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COAL SURFACE CONTROL FOR ADVANCED FINE COAL FLOTATION

Project No. DE-AC22-88PC88878

Quarterly Report No. 3
April 1, 1989 - June 30, 1989

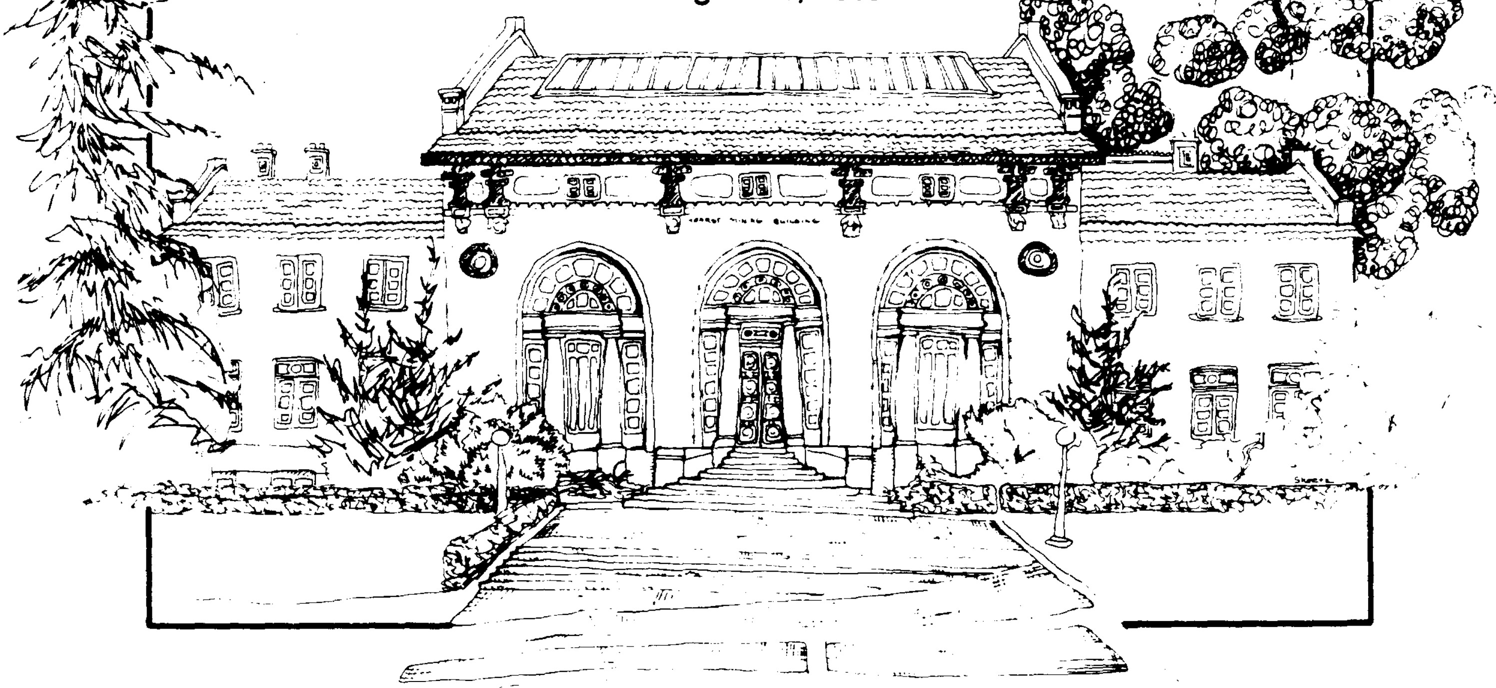
Prepared By

University of California, Berkeley
Columbia University
University of Utah
Praxis Engineers, Inc.

Prepared For

U.S. Department of Energy
Pittsburgh Energy Technology Center
Pittsburgh, Pennsylvania

August 15, 1989



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PREPARED BY

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COAL SURFACE CONTROL FOR ADVANCED FINE COAL FLOTATION

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1.0 INTRODUCTION

Historically coal surface characterization and control have not been seen as critical to coal cleaning owing to the emphasis on keeping particle size as coarse as possible. However, the current goal of near-total removal of pyritic sulfur necessitates fine grinding of coal to liberate the pyrite. At these fine sizes coal surface properties play an increasingly dominant role.

In order to investigate the properties of coal surfaces and their role in coal flotation, DOE awarded a contract to The University of California at Berkeley in October 1988. The project's main goal is to characterize the surface and control the behavior of coal during advance flotation processing. Also, the effect of weathering on the surface characteristics is of interest.

1.1 Scope of this document

The Department of Energy (DOE) awarded a contract entitled "Coal Surface Control for Advanced Fine Coal Flotation", to the University of California at Berkeley, Columbia University, the University of Utah and Praxis Engineers, Inc. The organizational chart for this project is presented in Figure 1.1, which also identifies key project personnel.

This document is the third quarterly report prepared in accordance with the project reporting requirements covering the performance period from April 1, 1989 to June 30, 1989. This report provides a summary of the technical work undertaken during this period, highlighting the major findings. A brief description of the work done prior to this quarter is also provided in this report.

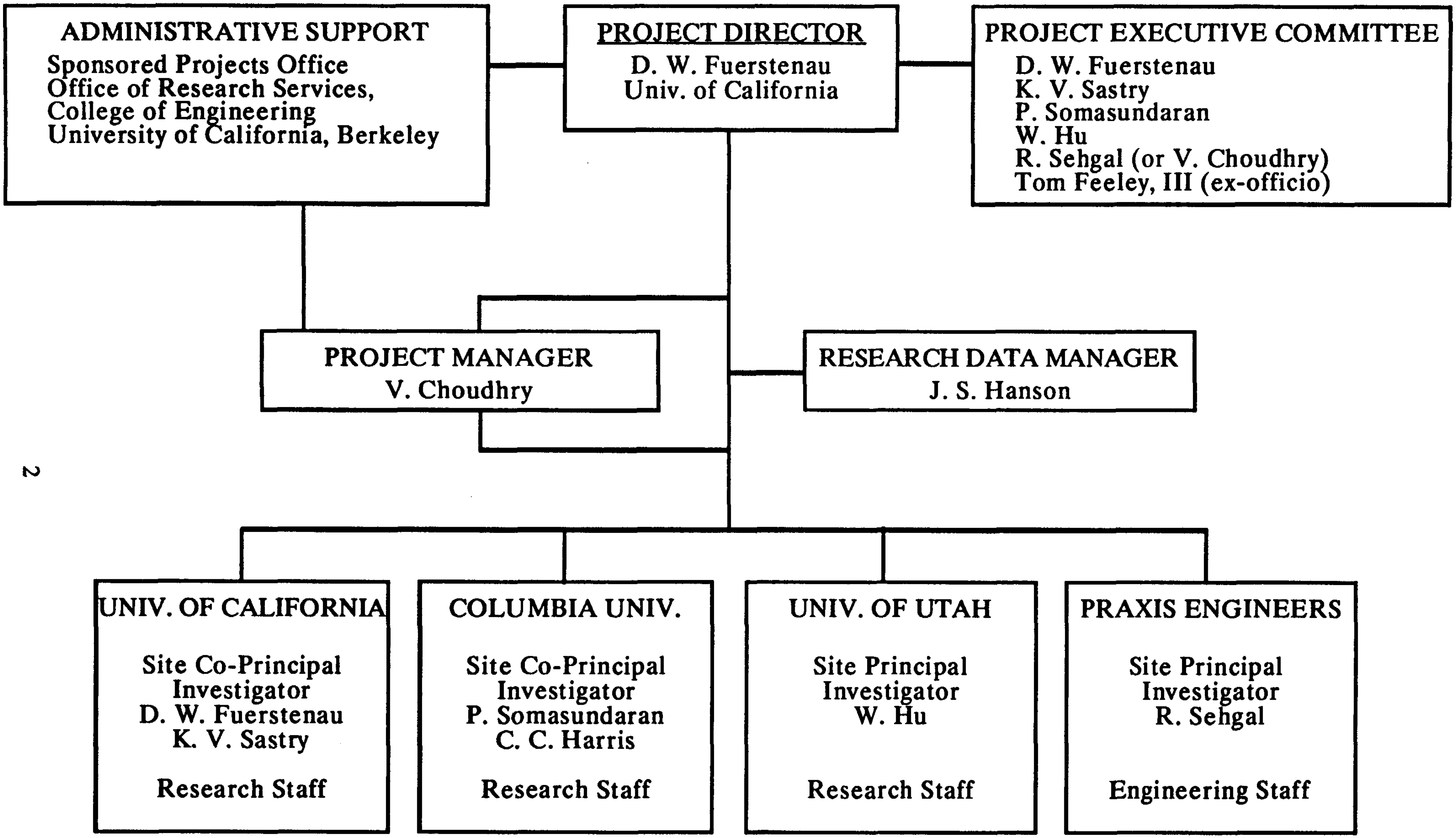

Figure 1.1 - Project organization chart

1.2 Overall Project Scope

The primary goal of this research project is to develop advanced flotation methods for coal cleaning in order to achieve 90% pyritic sulfur removal at 90% Btu yield, 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.

As a part of this contract, large quantities of coal samples have been procured from six major seams identified by DOE for use in this project for advanced flotation and weathering studies. Samples of the same coals are also to be supplied to the University of Pittsburgh for selective agglomeration research.

A second major objective is to investigate factors involved in the progressive weathering and oxidation of coal stored in three storage modes, 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 are to be collected and shipped to both the University of Pittsburgh and the University of California at Berkeley for characterization studies of the weathered coals.

1.3 Work Executed at Different Locations

The project team consists of research and engineering groups at the University of California, Columbia University, the University of Utah and Praxis Engineers, with the University of California acting as the Prime Contractor with DOE. The work proposed to be conducted at the four locations is based on their respective areas of expertise and is detailed in the Project Work Plan. The work undertaken at the various locations is identified in Table 1.1. This report is prepared in an integrated manner, combining work at each location by topic.

The project progress is being maintained in all the technical areas. All the DOE reporting requirements of technical, cost and labor reports were met generally on schedule.

Table 1.1 Work distribution at various locations by tasks.

Task	LOCATION
Project Work Plan: Task 1	
- Task 3 - 8 Work Plan	(B,C,U,P)
- Coal Procurement and Weathering Work Plan	(P)
Coal Procurement and Weathering: Task 2	
- Mine selection, sample procurement, preparation and shipping	(P)
- Weathering coal sampling and shipment	(P)
- Washability analysis	(P)
- Study the effect of weathering on flotation	(B)
Coal Characterization: Task 3	
- Proximate and petrographic analyses	(U)
- Electrokinetic measurements	(C)
- Coal surface functional groups	(C)
- Qualitative analysis of surface composition	(C)
- Morphological characterization	(C)
- Film flotation and contact angle measurement	(B)
Standard Beneficiation Test: Task 4	
- Coal samples for major testing effort	
Illinois No. 6	(B)
Pittsburgh No. 8	(C)
Upper Freeport PA	(U)
- Study the effect of variables such as impeller speed, aeration rate and conditioning times	(B)
- Study the effect of collector and frother	(B,C,U)
- Flotation kinetics tests	(B,C,U)
- Development of standard flotation test	(B,C,U)
Grinding Studies: Task 5	
- Rod mill vs. ball mill evaluation	(B)
- Effect of rod charge	(B,C)
- Development of standard grinding test	(B)
- Effect of collector and frother addition during grinding	(B,C,U)
- Effect of Grinding Environment	(B,C,U)

Table 1.1, continued.

	LOCATION
Surface Modification Studies: Task 6	
- Use of other surface modifying agents	(B,C,U)
Exploratory R&D and Support: Task 7	
- Exploratory R&D	(B,C,U)
- Input to the engineering development effort	(B,C,U,P)
Task Integration and Project Management: Task 8	
- Project reporting to DOE	(B,C,U,P)
- Project Management and Coordination	(B,C,U,P)
- QA/QC implementation	(Executive Committee)

University of California at Berkeley (B)
Columbia University (C)
University of Utah (U)
Praxis Engineers, Inc. (P)

1.4 Work Undertaken During the First Two Quarters

Considerable progress was made in the first two quarters (October 3, 1988 to March 31, 1988) on the project. As presented in Quarterly Reports No. 1 and No. 2, major areas where progress was made during the first two quarters were the following:

- Project Work Plan
  - Project Work Plan was approved by DOE on December 19, 1988
- Coal sample procurement
  - work initiated in November 1988
  - procurement of portable screens, drums, sampling tools, renting of trucks
  - reduction of top size of coal to 4 - 6 inches, with homogenization in the field
  - shipping of homogenized research samples and washability samples

- Setting up of weathering samples tests consisting of:
  - preparation at three base coal sites
  - storage of samples in pre-arranged 15 increments at +1 inch, 1 inch x 1/4 inch and l/4 inch x 0 sizes for covered and inert modes
  - open storage stockpiles for the three size fractions +1 inch, 1 inch x 1/4 inch and 1/4 inch to 0 at the three base coal sites

- Primary and secondary crushing of the three base coals
  - primary crushing to 1 inch nominal top size at Utah under inert (argon) conditions and shipping of 30 lb samples of the three base coals to Berkeley
  - secondary crushing to 1/4 inch nominal top size at Berkeley and Utah and splitting and inerting samples in to 500 g samples

- Development of Standard Grinding Test
  - Establishment of grinding times in a 8-1/2 inch diameter rod mill for 28 and 200 mesh grinds for the three base coals
  - Check of the repeatability and reproducibility of Standard Grinding Test

- Characterization of base coals
  - Physical characterization
  - Petrographic studies

- Development of Standard Flotation Test
  - Selection of DOE flotation cell
  - Study of major variables, air rate, impeller speed, pulp level, frother paddle speed, collector and frother dosage.
  - Establishing suitable values for these variables for all coals
  - Selecting coal specific variables for three base coals
  - Conducting statistical tests
  - Checking the reproducibility of The Standard Flotation Test

2.0 COAL WASHABILITY AND WEATHERING SAMPLING

2.1 Overview and Scope

As a part of Coal Procurement and Weathering (Task 2) effort coal samples were provided to the University of California and the University of Pittsburgh for research purposes. A portion of the parent sample was screened in the field for conducting coal weathering studies on coarse (+1 inch), medium (1 inch x 1/4 inch) and fine (1/4 inch x 0) sizes under inert, covered and open storage modes. Representative splits of the three base coals and three additional coals designated as "other coals" were also used to conduct washability studies. While the details of coal sample procurement, its distribution for various tasks and set up for weathering studies are discussed in the Quarterly Report No. 2, this report covers results of the washability tests conducted on the coals.

2.2 Coal Washability Studies

Washability tests were conducted earlier on the three base coals and three additional coals designated as other coals. The washability tests were conducted on 4 inch x 0, 1 1/2 inch x 0, 1/8 inch x 0, 28 M x 0 and 200 M x 0 coal. The specific gravity levels used were 1.30, 1.35, 1.40, 1.60 and 1.80. The data for the three base coals were compiled as topical reports and submitted to DOE with copies to the University of California, University of Pittsburgh and Kaiser Engineers enabling use of the data as a measure of the degree of liberation. During this quarter the data were analyzed for its consistency as a part of the QA/QC program. Check analyses were conducted on selected samples and data analysis was carried out. The data analysis indicated some discrepancies in the 200 M x 0 washabilities that were performed for the Upper Freeport PA sample, and the results were discussed with the laboratory where the sink-float centrifuging work was done. Subsequently, the tests where the results appeared erroneous were repeated and a revised topical report was issued using the new data. Details of this work are discussed in the following sections. Washability tests were carried out by the following laboratories:

Illinois No. 6	Commercial Testing and Engineering, Henderson, KY
Pittsburgh No. 8 Kentucky No. 9	Geochemical Testing, Somerset, PA
Upper Freeport PA Upper Freeport WV	Gould Energy-Warner Labs, Cresson, PA
Wyodak, WY	Core Labs, Inc., Casper, WY

2.2.1 Washability Data Evaluation Criteria

The sink-float tests were performed by screening coals to different size fractions and each size fraction subjected to sink-float testing. A careful review of the data was carried out to locate any errors in experimental techniques, thus identifying the tests which need to be repeated. In this study, the following three evaluation criteria were used.

(a) Weight percent of 200 M x 0 material - The weight of the 200 M x 0 material in the samples crushed to progressively lower top sizes (that is, 4 inch x 0, 1 1/2 inch x 0, 1/8 inch x 0, 28 M x 0 and 200 M x 0) was compared. An erratic change in the weight percent of this size fraction is indicative that a loss in fines may have occurred during sample preparation.

(b) Analysis of the samples - A comparison of the analysis of the total ash, total sulfur, pyritic sulfur and calorific value was conducted which indicates the accuracy of the sample splitting done during washability sample preparation.

(c) Floats at 1.3 specific gravity - Based on the assumption that size reduction leads to better liberation, the floats at 1.3 gravity should generally show a steady increase. Therefore, comparisons of the floats at 1.3 specific gravity were done to evaluate the consistency of the results.

2.2.2 Washability Data Analysis of Pittsburgh No. 8 coal

The washability of the Pittsburgh No. 8 sample was done by Geochemical Testing, Somerset, PA. Our data evaluation indicates that the results obtained for the Pittsburgh No. 8 sample are internally consistent. In making this conclusion a comparison of the ash, total sulfur, pyritic sulfur and calorific value was made for all the subsamples used in the sink-float tests. A statistical analysis of the data given in Table 2.1 indicates that the sub-samples, as prepared from the main sample are quite consistent as indicated by the low values of standard deviation and variance for all the parameters analyzed. As may be seen, the ash content of all subsamples lies between 11.5% and 12.0%, which is considered excellent.

Table 2.1 Statistical analysis of the composite values of ash and sulfur for Pittsburgh No. 8 sample.

Composite Size	Ash %	Tot. S %	Pyr. S %	Btu/lb %	200 M fines %
4" x 0	11.5	3.90	2.96	13,000	1.8
1-1/2" x 0	12.0	3.91	2.94	12,950	2.8
1/8" x 0	12.0	3.93	2.78	12,950	5.5
28 M x 0	12.0	3.89	2.77	12,850	24.0
200 M x 0	12.0	3.90	2.78	12,750	100
Mean	11.9	3.91	2.85	12900	
Std. Dev.	0.22	0.01	0.08	91	
Variance	0.05	0.0001	0.0007	8198	
SD/Mean	0.018	0.003	0.029	0.007	
95% CI(Mean)	0.27	0.013	0.102	112	

Table 2.2 Comparison of the floats at 1.3 and 1.6 specific gravity for Pittsburgh No. 8 sample.

	FLOAT AT 1.3				FLOAT AT 1.6			
Size	Wt. %	Ash %	Tot. S %	Btu/lb	Wt. %	Ash %	Tot. S %	Btu/lb
4" x 0	18.3	4.34	2.12	14,200	91.6	7.48	3.33	13,700
1 1/2" x 0	31.8	4.57	2.28	14,250	90.4	7.18	3.24	13,800
1/8" x 0	46.4	3.34	1.85	14,400	88.8	6.28	2.79	13,900
28 M x 0	51.1	2.66	1.59	14,400	87.3	5.29	2.21	14,000
200 M x 0	53.9	2.08	1.35	14,500	86.8	4.26	1.48	14,100

In order to estimate any unusual errors in sample handling that might result in the loss of fine coal, the weight of the 200 M x 0 fines was compared for the sample crushed to each top size. The data for the 200 M x 0 fines given in Table 2.1 show a gradual or steady increase in fines, which is expected.

