecane), frother (MIBC), and modifier (alcohol) increase the combustible material recovery (>90%) but decreases the pyritic-sulfur rejection (<77%); the reverse trend occurrs when low-levels are used. Therefore, in order to increase the pyritic sulfur rejection and maintain the high recovery of combustible material, tests with staged addition of collector and frother were conducted.

The optimum collector and frother dosages were added in three stages while the ethanol was added to the mill; the flowsheet is shown in Figure 5.1 and the results obtained are summarized in Table 5.1. It can be seen that in the first stage, the high pyritic sulfur rejection (91.6%) was offset by the corresponding low combustible material recovery (only 49.6%). The cumulative results of all three stages resulted in cumulative CMR of 93% and an overall pyritic-sulfur rejection of 69.4%. Since the previous optimization tests using butanol added to the mill, collector to the cell, and no frother showed promising results, tests with butanol added to the mill and staged collector addition (at a reduced level) were also performed. The flowsheets are shown in Figure 5.2 and 5.3, and the results summarized in Tables 5.2 and 5.3.

Table 5.1 - Flotation results using 6 lb/T ethanol in the mill and staged collector and frother addition to the cell for 200-mesh wet-ground Upper Freeport PA coal.

	PRODUCT ANALYSIS				REJECTION		
Product	Yield %	Ash %	Pyr. S %	ΣCMR %	ΣAsh %	ΣPyr. S %	EI_H
Concentrate #1	45.6	4.07	0.30	49.6	84.4	91.6	41.2
Concentrate #2	31.0	6.88	0.77	82.4	66.1	76.8	59.2
Concentrate #3	10.9	14.11	1.10	93.0	53.1	69.4	62.4

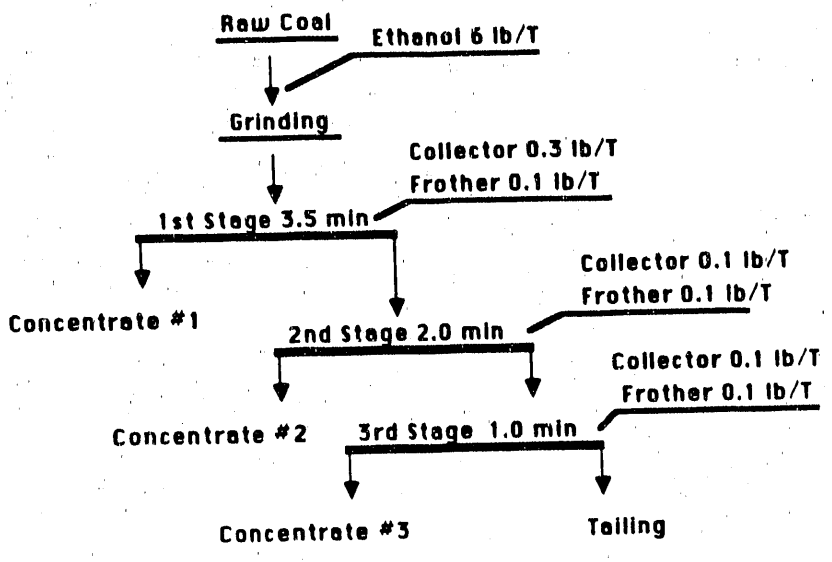

Figure 5.1 - Flotation flowsheet using ethanol in the mill and staged collector and frother addition to the cell.

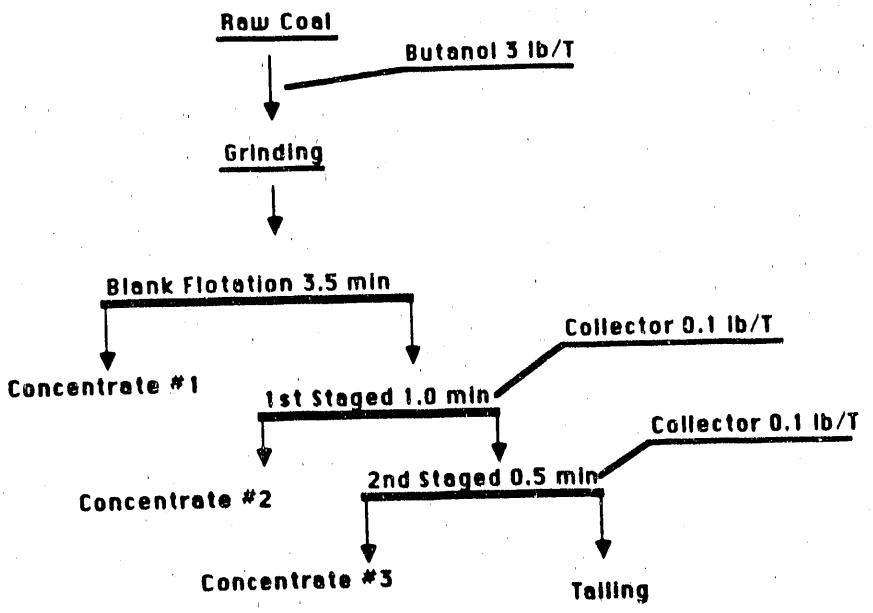

Figure 5.2 - Flotation flowsheet using 3 lb/T butanol in the mill and staged collector addition to the cell.

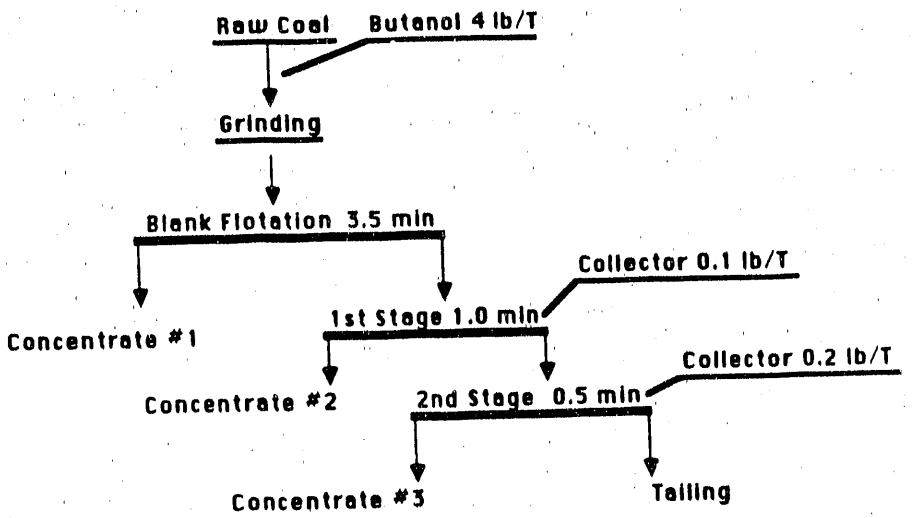

Figure 5.3 - Flotation flowsheet using butanol 4 lb/T in the mill and staged collector addition to the cell.

Flowsheet 5.2 and Table 5.2 indicate that when 3 lb/T butanol is added to the mill and 0.2 lb/T collector is added incrementally to the cell, neither the recovery nor pyritic sulfur rejection are better than the results shown in Table 5.1 where 6 lb/T ethanol was added to the grinding mill.

Flowsheet 5.3 and Table 5.3 indicate that, using 4 lb/T butanol in the mill and 0.3 lb/T collector incrementally added to the mill, the recovery is increased to 94.9% but the pyritic sulfur

Table 5.2 - Flotation results using 3 lb/T butanol in the mill and staged collector addition in the cell for 200-mesh wet-ground Upper Freeport PA coal.

	PRODUCT ANALYSIS				REJECTION		
Product	Yield %	Ash %	Pyr. S %	ΣCMR %	ΣAsh %	ΣPyr. S %	EI_H —
Concentrate #1	53.0	4.19	0.29	58.7	83.6	90.5	49.2
Concentrate #2	13.3	9.29	0.92	82.2	68.1	77.8	60.0
Concentrate #3	6.0	15.21	1.20	88.2	61.4	73.3	61.5

Table 5.3 - Flotation results using butanol 4 lb/T in the mill and staged collector addition in the cell for 200-mesh wet-ground Upper Freeport PA coal.