The next parameter evaluated was the weight percent of the floats at 1.3 specific gravity for each of the subsamples. The 1.3 float data for Pittsburgh No. 8 sample given in Table 2.2 indicate a gradual increase in the weight of the float material. The results, plotted in Figure 2.1, indicate that the weight of 1.3 floats increases somewhat rapidly initially with reduction of top size, but becomes asymptotic at about 1/8 inch top size or finer.

The total sulfur and ash content of the 1.3 floats also decreases with decreasing top size with a corresponding increase in the calorific value. The increase in the calorific value for the fine 200 M size may be primarily due to the liberation of hydrogen-rich macerals which tend to concentrate in the low gravity fraction. The physical separation at a low gravity becomes exceedingly difficult especially for fine coal. Even though the sample is dispersed prior to centrifuging the gravity separation of the fine coal is not simple or free from experimental errors.

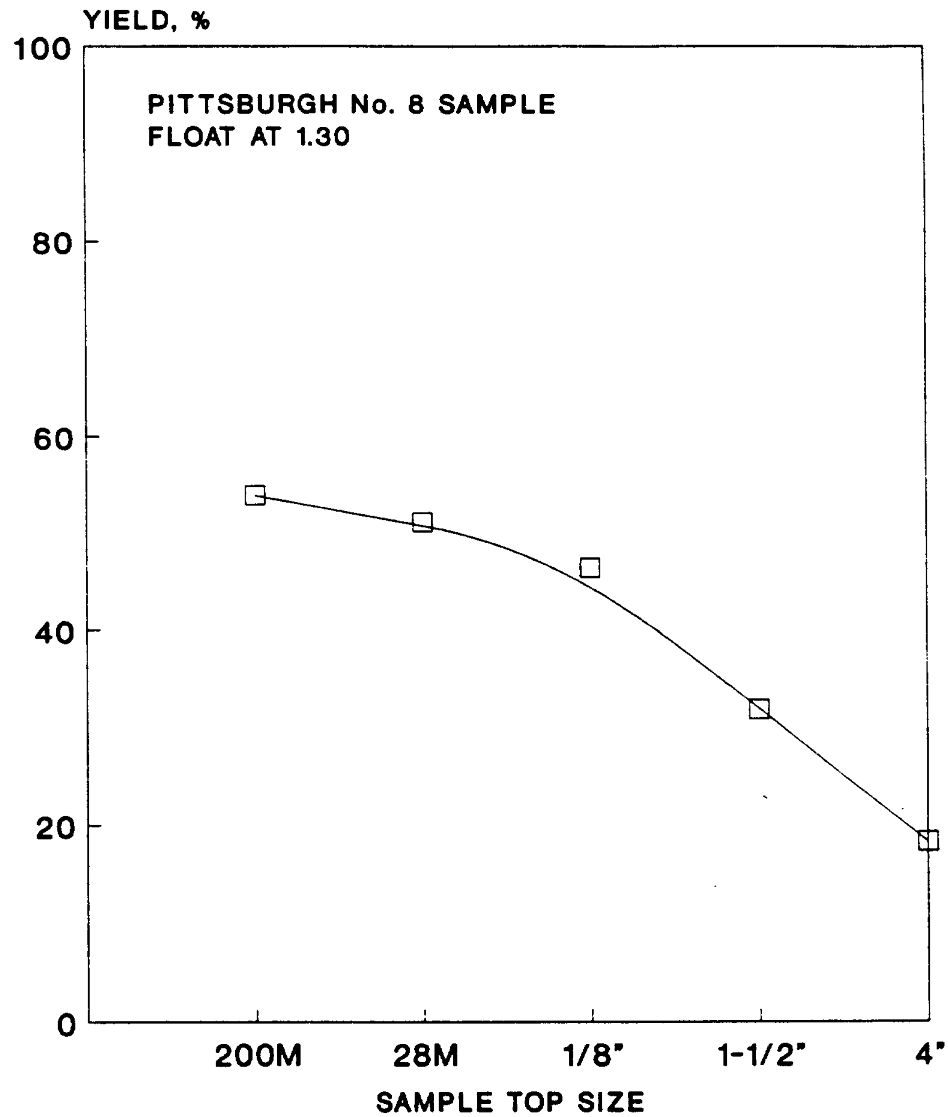

Figure 2.1 Float at 1.3 specific gravity for Pittsburgh No. 8 coal comminuted to progressively finer top sizes.

While the 1.3 float results (Table 2.2) appear to be very consistent, the weight of the float at 1.6 specific gravity showed a minor drop with decreasing size. This may be due to the separation of high-gravity mineral matter which reports to the higher gravity intervals upon liberation, thus causing a loss of weight in the 1.6 floats. Consequently, the ash of the 1.6 floats increases with the decreasing top size which in turn results in a corresponding increase in the calorific value. The same trend is observed for the floats at all other specific gravities tested. The ash content of the 1.6 floats drops consistently with the reduction in top size, resulting in a corresponding increase in the calorific value. The data evaluation presented here indicates that the washability data of Pittsburgh No. 8 sample is internally consistent.

2.2.3 Data Analysis for Upper Freeport PA Coal.

The washability testing of the Upper Freeport PA sample was undertaken by Gould Energy Warner Laboratories Division, Cresson, Pennsylvania. The data analysis included the criteria described earlier. As a first step, the analysis of the composite ash content, total sulfur, pyritic sulfur and calorific value was done for the sub-samples used in the sink-float testing of coal crushed to various sizes. Preliminary data evaluation indicated that the composite values of the ash for the 28 M x 0 and 200 M x 0 samples were considerably lower than those obtained for the coarser samples. The washability tests for the 28 M x 0 and 200 M x 0 were therefore repeated using reserve samples saved from the original test work. The data generated from these repeat tests were submitted to DOE as a revised Topical Report for this coal on July 14, 1989 and are summarized below.

Table 2.3 gives the statistical analysis of the composite washability samples comminuted to various top sizes. As shown in the table, the ash, total sulfur and pyritic sulfur and Btu values fall within a close range indicating the consistency of the sample split by the laboratory. For

Table 2.3 Statistical analysis of composite values of ash and sulfur for Upper Freeport PA.

Composite Size	Ash %	Tot. S %	Pyr. S %	Btu/lb %	200 M fines %
4" x 0	13.02	2.15	1.54	13,450	1.76
1 1/2" x 0	12.78	2.16	1.44	13,500	1.70
1/8" x 0	12.21	2.25	1.58	13,600	5.12
28 M x 0	12.37	2.19	1.34	13,400	21.36
200 M x 0	11.94	2.35	1.42	13,500	100.00
Mean	12.46	2.22	1.46	13,500	
Std. Dev.	0.39	0.08	0.09	71	
Variance	0.15	0.006	0.007	5112	
95% CI (Mean)	0.48	0.093	0.106	89	
SD/Mean	0.031	0.034	0.059	0.005	

example, the ash content is approximately within 0.5% of the mean value of 12.5% which is indicative of good uniformity of head samples for each size fraction.

Also, the weight percent of the 200 mesh material was checked to determine if a loss of fines had occurred during sample preparation. The data reported in Table 2.3 indicates that the increase in the 200 mesh fraction is consistent with the reduction of top size of the sub-sample.

The next parameter studied was the float at 1.3 specific gravity for each of the subsamples crushed progressively finer. The weight of floats at 1.3 specific gravity (Table 2.4 and Figure 2.2) increases steadily with the reducing top size. The highest value was 54.9%, as obtained for the 200 M x 0 sample, with an ash content of 1.87% and a total sulfur content of 0.79%.

Similar to the 1.3 float, a study of the 1.6 float indicates a drop in the weight of the float with reduction in top size. This is attributed to the liberation of mineral matter of high specific gravity, which would result in a loss of weight to the 1.7 and 1.8 gravity intervals. These results are quite identical to those obtained for the Pittsburgh No. 8 sample. Based on the foregoing analysis, it is our conclusion that the Upper Freeport PA washability data are internally consistent.

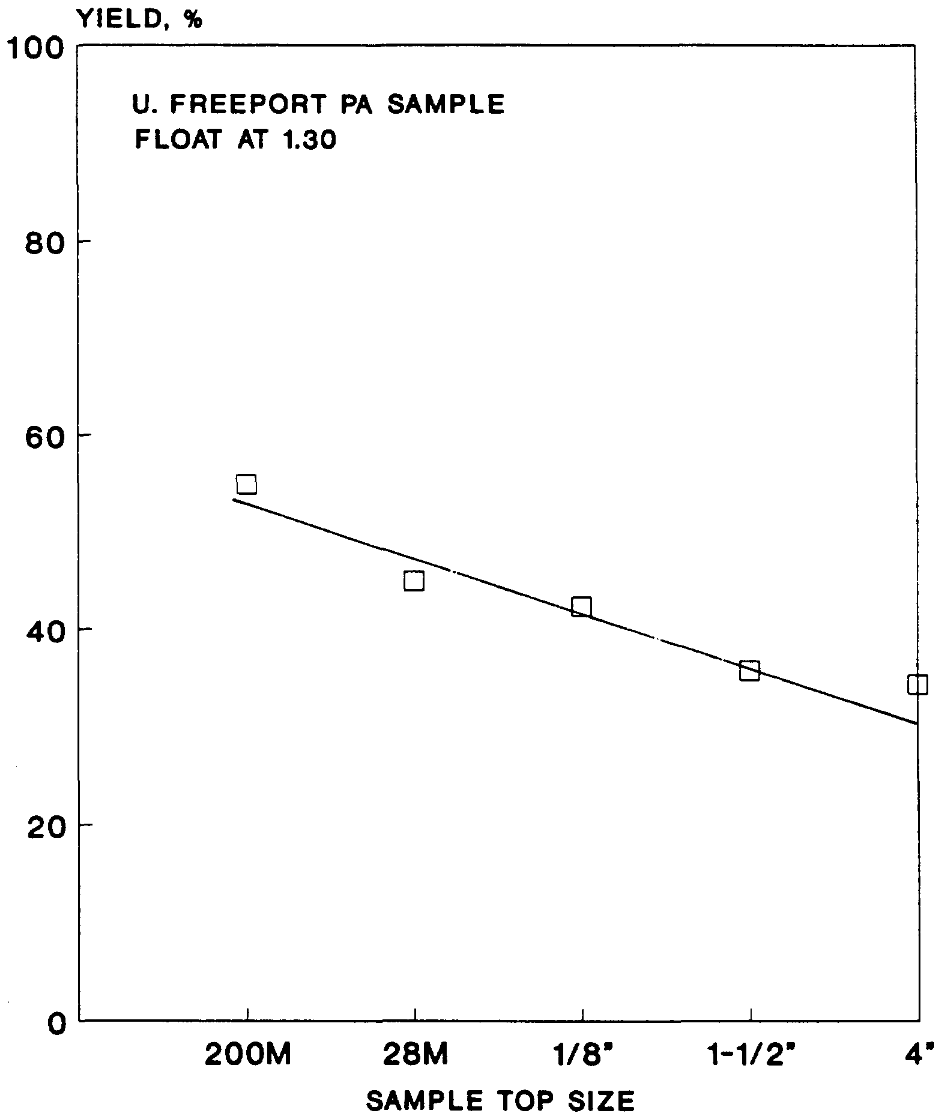

Figure 2.2 Float at 1.3 specific gravity for Upper Freeport PA sample comminuted to progressively finer top sizes.

Table 2.4 Comparison of the floats at 1.3 and 1.6 specific gravity for the Upper Freeport PA sample.

Size	FLOAT AT 1.3				FLOAT AT 1.6			
	Wt. %	Ash %	Tot. S %	Btu/lb	Wt. %	Ash %	Tot. S %	Btu/lb
4" x 0	34.3	3.62	1.23	15,050	91.2	9.16	1.50	14,100
1 1/2" x 0	35.1	3.71	1.20	15,050	91.2	8.98	1.49	14,150
1/8" x 0	42.3	3.29	1.12	15,200	89.9	8.07	1.31	14,350
28 M x 0	44.9	2.32	0.89	15,200	85.4	6.50	1.02	14,500
200 M x 0	54.9	1.87	0.79	15,250	81.0	3.75	0.82	14,600

2.2.4 Data Analysis for Illinois No. 6 Coal.

The sink-float test work for the Illinois No. 6 coal was done at Commercial Testing and Engineering Co. at Henderson, Ky. Washability data evaluation was done by compiling the ash and sulfur analysis of the subsamples crushed to pass various top sizes and the results are presented in Table 2.5. The ash and total sulfur values for all these samples were nearly identical for all the subsamples giving a low value for the standard deviation of 0.26 for ash and 0.17 for total sulfur.

As outlined previously, the weight percent of 200 M fines was also compared for this sample and the results are given in Table 2.5. It is interesting to note that the weight of 200 M material in the 4 inch x 0 sample is only 0.3% while in the sample crushed to 1-1/2 inch x 0 it is 0.2%, which is lower than the parent sample. While this trend is contrary to what would be expected, the relative values of the material in this size fraction are so small for this coal that it is not considered a major problem. The 200 mesh fines content in the subsequent subsamples is consistent, as may be seen in Table 2.5.

A comparison of the floats at 1.3 specific gravity (Table 2.6 and Figure 2.3) indicated that there is a steady increase in the 1.3 specific gravity float up to the subsample crushed to 1/8 inch top size. However, for the samples comminuted to 28 M, as well as to 200 M, the floats at 1.3

Table 2.5 Statistical analysis of composite values of ash and sulfur for Illinois No. 6 sample.

Composite Size	Ash %	Tot. S %	Pyr. S %	Btu/lb %	200 M fines %
4 " x 0	16.4	4.94	2.95	11,850	0.3
1 1/2" x 0	15.9	4.87	2.90	11,800	0.2
1/8" x 0	16.1	4.86	2.97	11,750	4.2
28 M x 0	15.6	4.72	2.88	11,800	6.7
200 M x 0	15.9	4.46	2.68	11,800	100
Mean	16.0	4.77	2.88	11,800	
Std. Dev.	0.26	0.17	0.10	35	
Variance	0.07	0.03	0.01	1207	
SD/Mean	0.017	0.036	0.036	0.003	

specific gravity dropped to below 5%, which is inconceivable. While the matter was taken up with the laboratory involved, a reserve sample of the 200 mesh coal was retested at Gould Energy Laboratory as a part of the QA/QC. Interestingly, the float at 1.3 for the 200 mesh sample obtained from those repeat tests was 34.5%, which is considered consistent with the rest of the results.

Currently, Commercial Testing and Engineering is preparing another sample for repeating the tests with coal comminuted to 28 mesh and 200 mesh top sizes. The data will be evaluated and a revised topical report will be issued for the washability of the Illinois No. 6 coal sample.

2.3 Comparative Study of Data for Three Base Coals.

As a part of QA/QC Program, proximate and ultimate analyses of the samples used for the washability studies and flotation research work were compared. The results of the analytical work done at UCB are provided in Tables 2.7 and 2.8. As may be seen in Table 2.7 the three separate splits originating from the same parent sample for each of the three base coals are identical

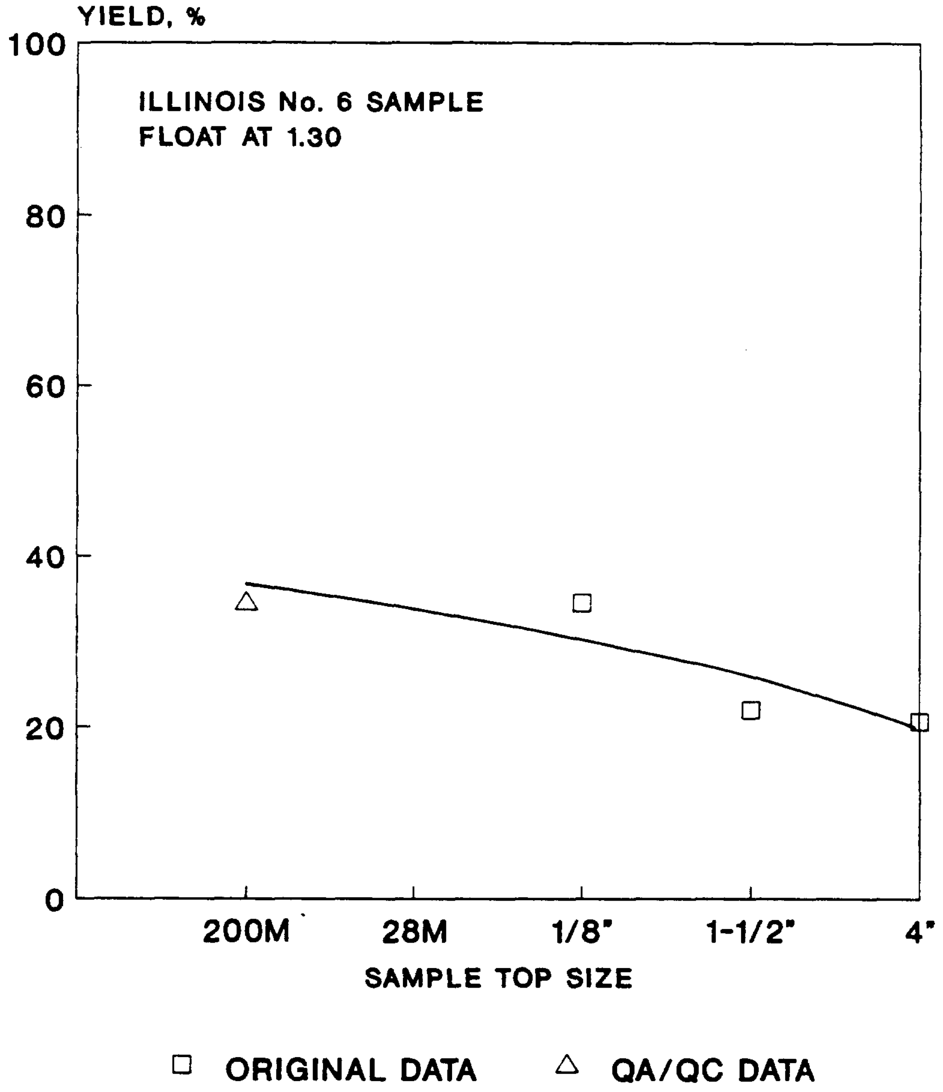

Figure 2.3 Float at 1.3 specific gravity for Illinois No. 6 sample comminuted to progressively finer top sizes.

Table 2.6 Comparison of the floats at 1.3 and 1.6 specific gravity for the Illinois No. 6 sample.