	PRODUCT ANALYSIS				REJECTION		
Product	Yield %	Ash %	Pyr. S %	ΣCMR %	ΣAsh %	ΣPyr. S %	EI_H —
Concentrate #1	70.6	4.38	0.33	76.6	73.9	85.6	62.2
Concentrate #2	13.3	11.83	0.97	89.9	60.6	77.7	67.6
Concentrate #3	5.6	20.75	1.62	94.9	50.8	72.1	67.0

rejection decreases slightly to 72.1% From these test results, it can be seen that the staged addition of collector and frother did not show beneficial effects for pyrite rejection.

5.2 Cleaning and Scavenging

In order to increase the pyritic-sulfur rejection and maintain the high recovery of combustible materials (or the recovery of heating value), tests with circuits that include cleaning and scavenging operations have been performed.

The test flowsheet with a cleaning and scavenging circuit together with ethanol is shown in Figure 5.4 and the results summarized in Table 5.4. It can be seen that the cleaning operation is beneficial for pyritic-sulfur rejection (93.2%). However, the yield is reduced significantly (50.2%) thus reducing the efficiency index. Therefore, scavenging of the cleaning product should be applied in order to increase the recovery. After two stages of scavenging, the yield increased to 88.8% and pyritic-sulfur rejection decreased to 79.8% giving a separation efficiency of 68.6%.

Table 5.4 - Flotation results using ethanol with cleaning and scavenging operations for 200-mesh wet-ground Upper Freeport PA coal.

	PRODUCT ANALYSIS				REJECTION		
Product	Yield %	CMR %	Ash %	Pyr S %	Ash %	PyrS %	EI_H —
Concentrate #1	50.2	55.0	3.29	0.22	85.9	93.2	48.2
Concentrate #2	24.7	81.3	5.99	0.57	73.3	84.5	65.8
Concentrate #3	7.4	88.8	10.54	1.03	66.7	79.8	68.6
Middlings	4.3	92.2	29.5	2.30	55.8	73.7	65.9

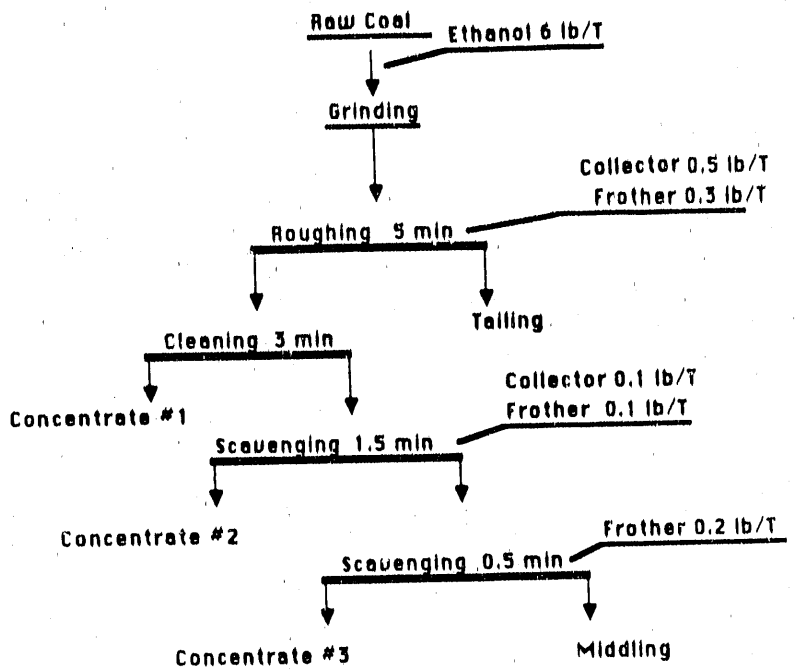

Figure 5.4 - Flotation flowsheet using ethanol with cleaning and scavenging.