	FLOAT AT 1.3				FLOAT AT 1.6			
Size	Wt. %	Ash %	Tot. S %	Btu/lb	Wt. %	Ash %	Tot. S %	Btu/lb
4" x 0	20.7	4.4	2.98	13,800	87.2	10.7	3.71	12,780
1 1/2" x 0	22.0	4.1	2.92	13,730	87.5	10.44	3.59	12,690
1/8" x 0	34.5	3.34	2.75	13,820	81.5	8.63	3.14	13,010
28 M x 0	1.8	1.31	2.45	14,170	84.9	8.35	3.02	13,010
200 M x 0	5.1	1.89	2.50	14,120	82.3	6.87	2.48	13,320
200 M x 0*	34.5	2.85	2.38	--	81.2	6.26	2.41	--

* Results of QA/QC Test done at Gould Energy Laboratories.

identical. For example, the volatile content of the three samples for Pittsburgh No. 8 coal falls between 35.1% to 36.1%. Similarly, the results of the elemental analyses also fall within a very close range considering that these samples were split in the field and not in a controlled laboratory environment.

Also, a number of sink-float fractions generated during the washability studies were preselected for comparison of the ash, total sulfur and pyritic sulfur analyses. The samples of these fractions were obtained from the commercial labs who conducted the original washability work and were reshipped in a round-robin fashion to another laboratory. The results of the repeat analyses are reported as the QA/QC data in Table 2.9. done by the labs involved.

Table 2.7 Comparison of proximate and sulfur analyses* of base coals

Coal	Moisture %	PROXIMATE ANALYSIS (DRY BASIS) V. Matter %	F. Carbon %	Ash %	Tot. S %
WASHABILITY SAMPLE					
Illinois No. 6	6.34	36.1	46.0	17.9	5.81
Pittsburgh No. 8	1.89	35.1	52.6	12.3	4.15
Upper Freeport PA	0.82	25.4	62.1	12.5	2.29
RESEARCH SAMPLE PREPARED AT UTAH					
Illinois No. 6	4.23	36.0	47.4	16.6	5.27
Pittsburgh No. 8	2.03	36.1	53.0	10.8	4.19
Upper Freeport PA	0.94	26.2	61.4	12.4	2.23
RESEARCH SAMPLE PREPARED AT BERKELEY					
Illinois No. 6	9.50	36.2	46.3	17.5	5.73
Pittsburgh No. 8	2.32	35.7	52.5	11.8	4.28
Upper Freeport PA	1.00	26.2	61.8	12.0	2.38

*The analysis work was done at Berkeley

Table 2.8 Comparison of the elemental analyses* of the base coals.

Coal	Moisture %	Carbon %	Hydrogen %	Nitrogen %	Sulfur %	Oxygen %
	ELEMENTAL ANALYSIS (DRY BASIS)					
WASHABILITY SAMPLE						
Illinois No. 6	6.34	63.9	5.29	1.01	5.81	6.07
Pittsburgh No. 8	1.89	71.0	5.01	1.23	4.15	6.29
Upper Freeport PA	0.82	76.1	4.76	1.34	2.29	3.00
RESEARCH SAMPLE PREPARED AT UTAH						
Illinois No. 6	4.23	63.9	5.04	1.39	5.27	7.88
Pittsburgh No. 8	2.03	72.4	5.06	1.47	4.19	6.07
Upper Freeport PA	0.94	76.3	4.66	1.45	2.23	3.02
RESEARCH SAMPLE PREPARED AT BERKELEY						
Illinois No. 6	9.50	63.8	5.65	1.24	5.73	6.10
Pittsburgh No. 8	2.23	71.0	5.12	1.45	4.28	6.40
Upper Freeport PA	1.00	75.6	4.70	1.45	2.38	3.85

*The analysis work was done at Berkeley.

Table 2.9 Analysis of select sink-float fractions

Grind	Size Fraction	Gravity Fraction	ORIGINAL DATA Ash %	ORIGINAL DATA Tot. S %	ORIGINAL DATA Pyr. S %	QA/QC DATA Ash %	QA/QC DATA Tot. S %	QA/QC DATA Pyr. S %
ILLINOIS NO. 6								
28 M	28 x 200 M	1.60 x 1.80	35.8	5.78	4.27	35.8	6.19	5.98
28 M	200 M x 0	1.40 x 1.60	11.7	2.54	0.77	11.7	2.42	0.69
28 M	200 M x 0	+ 1.80	60.1	16.50	15.66	62.9	16.97	15.05
PITTSBURGH NO. 8								
28 M	28 x 200 M	- 1.30	2.8	1.66	0.40	2.9	1.80	0.33
28 M	28 x 200 M	1.40 x 1.60	17.7	5.78	4.66	17.4	6.60	4.43
28 M	200 M x 0	1.35 x 1.40	4.8	1.46	0.36	4.7	1.66	0.34
UPPER FREEPORT PA								
28 M	28 x 200 M	- 1.30	2.9	0.92	0.61	3.1	0.96	0.20
28 M	200 M x 0	1.35 x 1.40	4.4	0.79	0.48	4.5	0.83	0.09
200 M	200 M x 0	1.35 x 1.40	5.9	0.90	0.14	4.3	0.85	0.08

2.4 Coal Weathering Samples

Sampling of weathering increments from the base coal sites are being carried out according to schedule. Ten weathering increments have been collected as of June 30, 1989 and samples shipped to The University of California and to the University of Pittsburgh.

Samples from all three weathering modes - open, covered and inert - have been collected for all three sizes - +1 inch, 1 inch x 1/4 inch and 1/4 inch x 0. The 1/4 inch x 0 samples of all three weathering modes were screened at 28 mesh at the laboratories and the samples reinerted in plastic bags and shipped along with the other samples.

2.5 QA/QC Analysis of Weathering Samples

The 28 M x 0 screened fractions of the weathering increments were tested for flotation at UCB and the products were analyzed for ash and total sulfur. The reconstituted feed ash and sulfur values from these tests are reported in Table 2.10 for Pittsburgh No. 8 coal. The consistency of ash and sulfur values among all inert, covered and open weathering increments show that we are able to reproduce the sampling and splitting procedures.

Similar results for the Illinois No. 6 and Upper Freeport PA coal are reported in Tables 2.11 and 2.12, respectively.

Table 2.10 Ash and total sulfur of 28 M x 0 screened fraction of Pittsburgh No. 8 weathering samples reconstituted from flotation product analysis.

		INERT		COVERED		OPEN	
Incr. No.	Date	Ash %	Tot. S %	Ash %	Tot. S %	Ash %	Tot. S %
1	11/22/88	17.19	6.42	17.14	6.37	17.13	6.21
2	12/07/88	16.92	6.04	NA	NA	17.91	5.92
4	01/05/89	17.12	6.65	17.18	6.81	18.06	6.63
6	02/10/89	17.63	6.53	16.99	5.96	17.70	6.30
8	04/06/89	17.42	5.86	17.10	6.55	17.31	6.43
Mean		17.26	6.30	17.09	6.42	17.62	6.30
Std. Dev.		0.25	0.30	0.07	0.31	0.35	0.24

Table 2.11 Ash and total sulfur of 28 M x 0 screened fraction of Illinois No. 6 weathering samples reconstituted from flotation product analysis.

		INERT		COVERED		OPEN	
Incr. No.	Date	Ash %	Tot. S %	Ash %	Tot. S %	Ash %	Tot. S %
1	12/07/88	26.93	4.98	NA	NA	26.72	4.84
2	12/20/88	28.11	4.48	NA	NA	30.48	4.90
3	01/04/89	27.67	4.95			30.02	4.62
4	01/17/89	27.65	5.02			26.87	5.14
5	01/31/89	28.05	4.56			28.07	4.74
6	02/14/89	28.48	4.94			NA	NA
7	03/13/89	24.17	4.97			29.36	4.79
Mean		27.3	4.84			28.6	4.84
Std. Dev.		1.35	0.21			1.46	0.16

Table 2.12 Ash and total sulfur of 28 M x 0 screened fraction of Upper Freeport PA weathering samples reconstituted from flotation product analysis.

		INERT		COVERED		OPEN	
Incr. No.	Date	Ash %	Tot. S %	Ash %	Tot. S %	Ash %	Tot. S %
1	11/30/88	9.10	3.25	9.82	3.45	9.46	3.44
4	01/16/89	9.36	2.83	9.48	2.90	9.85	2.82
8	04/07/89	9.43	2.88	9.36	3.11	9.68	2.85
Mean		9.29	2.94	9.54	3.15	9.67	3.04
Std. Dev.		0.14	0.23	0.21	0.23	0.17	0.29

3.0 CHARACTERIZATION OF BASE COALS

Characterization of the coal surface is one of the major objectives of this research project. During the second quarter various characterization studies of both bulk and surface properties were carried out. These focused mainly on weathered samples during the third quarterly period and will be reported in Chapter 7 of this report. The wettability (as assessed by the contact angles) of the research samples of the three base coals was studied in detail. Contact angles of the three base coals were measured using sessile-drop and captive-bubble methods on polished surfaces of pieces of each base coal. These measured contact angles were compared with the values calculated from the critical wetting surface tension measured by film flotation using a model developed in our laboratories.

For contact angle measurement, a hand-picked coal lump was cut to produce flat surfaces using a clean, sharp chisel. The specimen was dry-polished with polishing papers ranging from 240 to 600 grit in an argon-filled glove box. In this way the oxidation of the coal surface during polishing was eliminated. Argon gas was used to blow the debris from the polished surfaces. The dry polished surface was further polished on a polishing wheel with 0.3 micron alumina powder suspended in distilled water. The polished specimen was then washed with triple-distilled water several times to remove traces of the polishing powder. For some measurements, 100 x 150 mesh coal particles were pressed into a pellet in a 0.5-inch diameter mold at 10,000 psi for 10 minutes; contact angles on the pellet were measured in the same manner as the polished specimen.

The "equilibrium" contact angles were measured after a drop or a bubble had made perfect contact with the sample surface. The receding contact angle was measured after adding air to a small bubble that was already attached to the coal surface and the advancing contact angle was measured after removing some air from the same bubble that had been used for measuring the receding contact angle. Each reading is an average of the angles measured on both sides of the drop or the bubble in order to account for any slight specimen tilt or surface roughness. The reported contact angles are the average of at least ten such readings on two specimens for each coal. The contact angles were measured with a Rame-Hart contact angle goniometer.

Initially, dry-polished samples were used in measuring contact angles by the sessile drop method. However, the angles were found to be much larger than those obtained with either the wet-polished samples or coal pellets, as shown in Table 3.1. This behavior may be due to "smearing" of hydrophobic material across the dry-polished surface, resulting in larger and somewhat misleading contact angles.

The advancing, receding and equilibrium contact angles measured by the captive-bubble method are given in Table 3.2. The results show that the receding contact angles are always smaller than the advancing angles. Also, the receding contact angles are closer to the equilibrium contact angles. The difference between the receding and advancing angles is a common observation when measuring contact angles and is generally attributed to surface roughness, generally too fine to see without high-powered microscopes. The results are, however, meaningful. In all cases, Illinois No. 6 is much more hydrophobic than Pittsburgh No. 8 and Upper Freeport PA, which exhibit essentially equal hydrophobicity.

Because the advancing and receding contact angles measured by the captive-bubble technique can vary widely, care must be taken to chose which has the most meaning when comparing the

Table 3.1 Air/water advancing contact angles (in degrees) on the three base coals measured by the sessile-drop method on surfaces prepared by different methods.

Coal Sample	Dry-polished in Argon	Wet-polished in Air	Pellet
Illinois No. 6	70	44	41
Pittsburgh No. 8	82	60	66
Upper Freeport PA	81	62	69

Table 3.2 Air/water contact angles (in degrees) on the three base coals measured by the captive-bubble method on surfaces wet-polished in air.

Coal Sample	Advancing	Receding	Equilibrium
Illinois No. 6	42	23	25
Pittsburgh No. 8	61	45	49
Upper Freeport PA	61	42	50

results to other measurement techniques or physical processes. Since the sessile-drop method involves the addition of liquid to a drop on a surface the resulting measurement is a liquid-advancing contact angle. Analogously, the physical process involved in film flotation is also dependent on the liquid advancing around the coal particles. Table 3.3 shows the results of the contact angles measured from the sessile-drop method and those calculated from film flotation data. The results compare quite well with those of the advancing captive-bubble measurements.

Sessile-drops sitting on the coal surface can also be absorbed by the coal into the pores or dissolve soluble components present at the surface. The effect of time on the contact angle of a sessile-drop on the surface of the three base coals is given in Table 3.4. As before, Illinois No. 6 appears to be the most hydrophilic, with the bubble being completely absorbed by the coal within two minutes. This is indicative that the pores of Illinois No. 6 are hydrophilic and are filled rapidly with the liquid in the droplet. The other two coals again behaved similarly, showing a slow decrease in contact angle for 40 minutes, after which time the contact angles were about

Table 3.3 Air/water contact angles (in degrees) on the three base coals calculated from film flotation results and advancing contact angles measured by the sessile-drop and the captive-bubble methods on surfaces wet-polished in air.

Coal Sample	Film Flotation	Sessile-Drop	Captive-Bubble
Illinois No. 6	52	44	42
Pittsburgh No. 8	63	60	61
Upper Freeport PA	63	62	61

10 degrees indicating that the pores of these two coals have a much lower affinity for water than does Illinois No. 6. It should be noted that as the water absorbs into the coal, the contact angle becomes a receding angle since the liquid/vapor interface is contracting.

In summary, a detailed contact angle study of the three base coals was undertaken to help characterize the wettability of the coal. Good agreement was obtained among the various direct measurement techniques and the calculated contact angles from film flotation results. In all tests, Illinois No. 6 was consistently more hydrophilic than either Pittsburgh No. 8 or Upper Freeport PA coals. These results are consistent with all other characterization studies conducted thus far, and with flotation response.

Table 3.4 The change in contact angle (in degrees) of the three base coals as a function of the time after placing a water drop on a pellet surface (using the sessile-drop method).

	ELAPSED TIME, MINUTES								
Coal Sample	0	0.5	1	2	5	10	15	25	40
Illinois No. 6	46	32	24	7					
Pittsburgh No. 8	68	--	66	65	62	59	49	41	15
Upper Freeport PA	71	69	--	68	65	55	47	32	10

4.0 STANDARD FLOTATION TEST

The conditions used for the Standard Flotation Test were set in the previous quarterly period. However, the reproducibility of the tests at the three research institutions for each coal was not determined. During the past three-month period, the Standard Flotation Test for each of the three base coals were repeated at each of the universities involved and were compared to determine the reproducibility of the procedure.

Because the Standard Flotation Test is the basis for judging the effect of various coal preparation and treatment procedures, data handling becomes an important issue. The efficiency index that will be used to rank the grinding environments and coal surface modification treatments is an important tool in identifying positive effects and the index that is most appropriate depends on the recovery and rejection values. Extensive work has been directed at delineating the most appropriate index for our purposes and these findings will also be discussed in this chapter.

Other accomplishments during this period include the use of a sample of Babcock & Wilcox's kerosene in place of dodecane in the Standard Flotation Test and the use of the Kaiser Pittsburgh No. 8 research sample as the feed for the Standard Flotation Test to determine the integrity of their sample as compared to the research sample used by the Berkeley team.

4.1 Reproducibility of the Standard Flotation Test

The reproducibility of the Standard Flotation Test was investigated as part of the QA/QC program to insure that the results obtained at each university were consistent with each other. The data for the three base coals are given in Tables 4.1 to 4.3. Pittsburgh No. 8 coal is the most thoroughly tested of the three base coals. Table 4.1 shows these results to be quite consistent and reproducible at each of the three universities. The yield for each of the four grind sizes and environments differ by no more than 4.5 percent at most and 2.8 percent in the least.

Table 4.1 Reproducibility of Standard Flotation Tests of Pittsburgh No. 8 coal.

	Institution	Yield %	Ash %	Pyr. S %
28 Mesh, Dry	Columbia	81.3	6.9	2.0
	Berkeley			
	Utah	84.3	6.3	1.5
28 Mesh, Wet	Columbia	86.5	7.5	2.1
	Berkeley	89.8	8.0	
	Utah	85.3	6.6	1.6
200 Mesh, Dry	Columbia	75.6	6.0	1.4
	Berkeley			
	Utah	75.0	6.3	1.2
200 Mesh, Wet	Columbia	76.1	4.9	1.2
	Berkeley	77.8	5.8	1.2
	Utah	74.7	5.1	1.2

Table 4.2 Reproducibility of Standard Flotation Tests of Upper Freeport PA coal.

	Institution	Yield %	Ash %	Pyr. S %
28 Mesh, Dry	Utah	81.8	6.7	0.7
	Columbia	79.8	7.4	0.7
	Berkeley			
28 Mesh, Wet	Utah	75.5	6.4	0.6
	Columbia	79.5	7.5	0.8
	Berkeley	89.3	7.8	
200 Mesh, Dry	Utah	59.1	6.3	0.7
	Columbia	67.1	7.3	0.6
	Berkeley	74.1	7.2	
200 Mesh, Wet	Utah	68.2	5.3	0.5
	Columbia			
	Berkeley	81.3	6.5	0.7

Table 4.3 Reproducibility of Standard Flotation Tests of Illinois No. 6 coal.

	Institution	Yield %	Ash %	Pyr. S %
28 Mesh, Dry	Berkeley	74.6	9.6	1.8
	Utah	75.0	9.1	1.0
	Columbia			
28 Mesh, Wet	Berkeley	88.0	11.0	1.9
	Utah			
	Columbia			
200 Mesh, Dry	Berkeley	60.4	8.8	1.4
	Utah			
	Columbia			
200 Mesh, Wet	Berkeley	77.0	8.5	1.6
	Utah			
	Columbia			

The ash and pyritic sulfur analyses of the products are also reasonably reproducible. There is, however, a small difference in the 28 mesh grind samples.

4.2 Efficiency Index Comparison

An analysis was carried out to compare three definitions of efficiency, namely the Department of Energy's efficiency index (DOE EI), the matrix efficiency formulation ($\{E_{ij}\}$) as devised by Weibai Hu of the University of Utah, and the well-known Hancock separation efficiency (E_H). For the purposes of this illustrative discussion, undesirable or gangue material will be equated with pyritic sulfur and the rest of the desirable material will be equated with the BTU, in accordance with the efficiencies being defined for a binary system of valuables and waste.

It will be seen that DOE EI is non-linear with respect to both gangue rejection with constant yield and to yield with constant gangue rejection, further DOE EI can vary between zero and infinity. Both these aspects lead to difficult visualization of the real effectiveness of a separation corresponding to a number given by the DOE EI. The Hancock efficiency varies between -100% and +100% where negative numbers correspond to deleterious separation processes and the positive region corresponds to a desirable separation. Further, E_H is linear with respect to both gangue rejection with constant yield and to yield with constant gangue rejection. A perfect separation, that is, all valuable mineral being recovered and all gangue being rejected, will give E_H = 100; a sampling operation where no concentration of mineral or gangue takes place gives E_H = 0; and a truly imperfect separation, (where all the mineral is rejected and all the gangue is recovered) gives E_H = -100. This method of measuring efficiency is, perhaps, more easily visualized than the DOE EI methods. All that can be said about E_H can be said about Weibai Hu's formulation precisely because E_H and $\{E_{ij}\}$ are in fact equivalent by definition.