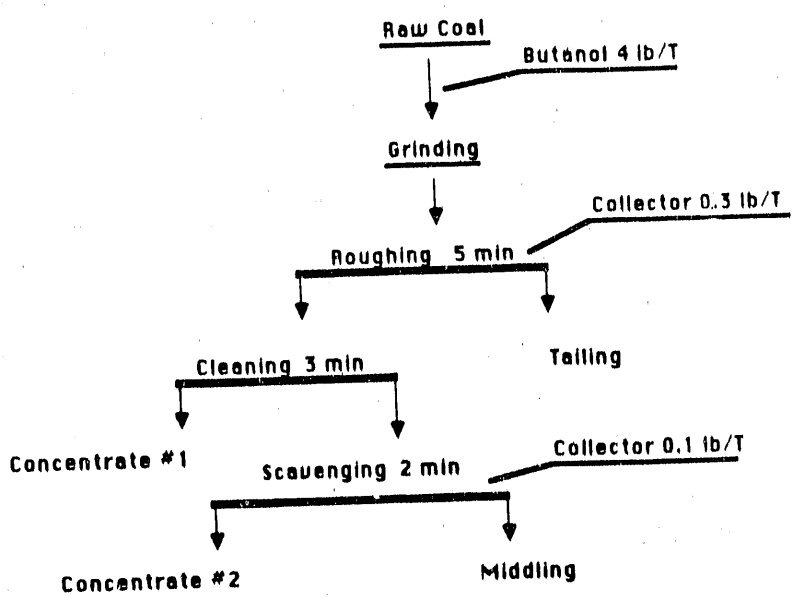

Figure 5.5 - Flotation flowsheet using butanol with cleaning and scavenging.

Table 5.5 - Flotation results using butanol with cleaning and scavenging upon 200-mesh wet-ground Upper Freeport PA coal.

Product	PRODUCT ANALYSIS Yield %	CMR %	Ash %	Pyr S %	REJECTION Ash %	PyrS %	EI_H
Concentrate #1	54.5	59.8	3.18	0.19	85.3	93.6	53.4
Concentrate #2	13.2	73.9	5.18	0.39	78.8	90.4	64.3
Concentrate #3	16.9	90.4	13.72	1.21	59.2	77.8	68.2

Without the cleaning circuit, the CMR was 92.2% and the pyritic sulfur rejection was 73.7%, giving a separation efficiency of 65.9.

The flowsheet of tests using butanol with cleaning and scavenging stages is detailed in Figure 5.5 and the test results are summarized in Table 5.5. The results indicate that a cleaning operation is very effective for pyritic-sulfur rejection (93.6%) but, as with ethanol, the yield is reduced. However, a scavenging cell helps to minimize the decrease in yield and a separation efficiency of 68.2 is achieved.

Based on the foregoing test results, it can be concluded that a cleaning operation is beneficial for pyritic-sulfur rejection with the major problem being some loss in yield. In order to maintain high heating-value recovery, tests should be carried out with scavenging and middling recycling to help increase the clean-coal recovery.

Table 5.6 - Flotation results of four cycles using butanol in the mill upon 200-mesh wet-ground Upper Freeport PA coal.

Product	PRODUCT ANALYSIS Yield %	CMR %	Ash %	Pyr S %	REJECTION Ash %	PyrS %	EI_H
Concentrate #1	57.3	62.5	3.51	0.18	82.6	93.6	56.1
Concentrate #2	77.5	84.0	4.21	0.19	71.8	90.9	74.9
Concentrate #3	79.4	86.3	4.32	0.19	72.3	90.6	76.9
Concentrate #4	81.3	87.7	4.45	0.25	68.7	87.5	75.2
Middlings	25.8		15.84	0.19			