EQUIVALENCE OF E_H AND $\{E_{ij}\}$

The efficiency matrix is defined as;

$$E_{i,j} = (\text{yield}) \times \left\{\begin{matrix} E_{1,i} & E_{1,ii} \\ E_{2,i} & E_{2,ii} \end{matrix}\right\}$$

where $E_{1,i}$ = (% BTU in conc / %BTU in feed),

$E_{1,ii}$ = (%BTU in tail / %BTU in feed),

$E_{2,i}$ = (%Pyr. S in conc / %Pyr. S in feed),

$E_{2,ii}$ = (% Pyr. S in tail / %Pyr. S in feed).

$\{E_{ij}\}$ is defined as the determinant of the above matrix. By a material balance, all material must either be recovered or rejected, therefore,

$$E_{1,i} + E_{1,ii} = 1, \quad \text{and}$$

$$E_{2,i} + E_{2,ii} = 1.$$

Therefore,

$$\{E_{i,j}\} = (\text{yield}) \times (E_{1,i}E_{2,ii} - E_{2,i}E_{1,ii})$$

$$= (\text{yield}) \times (E_{1,i}(1 - E_{2,i}) - E_{2,i}(1 - E_{1,i}))$$

$$= (\text{yield}) \times E_{1,i} - E_{2,i}$$

$$= (\text{yield}) \times \left(\frac{\%\text{BTU in conc}}{\%\text{BTU in feed}} - \frac{\%\text{Pyr. S. in conc}}{\%\text{Pyr. S. in feed}}\right)$$

$$= \left(100\% \times \frac{\text{wt conc}}{\text{wt feed}}\right) \times \left(\frac{\%\text{ mineral in conc}}{\%\text{ mineral in feed}} - \frac{\%\text{gangue in conc}}{\%\text{gangue in feed}}\right)$$

$$= E_H{}^1$$

which shows the equivalence of the two efficiencies.

DOE EI and E_H for constant yield

The standard flotation test results on Illinois No. 6 coal, for 200 mesh dry grind gave an average yield of 60.4% and a pyritic sulfur rejection of 76.4%. For purposes of this illustration of how the efficiency indices vary with recovery and rejection, a standard value of 60% yield and 80% pyritic sulfur rejection will be used. The pyritic sulfur in feed is taken to be 2.70%.

The Department of Energy efficiency index as defined for the project is

$$\text{DOE EI} = \left(\frac{\%\text{ Pyr. S in tail}}{\%\text{ Pyr. S in conc}}\right) \times \text{yield}$$

For a perfect separation all the pyritic sulfur reports to the tailings, the denominator of the above equation goes to zero, and DOE EI goes to infinity. At the other extreme, if the separation is perfectly deleterious all the pyritic sulfur reports to the concentrate, the numerator of the above equation goes to zero, as does the efficiency index DOE EI. All values in between these extremes are possible.

However, for the standard test result as used in this illustration, DOE EI = 360. Figure 4.1 shows how keeping the yield constant at 60% and allowing the pyritic sulfur rejected to vary, affects the indices. DOE EI asymptotically approaches each axis at the extremities, which means at low pyritic sulfur rejection a small perturbation hardly changes DOE EI yet at high pyritic sulfur rejection an equally small perturbation of the pyritic sulfur rejection gives a large change in DOE EI. The Hancock efficiency varies linearly with pyritic sulfur rejection, 100% pyritic sulfur rejection coinciding with $E_H = 60$. No matter how good the pyritic sulfur rejection is, the Hancock efficiency cannot give a value of 100% and cannot indicate a perfect separation because the recovery of the valuable component is less than 100%. DOE EI on the other hand tends to infinity as pyritic sulfur rejection tends to 100% irrespective of the yield and irrespective of the recovery (except for the case when recovery = 0) thus a perfect separation gives DOE EI = infinity. Both this dichotomy of the asymptote, and the non-linearity of the DOE EI graph may be regarded as counter intuitive and is difficult to visualize as a method of grading a separation.

DOE EI and E_H for constant pyritic sulfur rejection

Many of the same comments regarding the Department of Energy efficiency index and the Hancock efficiency apply equally well when pyritic sulfur is kept constant and the yield (and therefore recovery) is allowed to vary. DOE EI asymptotically tends to zero and infinity as yield tends to zero and 100% respectively, as can be seen from the plots given in Figure 4.2. Again E_H is seen to vary linearly while DOE EI undergoes an exponential increase and again E_H cannot

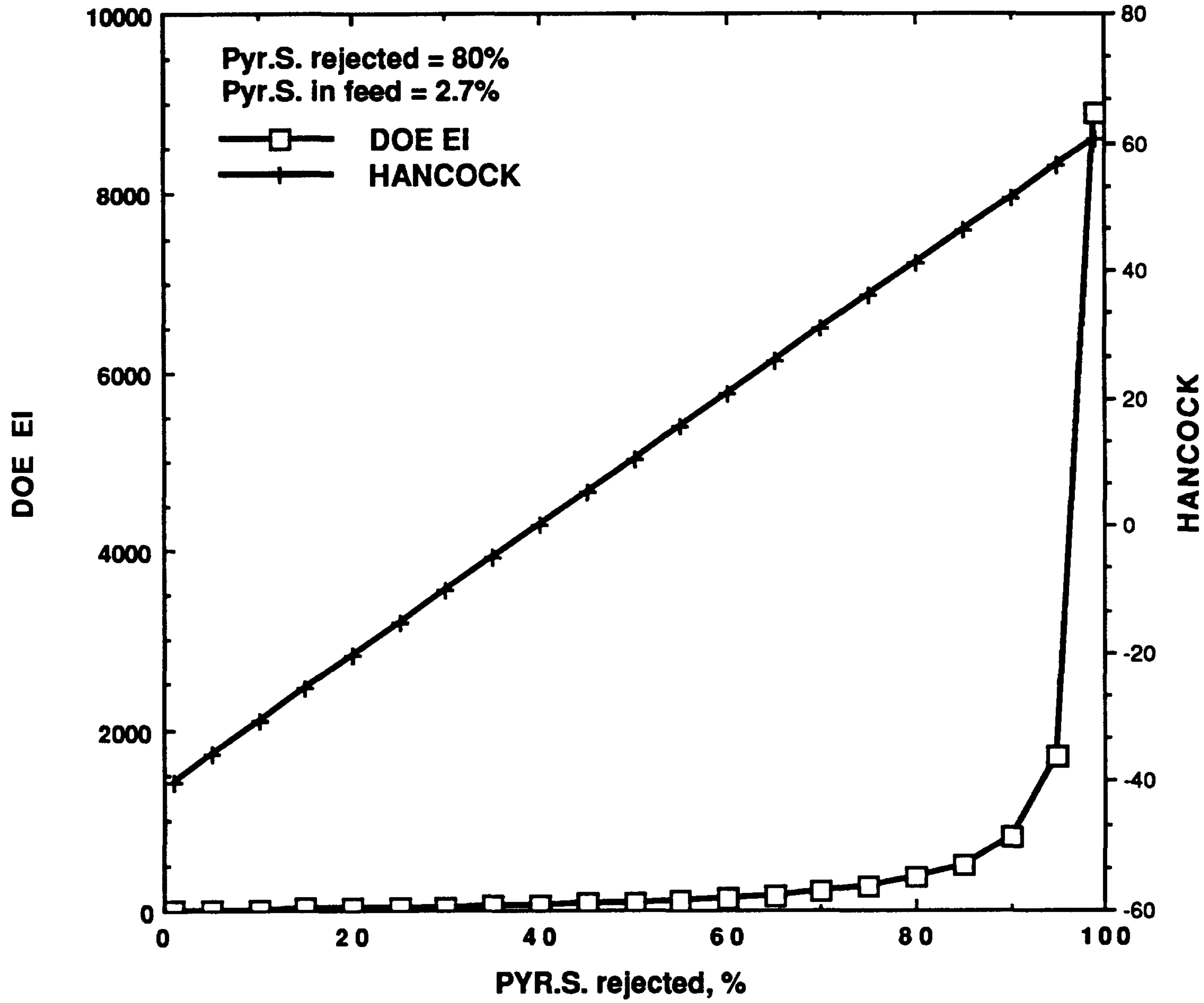

Figure 4.1 Comparison of the Hancock and DOE efficiency indexes as a function of pyritic sulfur rejection at a constant yield of 60 percent.

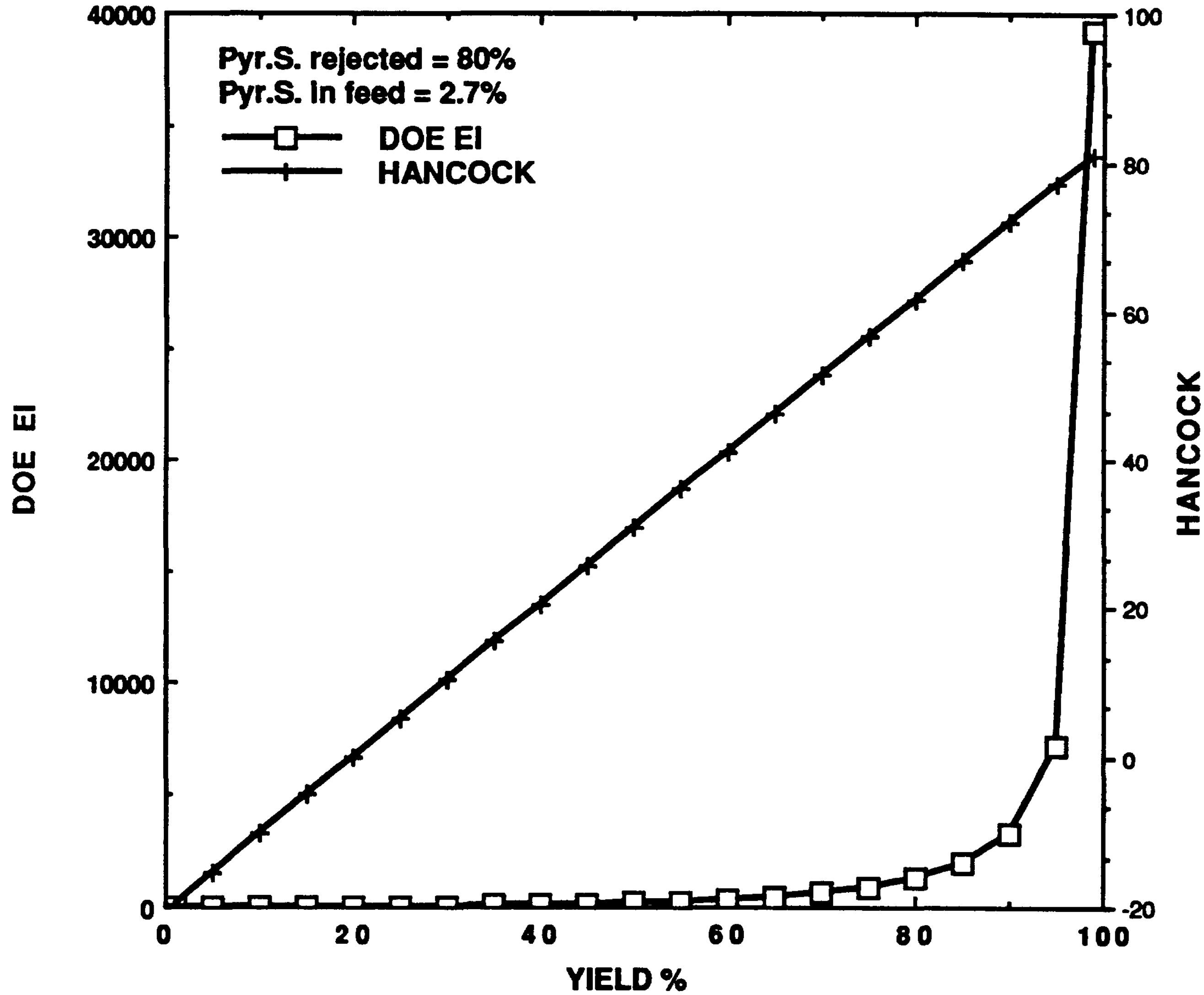

Figure 4.2 Comparison of the Hancock and DOE efficiency indexes as a function of yield at a constant pyritic sulfur rejection of 80 percent.

indicate a perfect separation, that is it cannot take a value of 100 (not all the pyrite is rejected) whereas DOE EI can tend to infinity, even though the separation is not perfect.

Finally, the standard flotation test results give DOE EI = 360. Figure 4.3 shows the locus of points of pyritic sulfur rejected vs. yield that also gives the value DOE EI = 360. It should be noted that for a pyritic sulfur rejection of 100% and a yield of zero, DOE EI is indeterminate and not 360, there is a discontinuity at that point. However, the main point is that in the region of standard test results the value DOE EI = 360 can mean many different things in regards to how much pyritic sulfur is rejected and how much of the combustible matter is recovered. The value of DOE EI has little intrinsic meaning.

A similar criticism of the Hancock efficiency can be made, that is one value of E_H does not correspond to one separation result. However, E_H does correspond to how much material has gone through the ideal separator of Schulz[2].

Schulz proposed defining the efficiency of a separation with the aid of his ideal separator. The ideal separator, as the name suggests, is a notional machine that recovers all desirable material and rejects all undesirable material. Any separation process can be regarded as a system in which some of the material goes through the ideal separator the rest of the material bypasses the machine and is separated randomly between recovery and rejection. The Hancock efficiency is such a measure. For the standard test results, DOE EI = 360 and E_H = 41.1 which could be interpreted as 41.1% of the material passing through the ideal separator. The rest of the material may report to either tailings or concentrate, so even for the Hancock separation efficiency, a single value can be ambiguous.

In summary it would seem then that Hu and Hancock have produced a measure of efficiency that corresponds to some notion commensurate with intuition, whereas the Department of Energy's efficiency is more difficult to interpret. Both methods, though, have their shortcomings

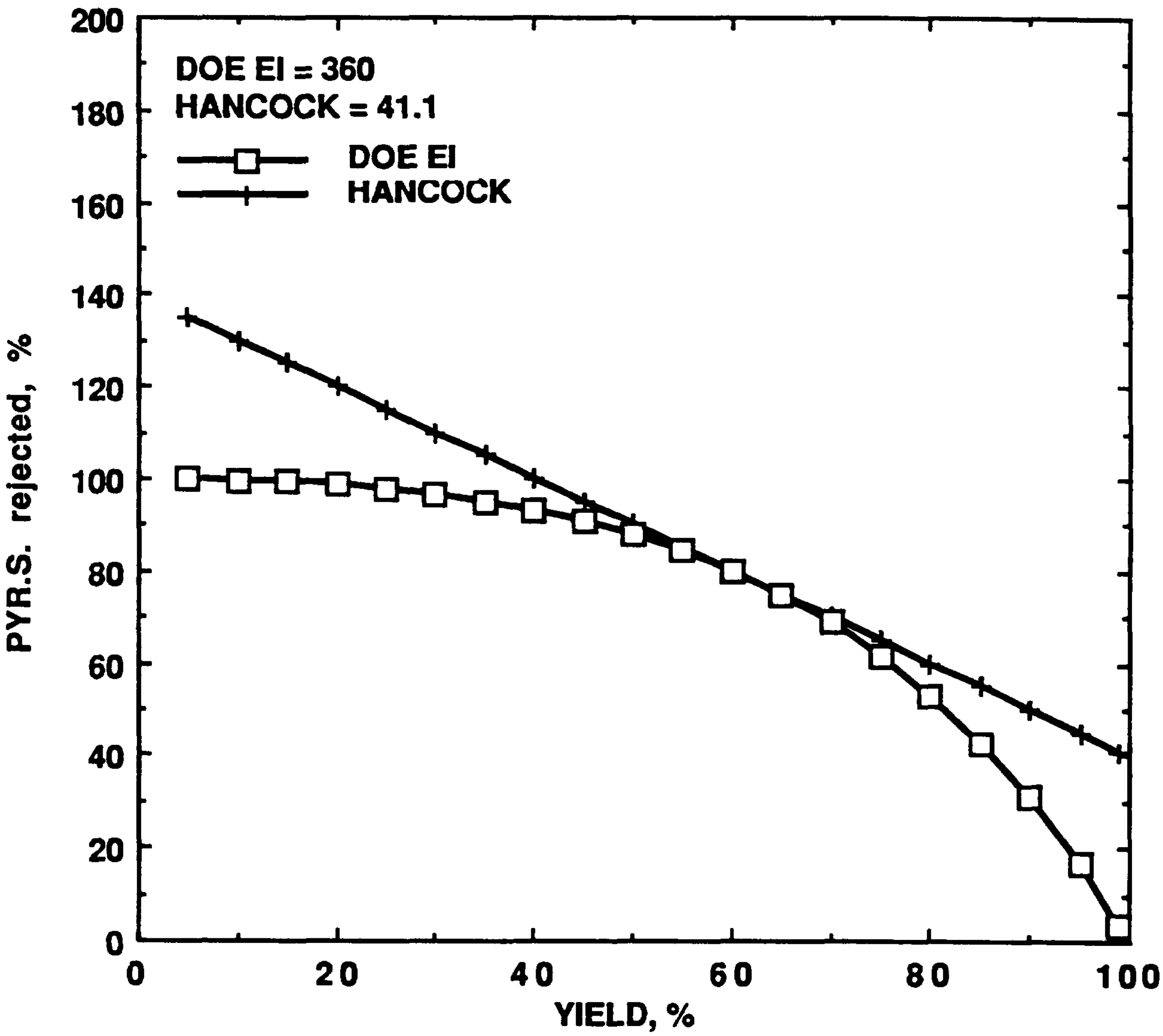

Figure 4.3 Comparison of the pyritic sulfur rejection as a function of yield at constant efficiency for Hancock and DOE indexes.

as a measure of efficiency because one value can correspond to a number of different states. Each different separation scenario is not defined by an efficiency unique to itself, but may share the same efficiency as any number of different separations. Maybe the main conclusion to be drawn is that one number alone cannot properly describe the efficiency of a separation process. At least E_H is positive for favorable separations, negative for unfavorable separations, in each of these regions varies from 0% to 100%, and returns a value of zero if nothing happens. It is intended that the Hancock efficiency will be used for future work on this project. However, the DOE efficiency index will also be used until it is considered that the use of the former is sufficiently representative of the measure of efficiency.

4.3 Results of Standard Flotation Test with Babcock and Wilcox Kerosene

Reagent-grade dodecane is used as the collector in the Standard Flotation Test to evaluate various surface modification treatments and grinding procedures. Dodecane was chosen because it is readily available and the properties of the liquid will not vary from lot to lot. On the other hand, kerosene, which is more commonly used as a collector in coal flotation, is a mixture of numerous hydrocarbons that boil within a specified range and therefore no two kerosene samples are necessarily identical in composition. Because Babcock and Wilcox will be using kerosene in their test work, a sample of their kerosene was obtained and used in place of dodecane in the Standard Flotation Test. The results for these tests along with the standard results obtained with dodecane are shown in Figure 4.4. These results show that the flotation yields obtained with the kerosene were about 5 percent greater than those obtained with dodecane. The products are currently being analyzed for ash and sulfur. These results are also being rechecked with a fresh kerosene sample also supplied by Babcock and Wilcox.