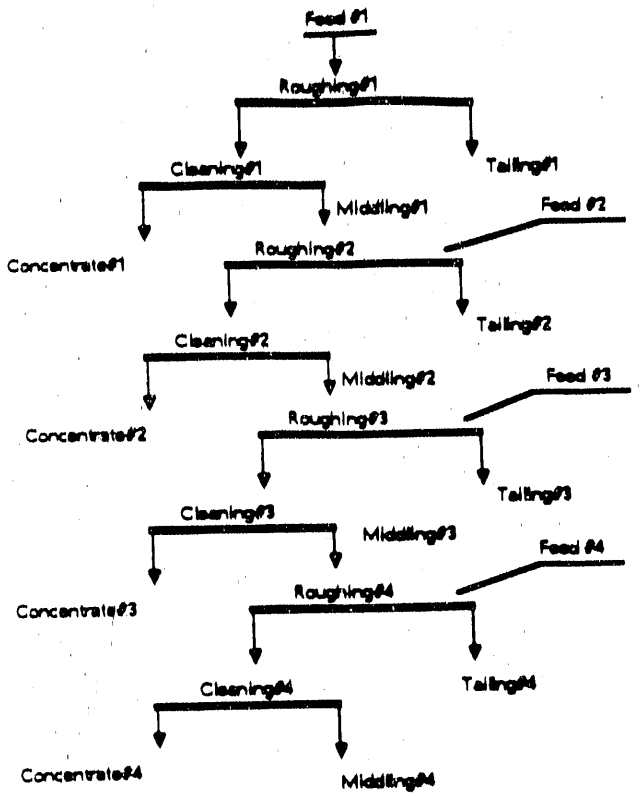

Figure 5.6 - Four cycle flotation circuit.

5.3 Scavenging and Middling Recycling

Work was performed on optimization tests with butanol for the treatment of middlings produced during cleaning operations for Upper Freeport PA coal. In continuous plant operation, the middlings are usually fed back to the feed-conditioning tank or roughing cell to mix with the new feed. In laboratory tests, the simulation of this flowsheet is called "closed-circuit" or "cylic circuit", as shown in Figure 5.6. The middlings of the first batch (Feed #1) are combined with the feed for the second batch (Feed #2), the middlings of the second batch with the feed for the third batch (Feed #3), and so on. The results of a four-cycle flotation circuit, given in Table 5.6, show that after three cycles the cumulative CMR attained was 86.3% with a 90.6% pyritic sulfur rejection, resulting in a separation efficiency of 76.9. It is very encouraging since an efficiency of 80 represents the 90-90 goal of the project and efficiencies based on CMR values have been shown to slightly undervalue the efficiency based on Btu recovery. Repeat tests incorporating more cycles to attain a complete material balance are underway.

6.0 WEATHERING STUDIES

Batch-scale flotation tests using the weathered minus 28 mesh fraction Pittsburgh No. 8 coal samples showed that recovery of combustible material was much lower for samples that had been weathered for 9 months or more compared to those samples weathered for 2 months or less (10 wt% vs. 60 wt%). This observation suggested that material that had been weathered for 9 months or more were more oxidized than earlier increments. The results of zeta potential measurements carried out with weathered Pittsburgh No. 8 coal, however, indicated that the Increment 12 sample (10 months, open mode) was more positively charged, that is less oxidized, compared to samples from earlier increments (Increment 4 and lower, that is 2 months and less of weathering).

In order to determine whether or not the unexpected zeta potential behavior exhibited by the Increment 12 sample is due to possible slime coating of the coal by ash constituents, zeta potential measurements were carried out on coal samples that had been deslimed. The desliming procedure consisted of agitating 0.07 gram of coal (minus 200 mesh material screened from the 28 mesh x 0 sample) for 15 minutes with 80 ml of argon-deoxygenated distilled water for 15 minutes in a 250 ml beaker using a magnetic stirrer. The slurry was allowed to settle for 5 minutes and the supernatant, containing the slimes, was decanted. This procedure was repeated until there were no more fines in the supernatant (three washings were found to be sufficient). The zeta potentials of the deslimed Increment 12 sample as a function of pH are given in Figure 6.1 along with those of the original Increment 12 sample and the (undeslimed) weathered samples from Increment 4 and earlier. It can be seen from this figure that the zeta potential curve of the deslimed samples coincides with that of the coal samples weathered for shorter period. This indicates that a slime coating is probably the cause of the higher zeta potential observed initially for the Increment 12 sample. Also, since there is no noticeable difference between the zeta potential curve of the coal samples weathered for two months and less that of

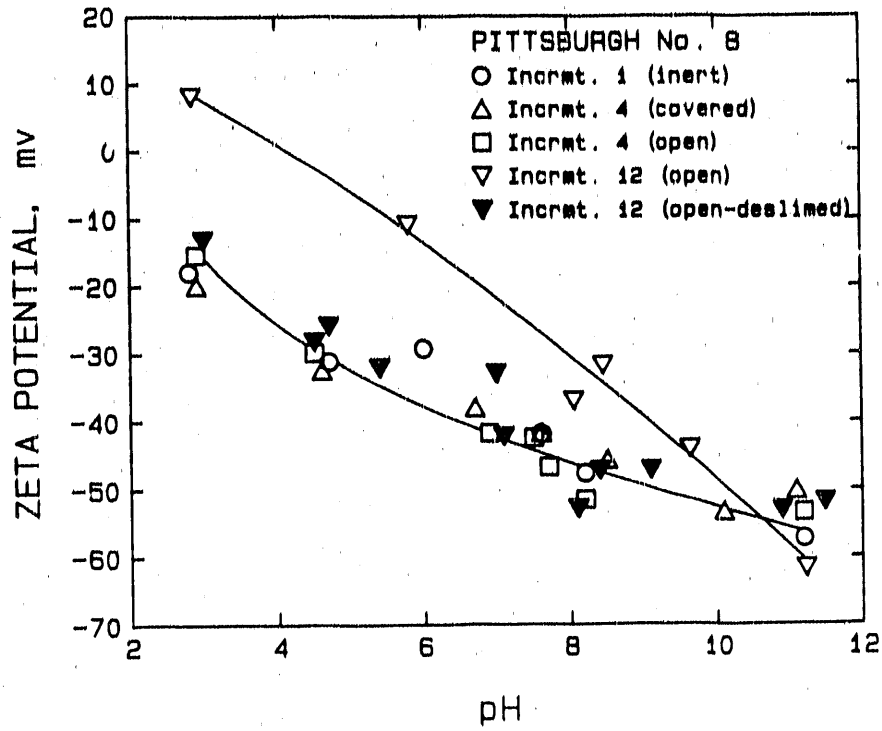

Figure 6.1 - Zeta potential-pH curves of weathered samples of Pittsburgh No. 8 coal.

the Increment 12 sample, it appears that zeta potential behavior is less sensitive to surface oxidation than is flotation.

APPENDIX

DATA FOR FIGURES

Figure 2.1 Particle size distribution for Illinois No. 6 coal washability test samples ground to 100, 200 and 400 mesh top size fraction.

100 MESH X 0		200 MESH X 0		400 MESH X 0	
Size	Cum. % pass.	Size	Cum. % pass.	Size	Cum. % pass.
2.8	4.1	2.8	6.1	2.8	17.3
3.9	6.6	3.9	10.2	3.9	23.4
5.5	10.4	5.5	12.8	5.5	33.5
7.0	15.4	7.8	17.4	7.8	44.1
11.0	21.8	11.0	25.6	11.0	55.9
16.0	29.9	16.0	35.0	16.0	69.1
22.0	37.6	22.0	44.7	22.0	80.8
31.0	47.3	31.0	58.1	31.0	92.2
44.0	56.9	44.0	75.4	44.0	100.0
62.0	65.0	62.0	90.0		
88.0	78.6	88.0	100.0		
105.0	89.7				
150.0	98.2				

Figure 2.2 Particle size distribution for Pittsburgh No. 8 coal washability test samples ground to 100, 200 and 400 mesh top size fraction.

100 MESH X 0		200 MESH X 0		400 MESH X 0	
Size	Cum. % pass.	Size	Cum. % pass.	Size	Cum. % pass.
2.8	2.1	2.8	4.5	2.8	9.9
3.9	4.3	3.9	7.1	3.9	14.6
5.5	8.3	5.5	11.7	5.5	21.8
7.8	13.1	7.8	18.3	7.8	31.0
11.0	18.8	11.0	26.8	11.0	43.5
16.0	25.5	16.0	35.5	16.0	58.8
22.0	32.8	22.0	47.9	22.0	71.6
31.0	41.3	31.0	63.0	31.0	89.0
44.0	51.3	44.0	78.8	44.0	100.0
62.0	60.9	62.0	92.2		
88.0	72.2	88.0	99.8		
105.0	84.4	125.0	100.0		
150.0	97.4				

Figure 2.3 Particle size distribution for Upper Freeport PA coal washability test samples ground to 100, 200 and 400 mesh top size fraction.