Table 4.4 Comparison between dodecane and kerosene as collectors in the flotation of wet ground 200 mesh coal.

Coal	Collector	Yield,%
Illinois No. 6	Dodecane	74.6
	Dodecane	75.1
	Kerosene	80.3
	Kerosene	80.5
Pittsburgh No. 8	Dodecane	75.9
	Dodecane	77.6
	Kerosene	81.5
	Kerosene	78.4

4.4 Standard Flotation Test with Kaiser's Pittsburgh No. 8 Research Sample

Enough coal was procured in the initial months of this project to supply various research organizations with samples that are identical to those used by the Berkeley and University of Pittsburgh teams. One such sample was supplied by Praxis Engineers to Kaiser Engineers. This sample was crushed by Babcock and Wilcox to 1/4-inch nominal topsize and five pounds sent to the University of California to determine if the sample was indeed identical to the original sample. The results of this test were used to determine if the results obtained by the Berkeley research team with the Pittsburgh No. 8 sample could be used by Kaiser Engineers to help in the engineering development program. The results of the Standard Flotation Test for both of the Pittsburgh No. 8 coal samples are given in Table 4.5. Both samples gave similar results, well within the statistical variation of the Standard Flotation Test. We have therefore concluded that the research sample sent to Kaiser Engineers and crushed by Babcock and Wilcox is identical to the research sample used by the Berkeley research team.

Table 4.5 Flotation results of wet-ground Pittsburgh No. 8 Coal obtained from Babcock and Wilcox (Kaiser Engineers) and the University of California.

		FLOTATION PRODUCT ANALYSIS		
Sample Source	Grind Size	Yield %	Ash %	Tot. S %
Kaiser	200 M	81.8	5.6	2.7
Berkeley		78.2	5.7	2.5
Kaiser	28 M	89.5	7.7	3.3
Berkeley		89.6	5.7	2.5

REFERENCES

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2. Schulz, N.F., "Separation Efficiency" Transactions, Society of Mining Engineers, AIME, Vol. 247, pp. 81-87 (1970).

5.0 GRINDING AND FLOTATION STUDIES

The overall objective of this part of the research program is to determine the effect of fine grinding under various environments on the surface properties and flotation characteristics of coal and its associated mineral matter, particularly pyrite. Work performed on the grinding and flotation of coal during this past quarter involved experimentation to establish the standard grinding test conditions, to determine the relative grindabilities of the three base coals, to investigate the effect of grinding environment and of gas bubble composition on their flotation yield, and to compare the effect of adding the standard dosage of collector (dodecane) to the mill before grinding or to the cell after grinding on the flotation kinetics of these coals. The effect of varying collector dosages added to the mill before grinding on the kinetics of coal flotation was also investigated. When the amount of collector added to the mill was smaller than the standard dosage, flotation tests were also carried out with coal suspensions that were conditioned adding to the cell the extent of collector required to bring its total up to the standard level.

5.1 Standard Grinding Test Conditions

During this reporting period, the standard grind times established previously were checked periodically to keep the percent passing consistently close to the 95% requirement. Minor adjustments were made on the grind times necessary for achieving a product 95% finer than 200 mesh in the case of Pittsburgh No. 8 coal and 95% finer than 28 mesh in the case of Upper Freeport PA coals ground under both dry and wet conditions. The new times to achieve these requirements were found to be 30 min and 16.5 min for 200 mesh Pittsburgh No. 8 ground dry and wet respectively, and 2 min 50 sec and 1 min 45 sec for 28 mesh Upper Freeport PA ground dry and wet, respectively. These revised standard grinding times are given in Table 5.1.

Table 5.1 Standard grinding conditions

PARAMETER	28 MESH GRIND	200 MESH GRIND
Grinding Equipment and Operating Conditions		
Rod mill size	8-in dia. x 10-in length, stainless steel construction	
Mill speed (60% critical)	56 rpm	56 rpm
Coal feed (dry basis)	500 g	500 g
Feed size	minus 1/4-inch crushed coal	
Target grind dry and wet	95% minus 28 M	95% minus 200 M
Wet grind H_20 for 500 g coal	700 ml	700 ml
Coal Specific Conditions		
No. of rods	24	42
Rod mix 3/4", 5/8", 1/2", resp.	9, 8, 7	16, 14, 12
Grind Times		
Illinois No. 6		
Dry	8 min.	48 min.
Wet	3 min. 20 sec.	15 min.
Pittsburgh No. 8		
Dry	4 min. 50 sec.	30 min.
Wet	3 min.	16 min. 30 sec.
Upper Freeport PA		
Dry	2 min. 50 sec.	21 min. 30 sec.
Wet	1 min. 45 sec.	11 min. 20 sec.

5.2 Relative Grindabilities of Base Coal Samples

Taking as a reference the dry grinding of Illinois No. 6 coal to obtain a product 95% finer than 28 mesh or 200 mesh, the relative grindabilities (in mass/unit energy) of the three base coals were calculated, and they are tabulated in Table 5.2. In all cases, Upper Freeport PA is the easiest to grind. Illinois No. 6 tends to require the most energy to achieve a given size reduction,

Table 5.2 Relative grindabilities* of the three base coals.

DRY GRIND	28 MESH	200 MESH
Illinois No. 6	1.0	1.0
Pittsburgh No. 8	1.7	1.6
Upper Freeport PA	2.8	2.7
WET GRIND	28 MESH	200 MESH
Illinois No. 6	2.4	3.2
Pittsburgh No. 8	2.7	2.9
Upper Freeport PA	4.6	4.2

*Grindability in mass/unit energy relative to dry grinding Illinois No. 6 coal to 95 ± 1% minus 28 mesh or minus 200 mesh.

either by dry or wet grinding. However, the wet 200 mesh grind for Pittsburgh No. 8 appears to require more energy than Illinois No. 6 at the same grind size.

5.3 Grinding and Flotation Under Different Environments

The objective of this study is to determine the effect of grinding environment and bubble gas composition on the flotation behavior of 200 mesh Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coal samples. These samples were dry and wet ground in the rod mill under either air or inert (argon) environments maintaining all other grinding conditions the same as those specified for the Standard Grinding Test. The ground samples were then floated with either air or argon bubbles. The standard dosages of collector (dodecane) and frother (MIBC) were added directly to the flotation cell. Flotation yields were obtained following the standard flotation test conditions.

Flotation yields obtained at Berkeley, Columbia and Utah with the three base coals dry and wet-ground under open or inert atmospheres and floated with air or argon bubbles are tabulated in Table 5.3. The results indicate that for all the combinations of grinding environment and bubble composition, wet grinding gives higher flotation yields than dry grinding for both Illinois No. 6 and Upper Freeport PA coals. In the case of Pittsburgh No. 8 coal, however, wet grinding appears to slightly lower the flotation yield.

The reduced yields of dry-ground Illinois No. 6 and Upper Freeport PA coals may be due to oxidation of the surface as a result of thermal effects during comminution. The reason for the absence of such an effect in the case of Pittsburgh No. 8 coal is not clear at present.

Bubble composition (air or argon) does not seem to affect the flotation yields of any of the three coals for both dry and wet grinding under a given environment (air or argon). In the case of Illinois No. 6 coal, wet grinding under argon marginally improves the flotation yield. However, the flotation yields of Pittsburgh No. 8 and Upper Freeport PA coals are not affected by the wet grinding environments. Dry grinding environment show no discernible trends on flotation yields for the three coals.

Table 5.3 The effect of grinding atmosphere and flotation gas composition on the yields of 200 mesh feed.

		FLOTATION YIELD, PERCENT			
	Grinding Method	AG/AF	AG/IF	IG/AF	IG/IF
Illinois No. 6	Dry	60.4	56.5	--	54.5
	Wet	75.3	78.3	81.3	82.6
Pittsburgh No. 8	Dry	86.9	85.9	77.8	80.8
	Wet	75.3	74.8	74.8	75.5
Upper Freeport PA	Dry	63.5	65.4	67.9	67.5
	Wet	75.0	75.9	75.4	74.9

AG/AF = air grind, air float
AG/IF = air grind, inert float

5.4 Grinding with Collector and Flotation Kinetics

The objective of this part of the investigation is to compare the effect of adding the standard dosage of collector (dodecane) to the rod mill before wet grinding on the flotation kinetics of the three base coals. The rationale of adding the collector to the mill before grinding is to ensure its adsorption as soon as the new surface is generated during the comminution of the coarse particles. Since the surface of coal is susceptible to oxidation, the addition of dodecane to the mill may also help minimize its oxidation.

The coal feed for flotation was wet-ground in air using the standard grinding procedure to obtain a product 95% finer than 200 mesh. Grinding was carried out under wet conditions to ensure that the collector would be well dispersed and thereby adsorb more uniformly on the coal surface. With the exception of the total flotation time, all other standard flotation test conditions were held constant when floating the coal samples ground without collector. In the case of the coal samples ground with collector, these flotation conditions were also held constant but the conditioning period with dodecane was carried out without adding it to the flotation cell. In both cases, concentrates were collected at 0.5, 1.0, 2.0, 4.0 and 5.0 minutes of flotation, dried, and weighed separately. From these results, assuming flotation follows first-order behavior, plots of $ln(1 - \text{yield})$ versus flotation time were constructed, and they are presented as Figures 5.1 to 5.3, to show the flotation kinetic behavior of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals, respectively, for conditions corresponding to addition of the standard dosage of collector to the cell after wet grinding and to the mill before grinding. These kinetic plots indicate that the addition of the standard dosage of collector to the mill before grinding improves the flotation yield of Illinois No. 6 coal, but it appears to have a detrimental effect on the flotation kinetics of both Pittsburgh No. 8 and Upper Freeport PA coals.

Because of the relatively low standard dosage of collector for Pittsburgh No. 8 and Upper Freeport PA coals, which is about 33% and 8% of that for Illinois No. 6 coal, respectively, dodecane may be consumed before the new surfaces are created during grinding. Adding the standard dosage of collector to the cell produces a relatively more uniform distribution of dodecane on the surface of all the coal particles, resulting in faster flotation under standard conditions. In the case of Illinois No. 6 coal the amount of dodecane added to the mill before grinding may cause aggregation/agglomeration of the coal particles, thereby enhancing its flotation kinetics.

Figures 5.1 to 5.3 also indicate that, irrespective of the point of addition of the standard dosage of collector, the kinetics of flotation of the three base coals under investigation follows a first order reaction at short flotation times. The two-step flotation curves indicate the presence of a fast-floating fraction and a slow-floating fraction. The fast-floating fraction probably comprised of coarse particles or flocs, whereas the finer particles constitute the slow-floating fraction. Because of this flotation behavior of the three base coals, it was then decided to investigate the effect of collector dosage added to the mill on the initial flotation kinetics. The results of this study are discussed in the section below.

5.5 Flotation Kinetics of Coal Ground with Varying Collector Dosages

The objective of this study is to delineate the effect of collector dosage added to the mill on the initial flotation kinetics of the three base coals. For these flotation tests, the material was ground with varying collector dosages added to the mill before grinding and floated with air following the procedure described before. Concentrates were collected at 0.5 and 1.0 minutes of flotation, dried and weighed separately. From these results, the flotation yields were calculated and they were used to construct the kinetic plots presented as Figures 5.4 to 5.6, where the

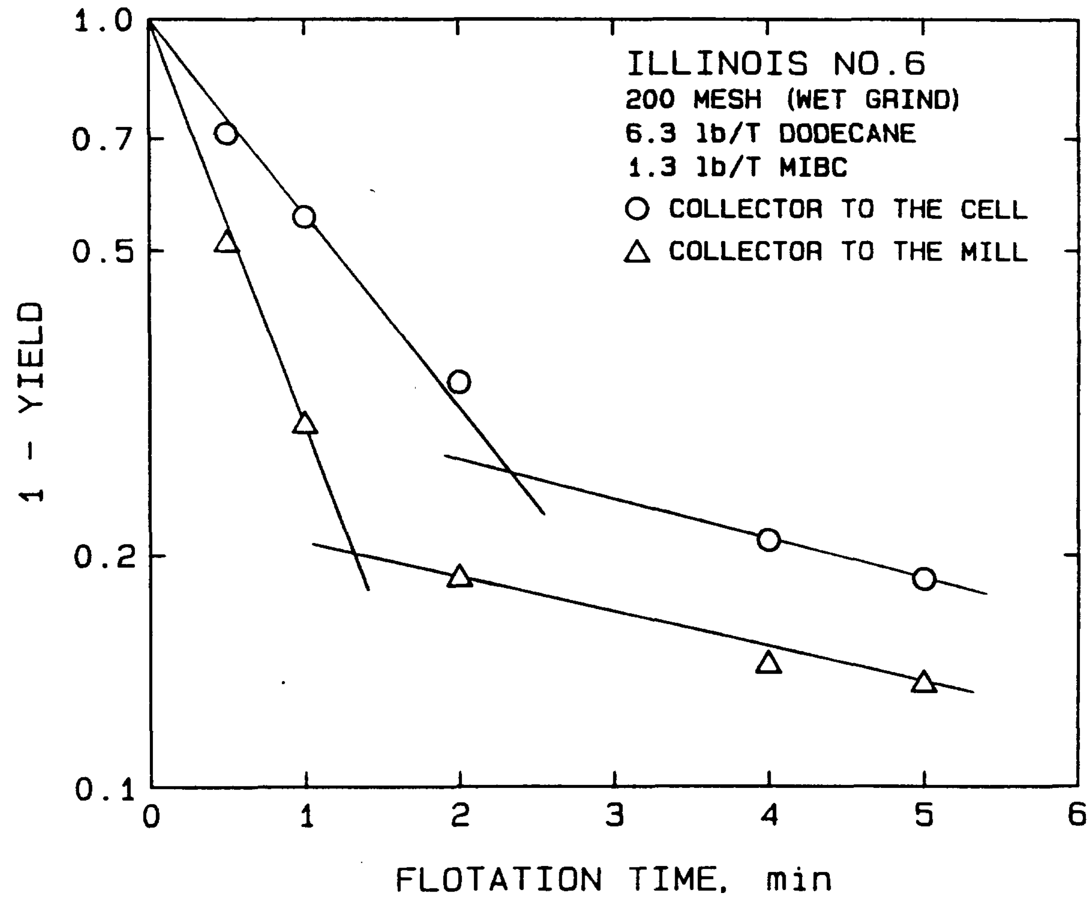

Figure 5.1 Flotation kinetics of Illinois No. 6 coal wet ground to 200 mesh with the standard collector dosage added to the rod mill or flotation cell.

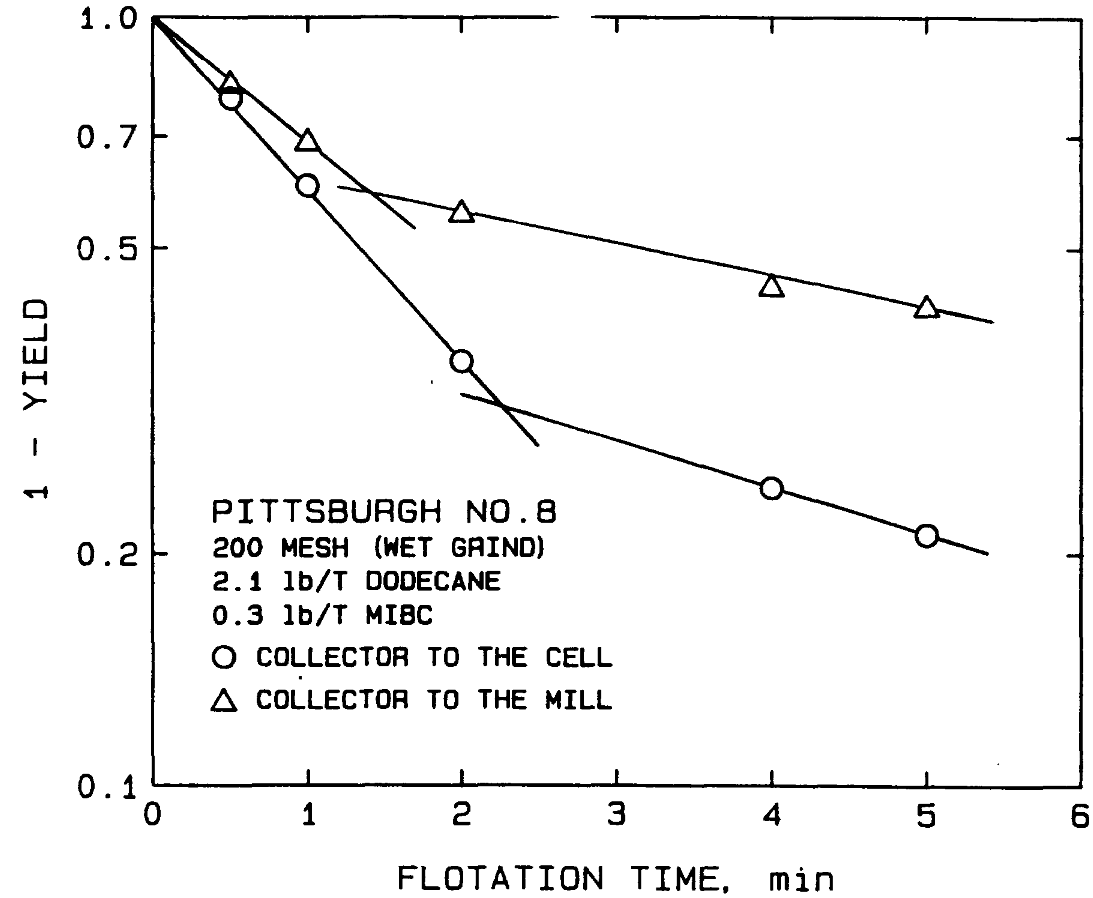

Figure 5.2 Flotation kinetics of Pittsburgh No. 8 coal wet ground to 200 mesh with the standard collector dosage added to the rod mill or flotation cell.

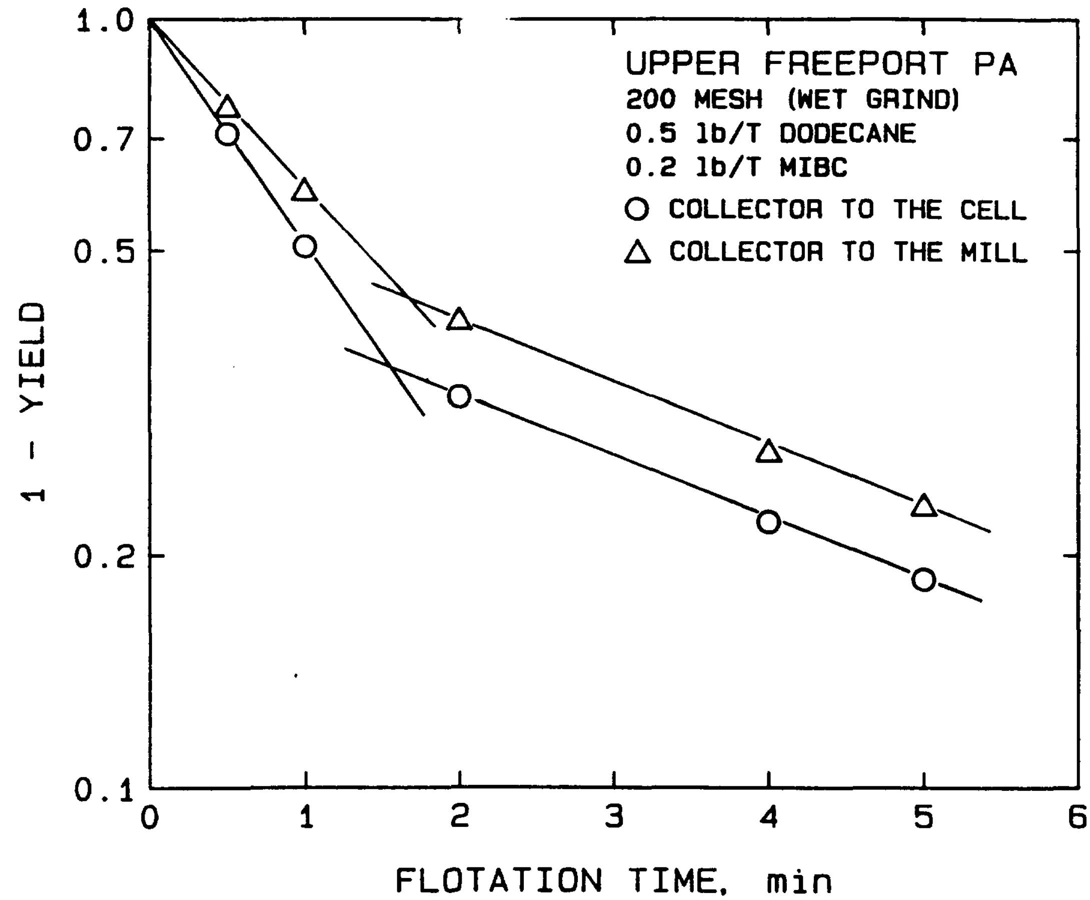

Figure 5.3 Flotation kinetics of Upper Freeport PA coal wet ground to 200 mesh with the standard collector dosage added to the rod mill or flotation cell.

$ln(1 - \text{yield})$ is plotted versus flotation time for different dosages of collector added to the mill before grinding. It can be seen from these plots that increasing the dosage of collector added to the mill increases the rate of flotation of all three base coals.

The straight lines on these plots follow the equation

$$ln(1 - \text{yield}) = - k t$$

where t is the flotation time and k is the flotation rate constant, which corresponds to the slope of these lines. This slope was computed and the values for different dosages of dodecane added to the mill are plotted in Figure 5.7, which shows that the initial flotation rate constant (for 1.0 min flotation time) for all the three base coals increases with increasing collector amounts added to the mill. This figure also indicates that at collector dosages greater than 9 lb/T, these coals seem to exhibit similar flotation kinetics. Aggregation/agglomeration of the coal particles may be taking place under these conditions.

5.6 QA/QC Grinding Tests

Coal feed samples for flotation studies are comminuted using a rod mill following the standard grinding test procedures to obtain a fraction 95 ± 1% passing 28 mesh or 200 mesh. To fulfill the QA/QC plan, during this past quarter a comparison of grinding data obtained at Berkeley, Columbia and Utah was made to check the percent of material passing these two grinds after comminuting the three base coals at these three different sites for the standard grinding times. These QA/QC Standard Grinding Test results are presented in Tables 5.4 to 5.6.

Table 5.4 Comparison of results of QA/QC tests of the standard grinding procedure for Illinois No. 6 coal.

Site	Grind	Grinding Method	Grinding Time	Percent Passing
Berkeley	28 M	Dry	8 min.	95.8
Columbia	28 M	Dry	8 min.	--
Utah	28 M	Dry	8 min.	--
Berkeley	28 M	Wet	3 min. 20 sec.	95.0
Columbia	28 M	Wet	3 min. 20 sec.	--
Utah	28 M	Wet	3 min. 20 sec.	--
Berkeley	200 M	Dry	48 min.	95.9
Columbia	200 M	Dry	48 min.	--
Utah	200 M	Dry	48 min.	--
Berkeley	200 M	Wet	15 min.	95.5
Columbia	200 M	Wet	15 min.	93.6
Utah	200 M	Wet	15 min.	--

Table 5.5 Comparison of results of QA/QC tests of the standard grinding procedure for Pittsburgh No. 8 coal.

Site	Grind	Grinding Method	Grinding Time	Percent Passing
Berkeley	28 M	Dry	4 min. 50 sec.	95.8
Columbia	8 M	Dry	4 min. 50 sec.	95.0
Utah	28 M	Dry	4 min. 50 sec.	--
Berkeley	28 M	Wet	3 min.	96.1
Columbia	28 M	Wet	3 min.	96.0
Utah	28 M	Wet	3 min.	--
Berkeley	200 M	Dry	31 min.	96.0
Columbia	200 M	Dry	31 min.	94.0
Utah	200 M	Dry	31 min.	--
Berkeley	200 M	Wet	18 min.	97.5
Columbia	200 M	Wet	18 min.	96.0
Utah	200 M	Wet	18 min.	--

Table 5.6 Comparison of results of QA/QC tests of the standard grinding procedure for Upper Freeport PA coal.

Site	Grind	Grinding Method	Grinding Time	Percent Passing
Berkeley	28 M	Dry	3 min. 15 sec.	97.3
Columbia	28 M	Dry	3 min. 15 sec.	96.7
Utah	28 M	Dry	3 min. 15 sec.	95.0
Berkeley	28 M	Wet	1 min. 55 sec.	97.7
Columbia	28 M	Wet	1 min. 55 sec.	95.4
Utah	28 M	Wet	1 min. 55 sec.	95.0
Berkeley	200 M	Dry	21 min. 30 sec.	92.9
Columbia	200 M	Dry	21 min. 30 sec.	92.8
Utah	200 M	Dry	21 min. 30 sec.	95.0
Berkeley	200 M	Wet	11 min. 20 sec.	95.5
Columbia	200 M	Wet	11 min. 20 sec.	92.0
Utah	200 M	Wet	11 min. 20 sec.	94.9

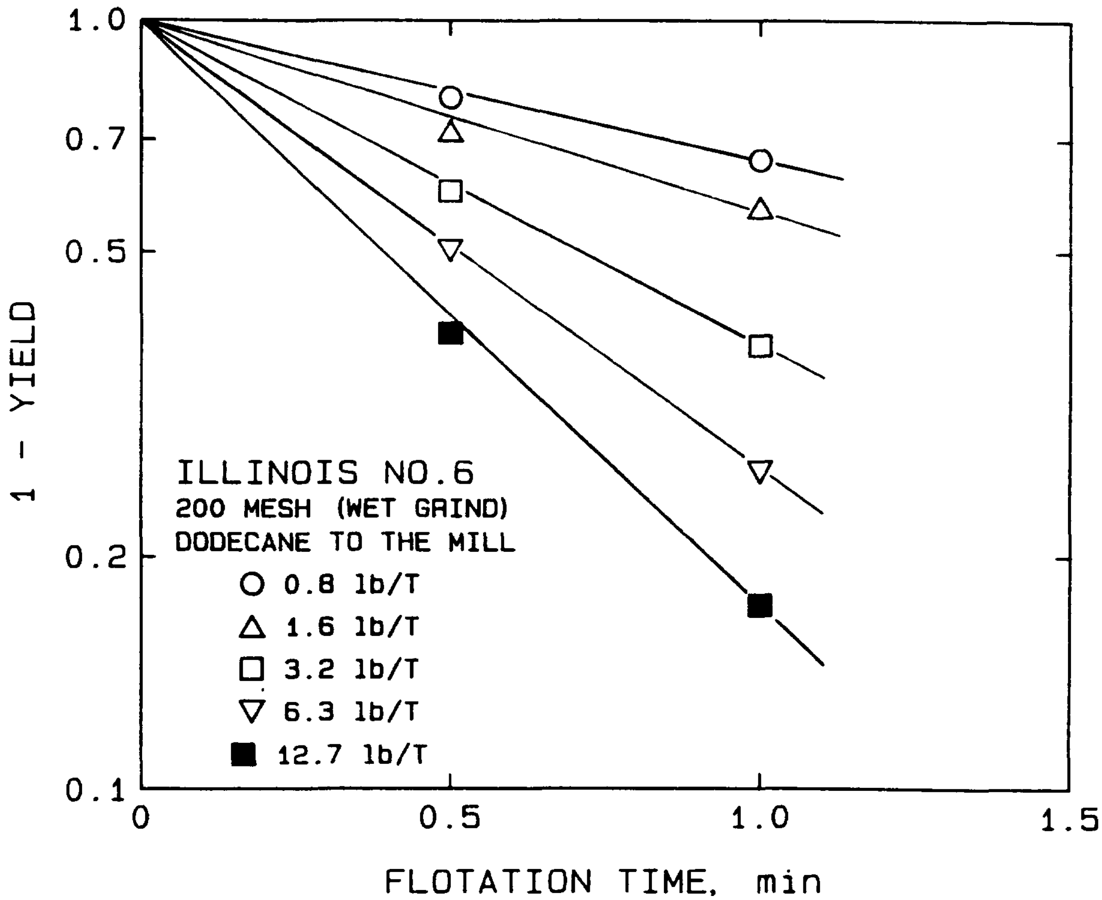

Figure 5.4 Initial flotation kinetics of Illinois No. 6 Coal with different dodecane additions to the rod mill.

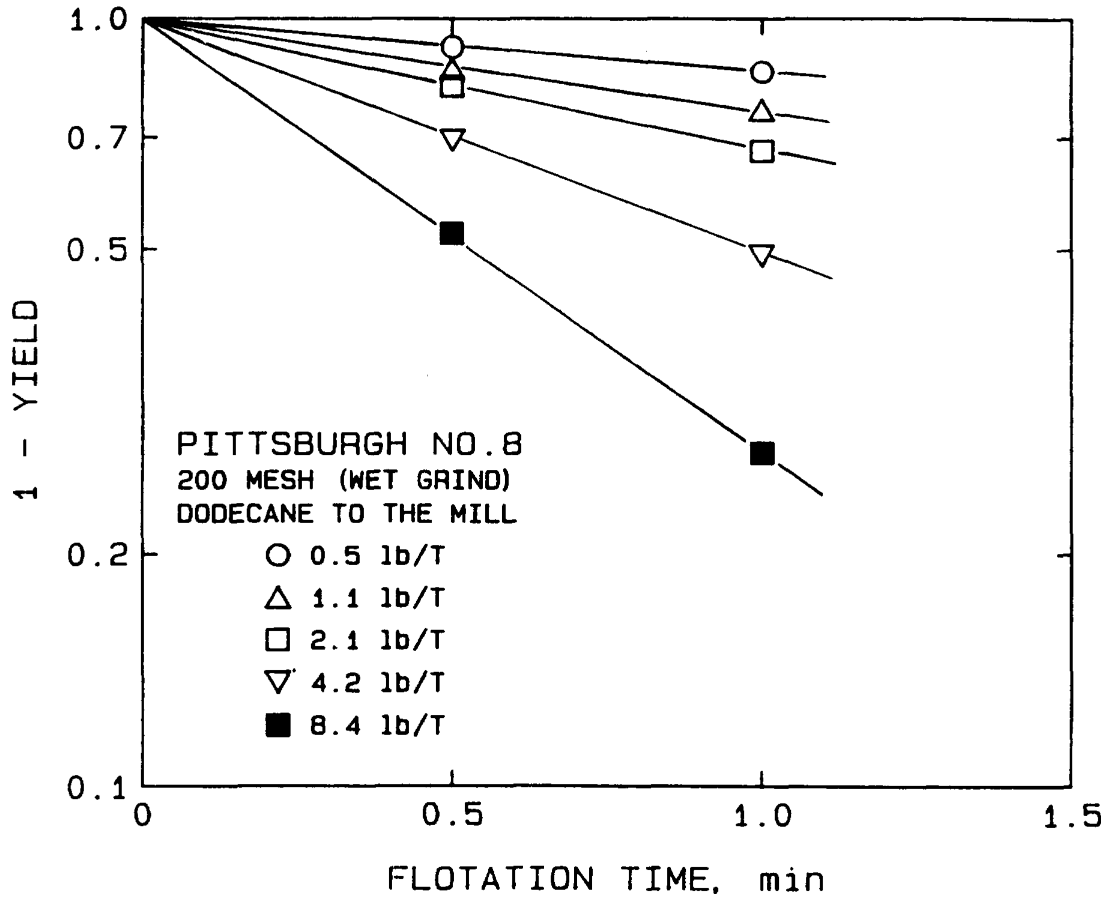

Figure 5.5 Initial flotation kinetics of Pittsburgh No. 8 Coal with different dodecane additions to the rod mill.

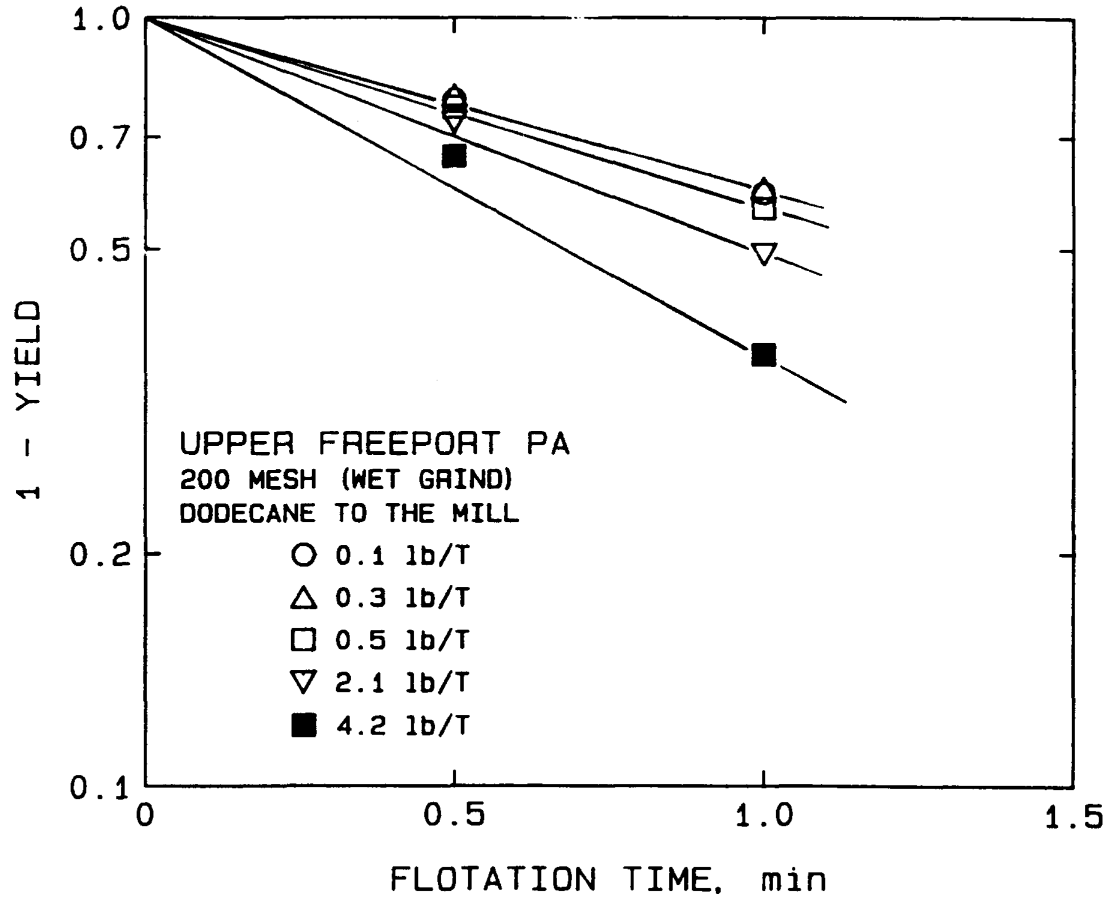

Figure 5.6 Initial flotation kinetics of Upper Freeport PA coal with different dodecane additions to the rod mill.

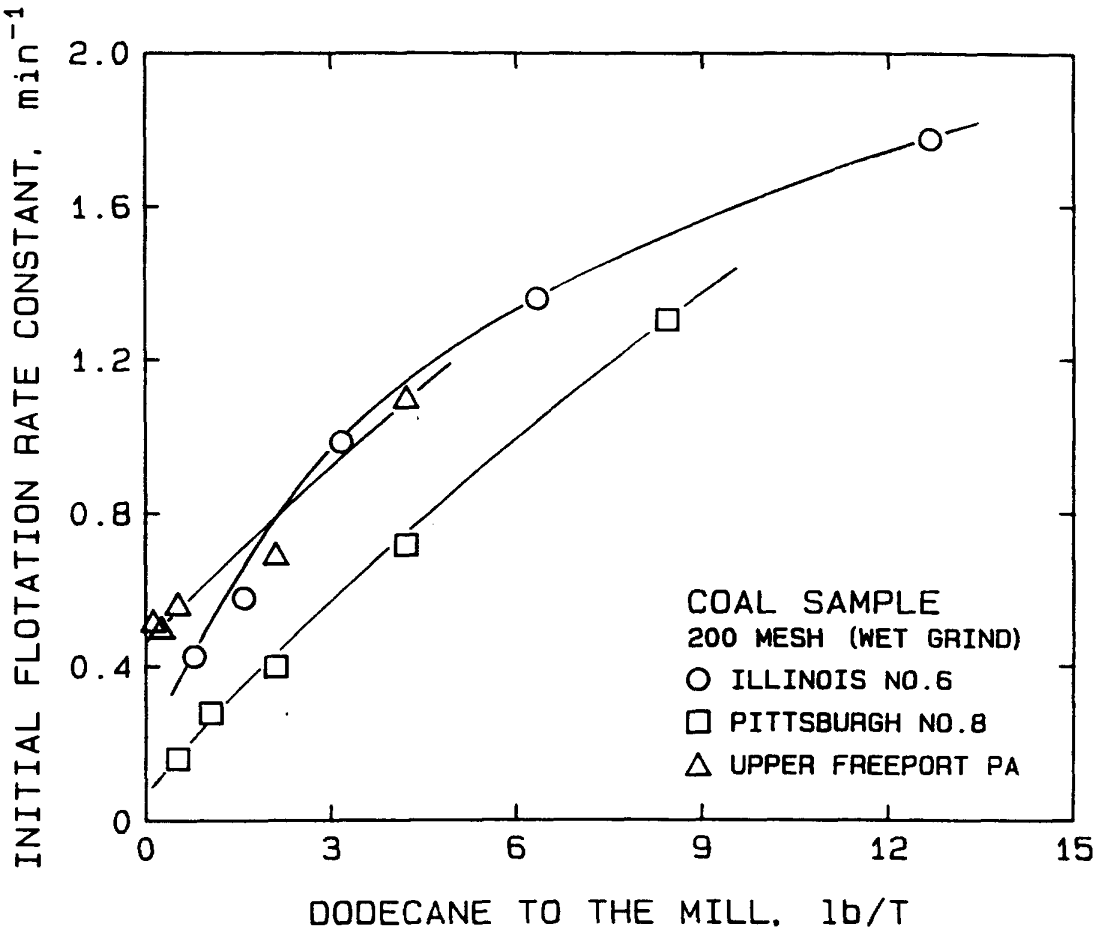

Figure 5.7 Initial flotation rate constants for the three base coals as a function of the dodecane addition to the rod mill.