100 MESH X 0		200 MESH X 0		400 MESH X 0	
Size	Cum. % pass.	Size	Cum. % pass.	Size	Cum. % pass.
2.8	4.7	2.8	10.7	2.8	13.8
3.9	7.3	3.9	14.5	3.9	19.1
5.5	11.8	5.5	21.9	5.5	28.0
7.8	17.2	7.8	30.5	7.8	39.2
11.0	24.1	11.0	40.8	11.0	51.2
16.0	31.7	16.0	53.2	16.0	64.4
22.0	40.0	22.0	66.1	22.0	77.7
31.0	49.8	31.0	80.5	31.0	90.7
44.0	58.6	44.0	93.7	44.0	100.0
62.0	68.2	62.0	100.0		
150.0	95.9				

Figure 3.1 Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Illinois No. 6 for both undeslimed and deslimed fresh ground samples.

UNDESLIMED		DESLIMED	
Contact time, ms	Fruitful contact	Contact time, ms	Fruitful contact
200	14	100	6
250	22	150	14
300	18	200	14
350	28	250	20
400	24	300	22
420	32	350	34
450	46	400	41
470	46	450	44
500	50	470	51
520	56	500	52
550	51	550	60
570	54	600	66
600	62	650	60
620	60	700	64
670	65	750	82
700	78	800	74
800	74	850	70
900	84	900	80

Figure 3.2 Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Pittsburgh No. 8 for both undeslimed and deslimed fresh ground samples.

UNDESLIMED		DESLIMED	
Contact time, ms	Fruitful contact	Contact time, ms	Fruitful contact
100	22	50	18
150	36	70	18
200	42	100	32
250	46	120	38
260	50	150	46
270	50	170	46
290	56	180	50
300	53	200	52
320	56	210	58
350	59	220	62
400	62	250	72
500	74	280	84
600	76	300	90
		330	90
		350	94

Figure 3.3 Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Upper Freeport PA both undeslimed and deslimed fresh ground samples.

UNDESLIMED		DESLIMED	
Contact time, ms	Fruitful contact	Contact time, ms	Fruitful contact
50	26	50	18
70	34	60	18
80	38	70	32
90	42	80	38
100	46	90	46
110	55	100	46
130	64	150	50
150	73	200	52
200	86	250	58
250	90	300	62
300	96		

Figure 4.1 Combustible material recovery vs. pH curves; determination of effect of pre-washing the coal and of maintaining the same pH for flotation as that of the slurry from the grinding mill.

UNWASHED COAL		PRE-WASHED COAL		UNWASHED COAL	
pH	CMR	pH	CMR	pH	CMR
4	73.0	4	78.0	4.8	77.8
4	69.0	6	84.3	5.2	85.0
6	69.0	8	84.3	7.5	79.1
8	44.8	10	64.4	9.2	66.2
10	23.8				

Figure 4.2 Pyrite rejection efficiency vs. combustible material recovery curves determination of effect of pre-washing the coal and maintaining the same pH for flotation as that of the slurry from the grinding mill.

UNWASHED COAL		PRE-WASHED COAL		UNWASHED COAL	
CMR	Pyr. S	CMR	Pyr. S	CMR	Pyr. S
%	%	%	%	%	%
23.8	95.9	64.4	82.7	66.2	85.6
44.8	90.5	78.04	73.9	77.8	78.4
69.0	80.7	84.3	71.2	79.1	79.6
69.0	81.2	84.3	71.2	85.0	73.1
73.0	79.5				

Figure 6.1 Zeta potential-pH curves of weathered samples of Pittsburgh No. 8 coal.

INCREMENT 1 (INERT)		INCREMENT 4 (OPEN)		INCREMENT 4 (COVERED)		INCREMENT 12 (OPEN)	
pH	Zeta pot. mv	pH	Zeta pot. mv	pH	Zeta pot. mv	pH	Zeta pot. mv
2.8	-18.0	2.9	-15.4	2.9	-20.0	2.85	8.1
4.7	-31.1	4.5	-29.8	4.6	-32.4	5.8	-10.9
6.0	-29.2	6.9	-41.7	6.7	-38.0	8.05	-36.9
7.6	-41.8	7.5	-42.4	7.6	-41.7	8.45	-31.7
8.2	-47.6	7.7	-46.7	8.5	-45.7	9.63	-44.1
11.2	-57.3	8.2	-51.5	10.1	-53.3	11.23	-61.7
		11.2	-53.4	11.1	-50.2		

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