6.0 EFFECT OF pH AND SURFACE MODIFIER ADDITION ON FLOTATION PERFORMANCE

The efficiency of separation of coal from pyrite by flotation can be enhanced by appropriately modifying the surface of either of the two components so that the relative hydrophobicity of coal with respect to that of pyrite is increased. This should be achieved by either making the surface of coal more hydrophobic or by decreasing the hydrophobicity of pyrite. Evaluation of the effect of pH and various surface-modifying reagents to achieve the above objectives have been undertaken during the past quarter. Flotation performance was evaluated in terms of the efficiency index, as defined in Section 4.

6.1 Effect Of pH Using Lime

It is well known that lime acts as a pH modifier and it is often used to depress the flotation of pyrite in many industrial operations, although the mechanism of this phenomenon is not well established. Therefore, lime was used as the pH modifier in our study to evaluate the effect of pH on the flotation performance of Pittsburgh No. 8 and Upper Freeport coals. The pH was adjusted over the range of 3.5 to 10, and the same collector (n-dodecane) and frother (MIBC) additions as in Standard Flotation Tests were used.

6.1.1 Pittsburgh No. 8 Coal

The effect of pH on the efficiency of pyritic sulfur removal from coal was evaluated for 28 mesh and 200 mesh grinds, using both the dry and wet grinding procedures. Results obtained were analyzed in terms of flotation yield, pyrite rejection efficiency and the Hancock efficiency index. Figure 6.1 shows the effect of pH on the flotation performance of 28 mesh dry ground Pittsburgh No. 8 coal. It can be seen from this figure that the flotation yield remains constant

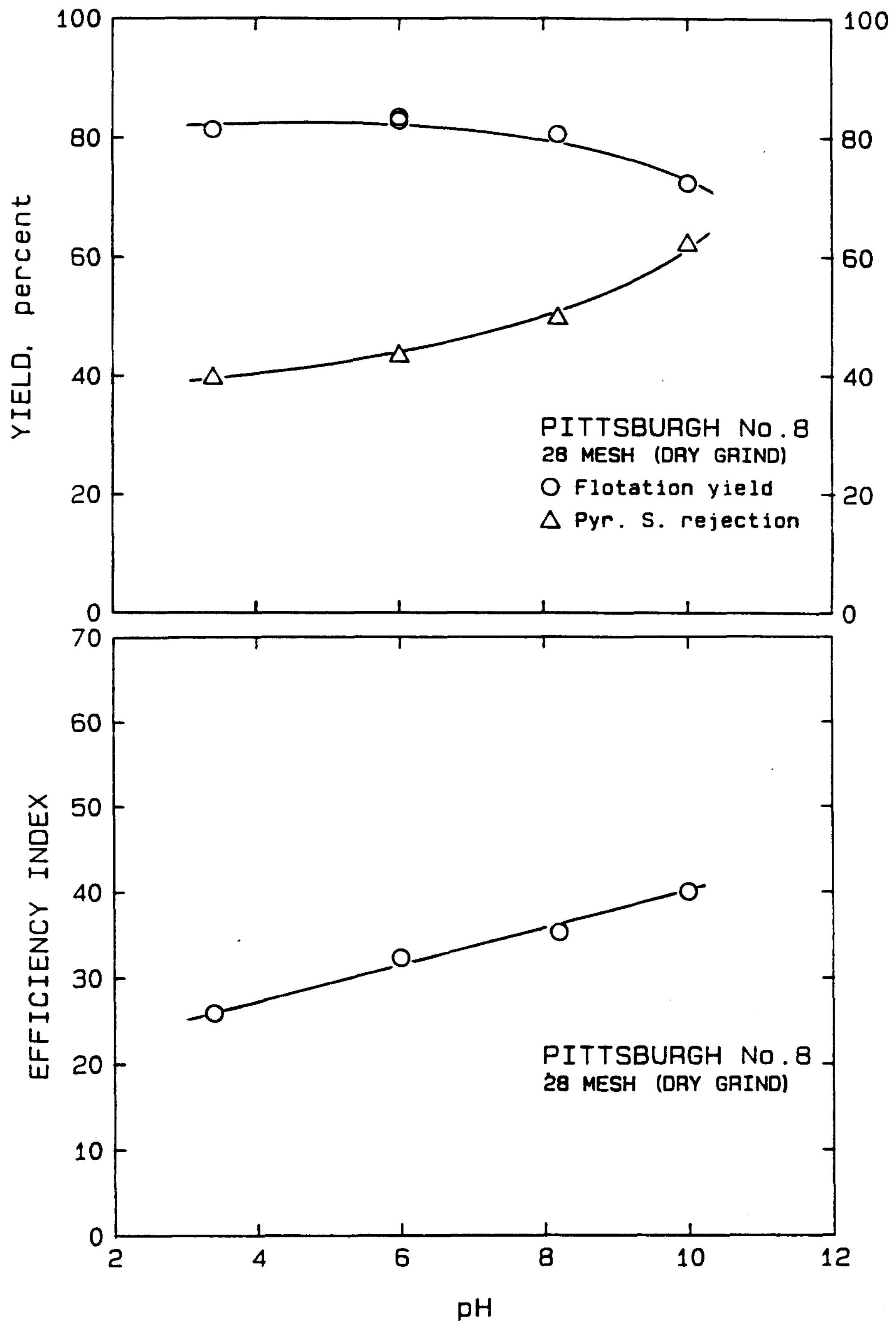

Figure 6.1 The effect of pH on the flotation performance of 28 mesh dry-ground Pittsburgh No. 8 coal.

in the pH range between 3.5 to 8 beyond which there was a sharp drop (at pH 10). The pyrite rejection efficiency, on the other hand, increases steadily with pH in the entire range studied, possibly due to the pyrite-depressant effect of lime rather than just a simple pH effect. The efficiency index, also plotted in Figure 6.1, exhibits a direct linear relationship with pH. In the case of the 28 mesh wet ground material, both the flotation yield and the pyrite rejection efficiency, and consequently the selectivity index, remain constant in the pH range 3.5 to 8 (Figure 6.2). At pH 10 there is a marginal decrease in the flotation yield which, however, is compensated by about an equal increase in the pyrite rejection efficiency. Therefore, the selectivity index is maintained at a level similar to that at lower pH values.

The effect of pH was more pronounced in the case of 200 mesh dry grind (Figure 6.3) compared to the 28 M dry grind. While flotation yield decreased steadily with pH, the pyrite rejection efficiency increased continuously, evidently due to the specific effect of lime. The efficiency index, on the other hand, remained constant in the pH range 4.5 to 8 and dropped sharply at pH 10. This is due to a steeper decrease in flotation yield compared to the increase in pyrite rejection efficiency when the pH was changed from 8 to 10. The flotation yield of wet-ground 200 mesh No. 8 coal, as in the case of dry-ground 200 mesh material, decreased after pH 8. (Figure 6.4). However, the pyrite rejection efficiency appears to pass through a maximum at pH 6. The efficiency index response was similar to that of 200 mesh dry grind.

6.1.2 Upper Freeport Coal

The effect of pH on the efficiency of pyrite removal from Upper Freeport coal was studied with wet-ground 200 mesh samples. The flotation response of this coal, shown in Figure 6.5, was sharply different from that of Pittsburgh No. 8 coal. The results obtained showed that the flotation yield increases slightly up to pH 6 beyond which it tends to drop. The pyritic sulfur

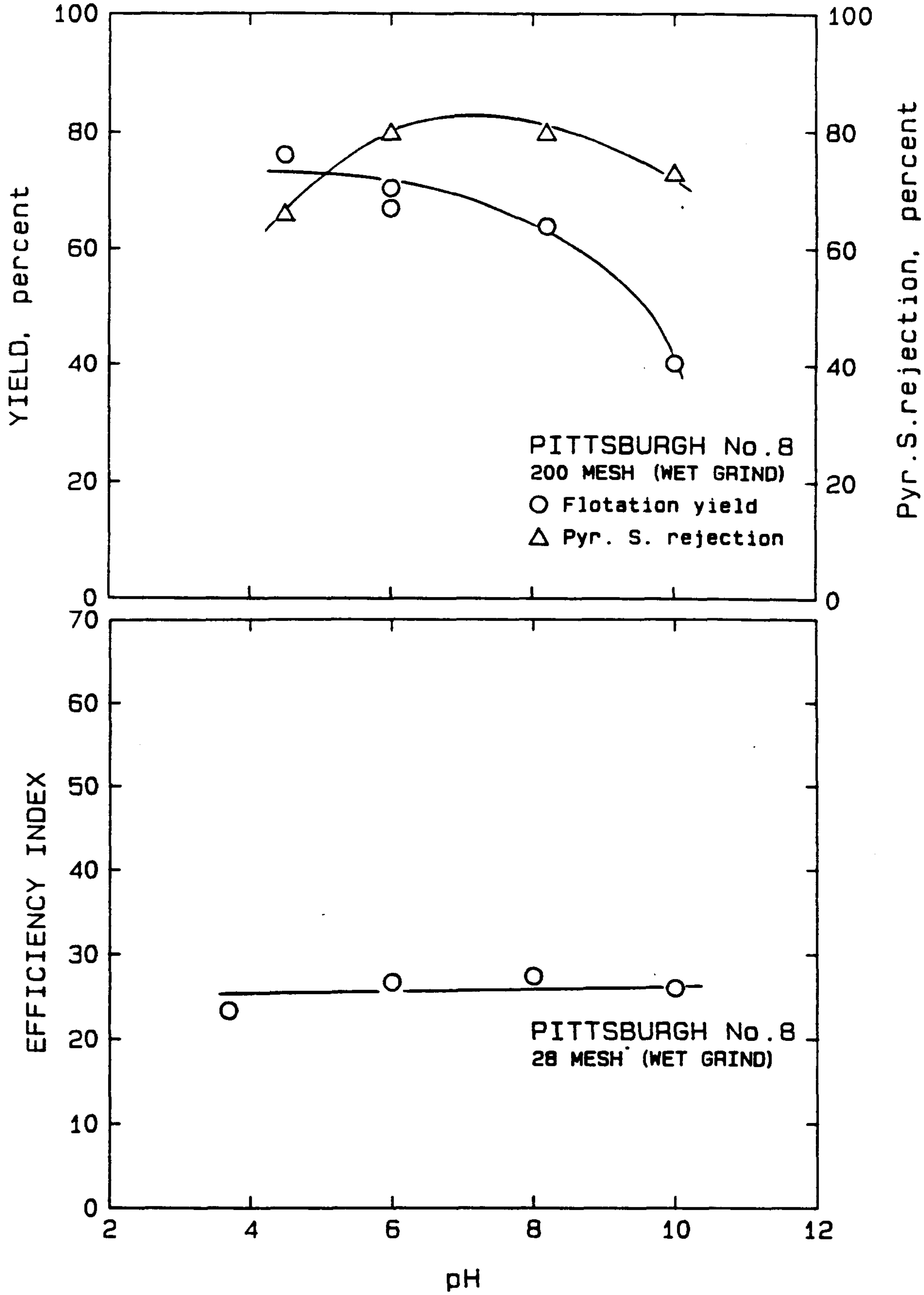

Figure 6.2 The effect of pH on the flotation performance of 28 mesh wet-ground Pittsburgh No. 8 coal.

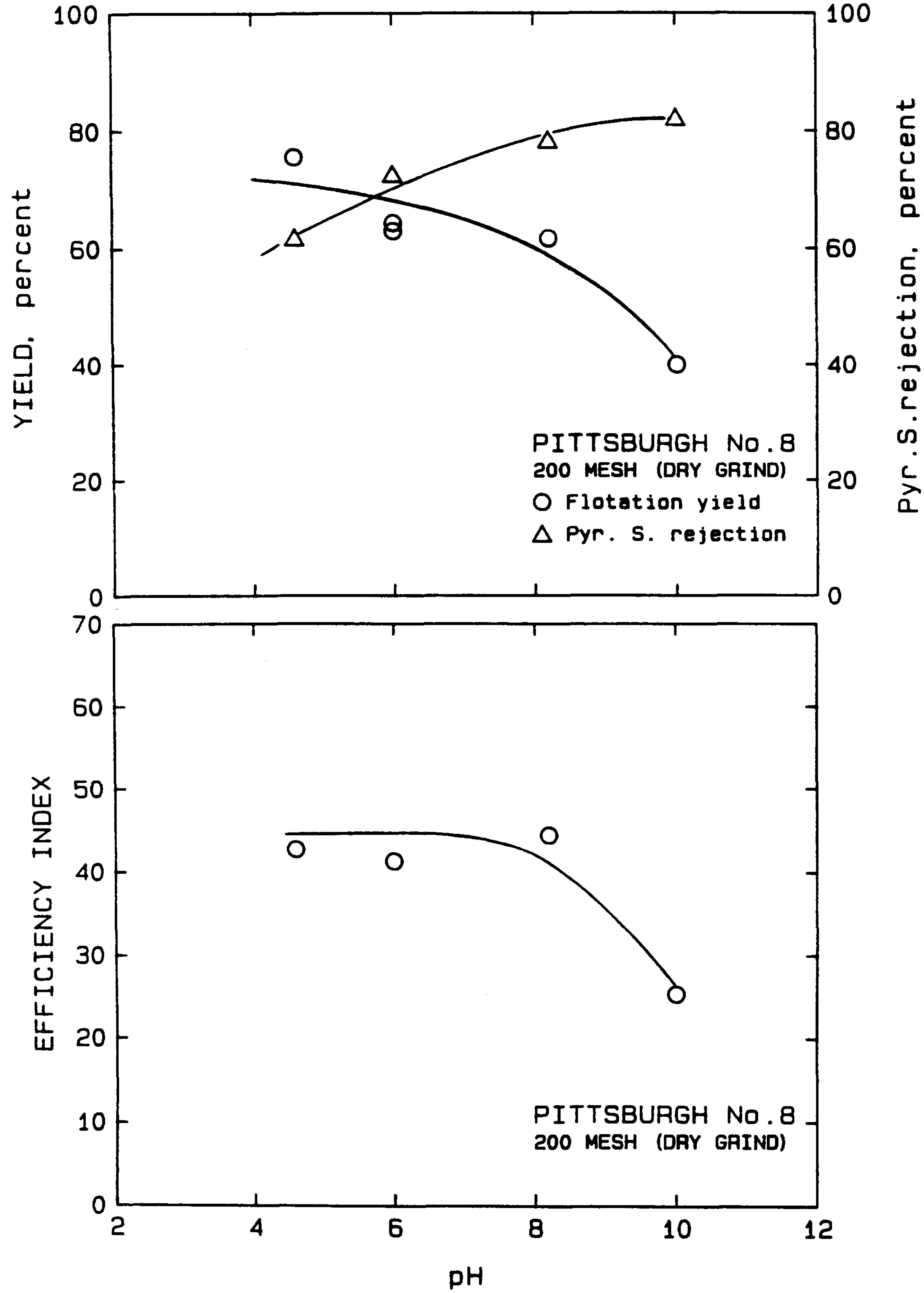

Figure 6.3 The effect of pH on the flotation performance of 200 mesh dry-ground Pittsburgh No. 8 coal.

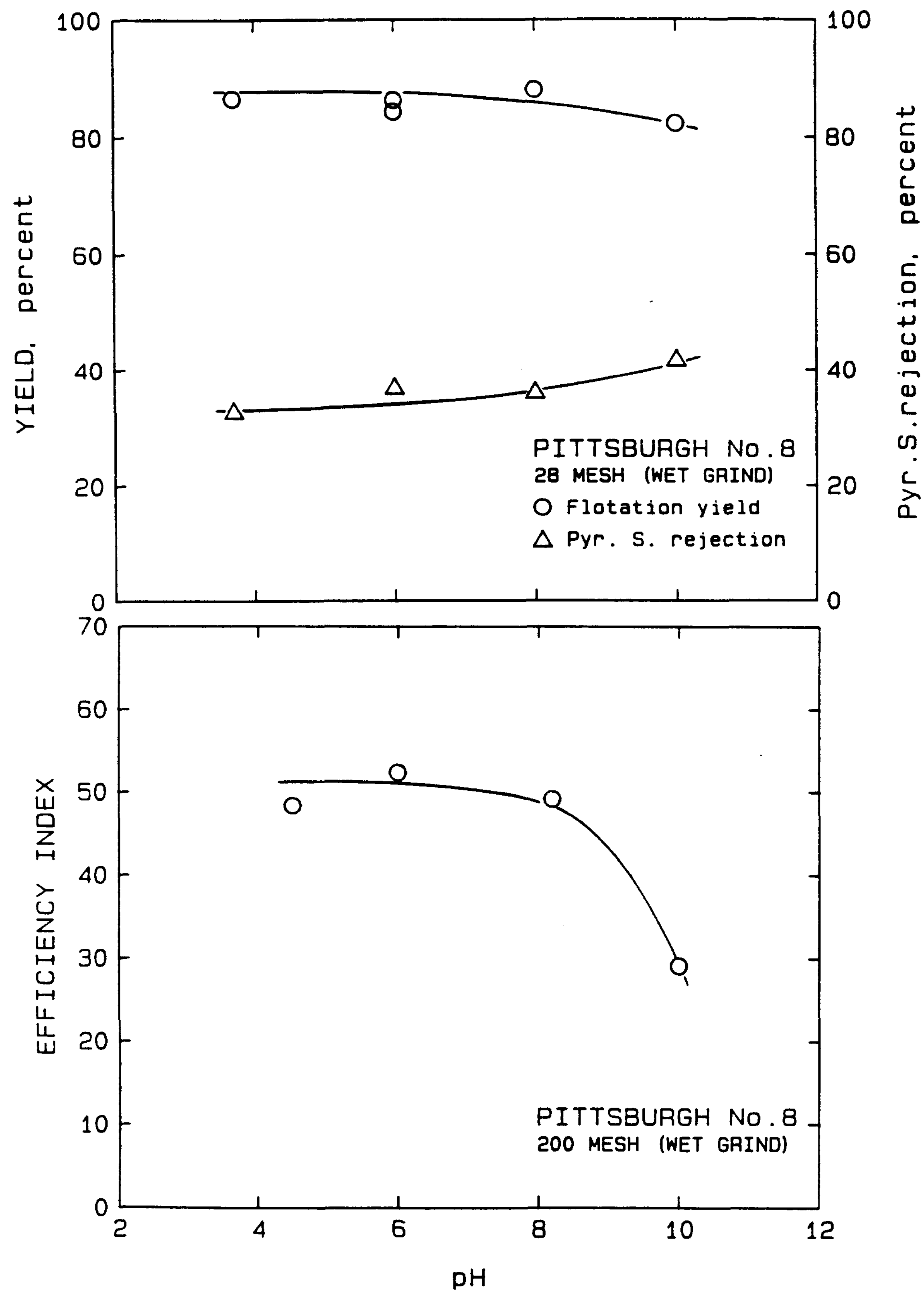

Figure 6.4 The effect of pH on the flotation performance of 200 mesh wet-ground Pittsburgh No. 8 coal.

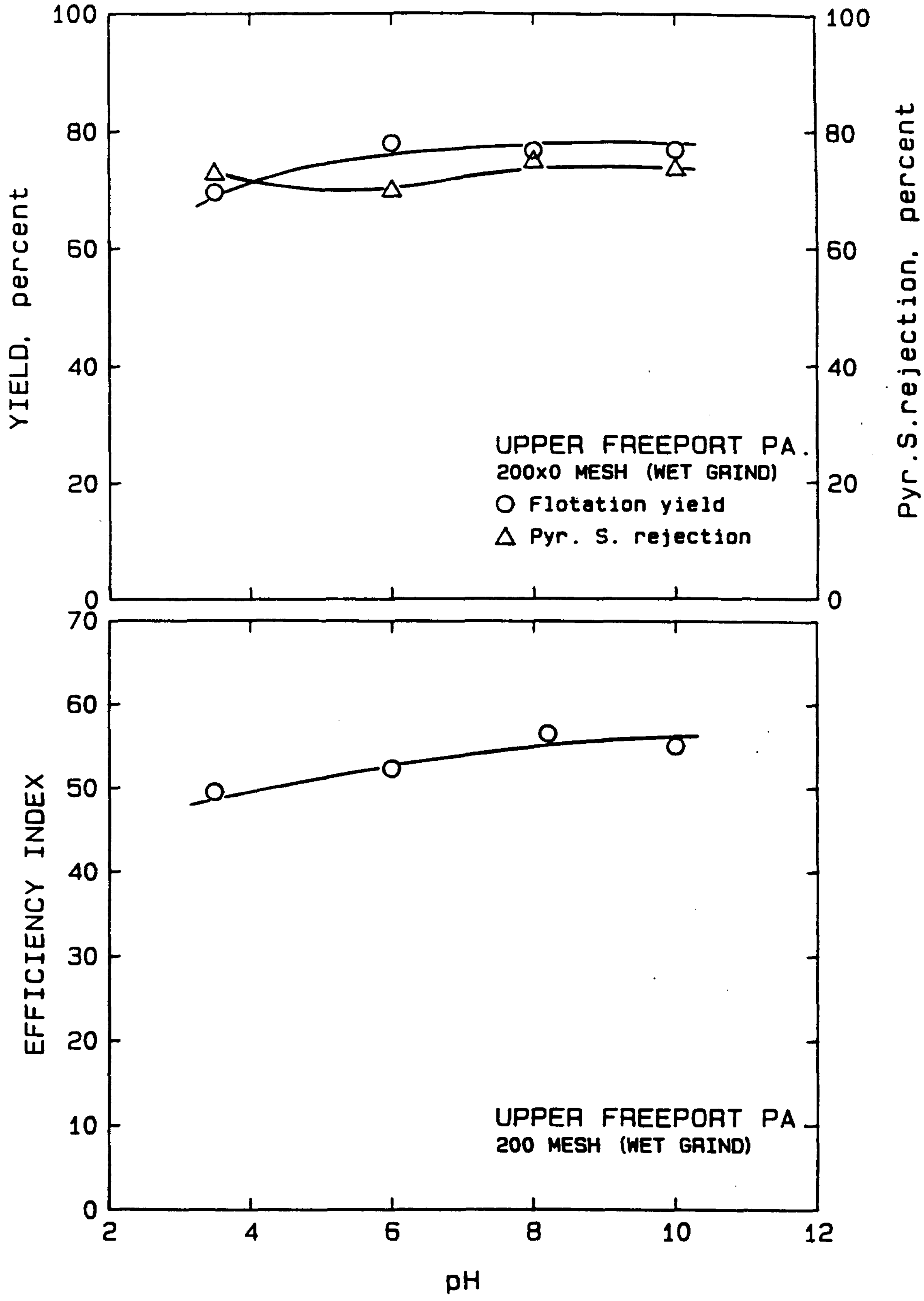

Figure 6.5 The effect of pH on the flotation performance of 200 mesh wet-ground Upper Freeport PA coal.

rejection tends to increase slightly over the pH range tested. The efficiency index increased slightly with pH in the range 3.5 to 8 beyond which it appears to remain constant. Tests with 28 mesh grind are currently being undertaken.

In summary, the effect of lime as pH modifier on flotation performance of coal appears to be a specific function of various factors such as origin, size and mode of grinding.

6.2 Effect of Surface Modifiers

Surface modifying reagents used in our study were all selected to enhance the hydrophobicity of coal, unlike the experiments with lime being added to depress pyrite. The effect of four major classes of reagents (anionic, non-ionic, polymerizable monomers and non-reactive reagents) was examined this quarter. While evaluating the effect of various reagents, the collector (n-dodecane) and frother (MIBC) additions were varied as required to compensate for collecting or frothing properties of the added reagents. A detailed summary of the results obtained is discussed in the following sections to compensate for collecting or frothing properties of the added reagents.

6.2.1 Effect of Anionic Reagents

The two anionic reagents examined during this quarter were 2,n-butyl thiophene and 2-ethylhexyl sulfosuccinate (Aerosol OT). Alkylated thiophene may chemisorb on coal, as its structure is similar to those compounds in coal, which incorporates organic sulfur into cyclic rings containing carbon. Dialkyl sulfosuccinates, introduced in 1939 by the American Cyanamid company under the Aerosol trademark, are a widely used class of wetting surfactants. The reagent used in this study, Aerosol OT, accounts for 80 percent of dialkyl sulfosuccinates produced in the United States.

6.2.1.1 Effect of 2,n-butyl thiophene

The flotation performance of wet-ground 28 and 200 mesh Pittsburgh No. 8 coal was evaluated after adding 2,n-butyl thiophene during grinding. Flotation was carried out at pH 6 and the results obtained are summarized in Tables 6.1 and 6.2 for the 28 mesh and 200 mesh grinds, respectively. The results of the standard flotation test are also included in these tables, for comparison. An examination of results in Table 6.1 shows that, at standard collector and frother dosages (2.10 lb/T and 0.43 lb/T respectively), the addition of 0.7 lb/T of surface modifier results in a substantial increase in the efficiency index from 27 to 45. However, increasing the dosage to 1.4 lb/T results in a marginal decrease in the efficiency index from that obtained at 0.7 lb/T. Also, at 1.4 lb/T 2,n-butyl thiophene dosage, decreasing the level of dodecane to less than a third of the standard amount does not cause any significant reduction in the flotation yield. This indicates that the surface modifier has also increased the hydrophobicity of coal and therefore much less collector addition was needed to obtain similar flotation recovery. Also, an increase in the efficiency index upon the addition of the surface modifier indicates that 2,n-butyl thiophene selectively enhances the hydrophobicity of the coal. However, in the case of 200 mesh grind, the

Table 6.1 Effect of surface modifier (2,n-butyl thiophene) addition on the flotation performance of 28 mesh wet ground Pittsburgh No. 8 coal at pH 6.

REAGENT DOSAGE			FLOTATION PROD ANAL			PERCENT REJ		
Coll. lb/T	Frot. lb/T	Mod. lb/T	Yield %	Ash %	Pyr S %	Ash	Pyr S	EI
2.10	0.43	--	86.5	7.5	2.09	46.0	33.0	23*
2.10	0.43	--	84.3	6.6	2.01	53.6	37.2	27
2.10	0.43	0.70	88.1	7.7	1.45	43.5	52.7	45
2.10	0.43	1.40	88.5	7.9	1.70	41.7	44.3	37
0.65	0.43	1.40	85.6	7.0	1.58	50.1	49.9	40

* Standard flotation test; pH = 3.4

Table 6.2 Effect of surface modifier (2,n-butyl thiophene) addition on the flotation performance of 200 mesh wet ground Pittsburgh No. 8 coal at pH 6.

REAGENT DOSAGE			FLOTATION PROD ANAL			PERCENT REJ		
Coll. lb/T	Frot. lb/T	Mod. lb/T	Yield %	Ash %	Pyr S %	Ash	Pyr S	EI
2.10	0.30	--	77.5	4.9	1.20	68.3	65.5	48*
2.10	0.30	--	66.5	4.8	0.81	73.4	80.0	52
2.10	0.30	0.70	57.7	4.7	0.87	71.4	76.5	56
1.70	0.30	1.40	71.5	4.9	0.87	72.6	79.6	55

* Standard flotation test; pH = 4.3

effect of 2,n-butyl thiophene was found to be only marginal as shown in Table 6.2. Relatively less influence of 2,n-butyl thiophene with finer particles may be due to the fact that even the base separation (without surface modifier addition) is at the high end of the efficiency index (EI ~ 50), compared to the coarser feed (EI~27). Further tests are planned using this surface modifier on Pittsburgh No. 8 and also the other two base coals.

6.2.1.2 Effect of Aerosol OT

The effect of Aerosol OT addition on improving the efficiency of pyritic sulfur removal was evaluated for wet-ground 200 mesh Pittsburgh No. 8 and Upper Freeport coals. The flotation results obtained, along with the calculated values of the efficiency index, for Pittsburgh No. 8 coal are summarized in Table 6.3. Examination of these results shows that, up to a dosage of 0.35 lb/T, the addition of Aerosol OT improves the flotation recovery. However, enhancement in the efficiency index is not achieved with Aerosol OT additions less than 0.7 lb/T. Also, as observed in the case of 2,n-butyl thiophene, decreasing the collector dosage in the presence of Aerosol OT does not cause any reduction in the flotation recovery. These results indicate that Aerosol OT, like 2,n-butyl thiophene, selectively enhances the hydrophobicity of coal.

Table 6.3 Effect of surface modifier (Aerosol OT) addition on the flotation performance of 200 mesh wet ground Pittsburgh No. 8 coal at pH 6.

REAGENT DOSAGE			FLOTATION PROD ANAL			PERCENT REJ		
Coll. lb/T	Frot. lb/T	Mod. lb/T	Yield %	Ash %	Pyr S %	Ash	Pyr S	EI
2.10	0.30	--	77.5	4.9	1.20	68.3	65.5	48*
2.10	0.30	--	66.5	4.8	0.81	73.4	80.1	52
2.10	0.30	0.18	73.1	5.0	1.00	69.5	72.9	52
2.10	0.30	0.35	82.8	5.7	1.15	60.7	64.7	53
2.10	0.30	0.70	83.6	5.6	0.97	61.0	70.0	60
1.20	0.30	0.70	83.6	5.6	0.97	61.0	70.0	60

* Standard flotation test; pH = 4.3

Flotation results obtained with Upper Freeport coal (Table 6.4) show that, in the dosage range tested, Aerosol OT has less effect than observed with Pittsburgh No. 8 coal.

6.2.2 Effect of Polymerizable and Non-polymerizable Organic Monomers

The monomers used in our study during this quarter were styrene and vinyl acetate. These are high volatile organics which can vaporize in the dry grinding environment. The vapors can adsorb on to coal surfaces and may polymerize with broken high energy bonds created by comminution. Because styrene and vinyl acetate may have some specific interaction on the flotation of coal that is not a result of a polymerization reaction, flotation tests with ethyl benzene and ethyl acetate, homologues of styrene and vinyl acetate, were also carried out. The effect of these organic additives on the flotation performance of the three base coals dry-ground to minus 200 mesh was examined and the results are summarized in Tables 6.5 - 6.7. It can be seen from these results that the addition of these monomers does not cause any significant improvement in sulfur removal efficiency and that the nonpolymerizable homologues have an equal effect on

Table 6.4 Effect of surface modifier (Aerosol OT) addition on the flotation performance of 200 mesh wet ground Upper Freeport PA coal.

REAGENT DOSAGE			FLOTATION PROD ANAL			PERCENT REJ		
Coll. lb/T	Frot. lb/T	Mod. lb/T	Yield %	Ash %	Pyr S %	Ash	Pyr S	EI
0.26	0.23	--	69.5	5.3	0.50	69.0	73.0	49*
0.26	0.23	0.05	76.9	6.2	0.55	63.3	70.0	54
--	0.23	0.05	68.7	5.8	0.47	69.3	75.0	49
0.26	0.23	0.09	83.3	7.3	0.64	53.5	58.4	47
--	0.23	0.09	78.5	6.6	0.58	60.1	65.0	49

* Standard flotation test

flotation, indicating that the monomers are probably not polymerizing with the coal surface. One possible reason for the ineffectiveness of these reagents is the presence of inhibitors in the monomer added to prevent a dangerous spontaneous polymerization of the liquid. These inhibitors may also prevent the polymerization reaction between the coal surface and the monomer. Therefore, it is proposed to purify the monomers by distillation, effectively removing the inhibitors, and then evaluate the effect of a pure monomer as a surface modifier.

6.2.3 Effect of Non-ionic Reagents

Flotation performance of wet-ground 200 mesh Upper Freeport coal with such non-ionic reagents such as methanol and ethanol was studied and the results obtained are summarized in Figures 6.6 and 6.7. Examination of these results indicates that there is a marginal effect of ethanol in selectively floating coal from pyrite. However, methanol does not show any effect. Further analysis of these results shows that, although ethanol addition has a favorable effect on pyrite rejection, flotation recoveries obtained are generally decreased.

Table 6.5 Effect of surface modifier (organic monomer) addition on the flotation performance of 200 mesh dry ground Illinois No. 6 coal.

	REAGENT DOSAGE			FLOTATION PROD ANAL		
Surface Modifier	Collector lb/T	Frother lb/T	Mod. lb/T	Yield %	Ash %	Tot. S %
None	6.30	1.30	--	59.1	8.9	3.4
Styrene	6.30	1.30	3.20	62.8	8.7	3.7
	6.30	1.30	6.40	67.3	8.6	3.7
Ethyl Benzene	6.30	1.30	3.20	63.2	8.3	3.6
	6.30	1.30	6.40	65.5	9.1	3.6
Vinyl Acetate	6.30	1.30	4.80	62.9	8.9	3.7
Ethyl Acetate	6.30	1.30	4.80	67.6	8.8	3.6

Table 6.6 Effect of surface modifier (organic monomer) addition on the flotation performance of 200 mesh dry ground Pittsburgh No. 8 coal.

	REAGENT DOSAGE			FLOTATION PROD ANAL		
Surface Modifier	Collector lb/T	Frother lb/T	Mod. lb/T	Yield %	Ash %	Tot. S %
None	2.10	0.30	--	77.4	6.5	2.59
Styrene	2.10	0.30	4.80	83.7	6.6	2.78
Ethyl Benzene	2.10	0.30	4.80	84.5	7.0	2.85
Vinyl Acetate	2.10	1.30	4.80	84.9	6.7	2.78

Table 6.7 Effect of surface modifier (organic monomer) addition on the flotation performance of 200 mesh dry ground from Upper Freeport PA Coal.

	REAGENT DOSAGE			FLOTATION PROD ANAL		
Surface Modifier	Collector lb/T	Frother lb/T	Mod. lb/T	Yield %	Ash %	Tot. S %
None	0.52	0.23	--	74.1	7.2	1.41
Styrene	0.52	0.23	4.80	74.7	7.1	1.49
Ethyl Benzene	0.52	0.23	4.80	74.0	7.1	1.38
Vinyl Acetate	0.52	0.23	4.80	74.0	7.2	1.44

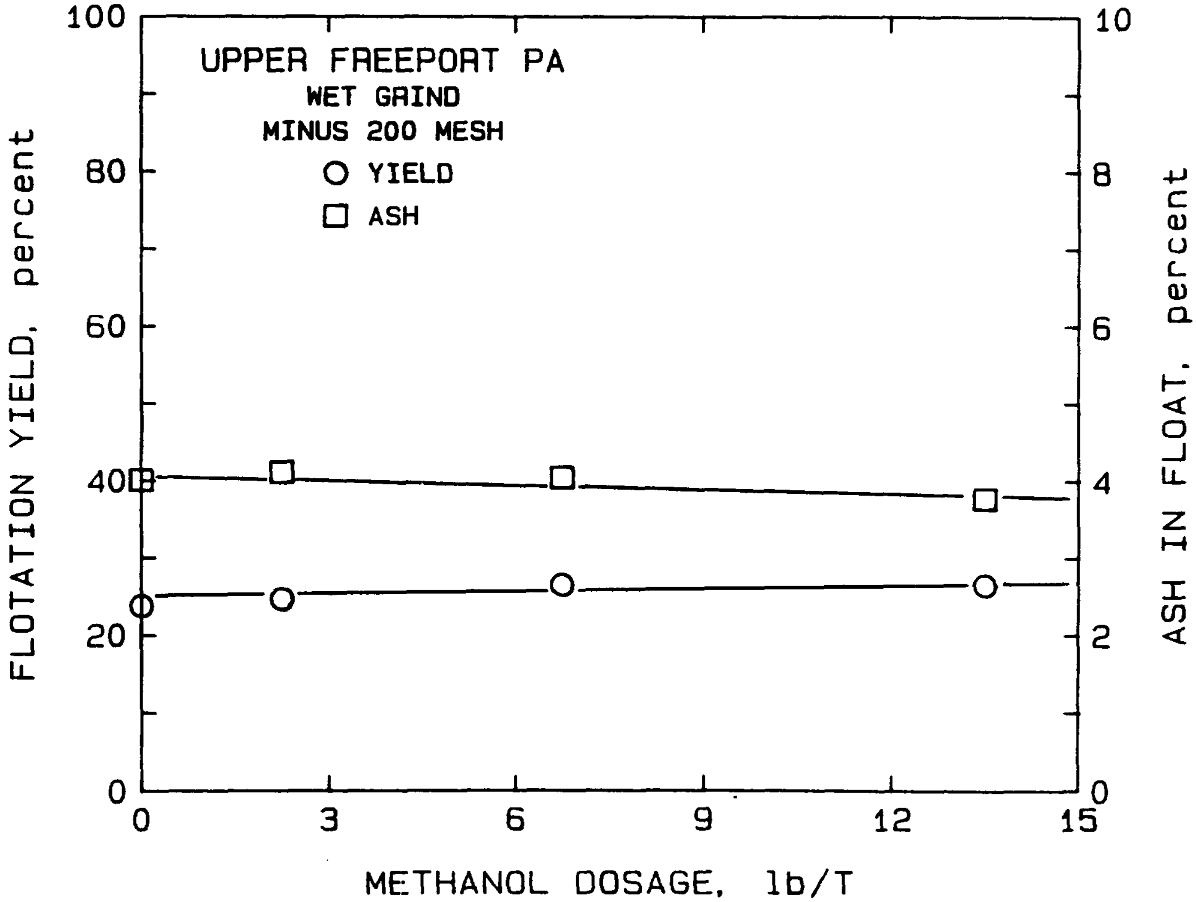

Figure 6.6 The effect of methanol addition on the flotation performance of Upper Freeport PA coal.

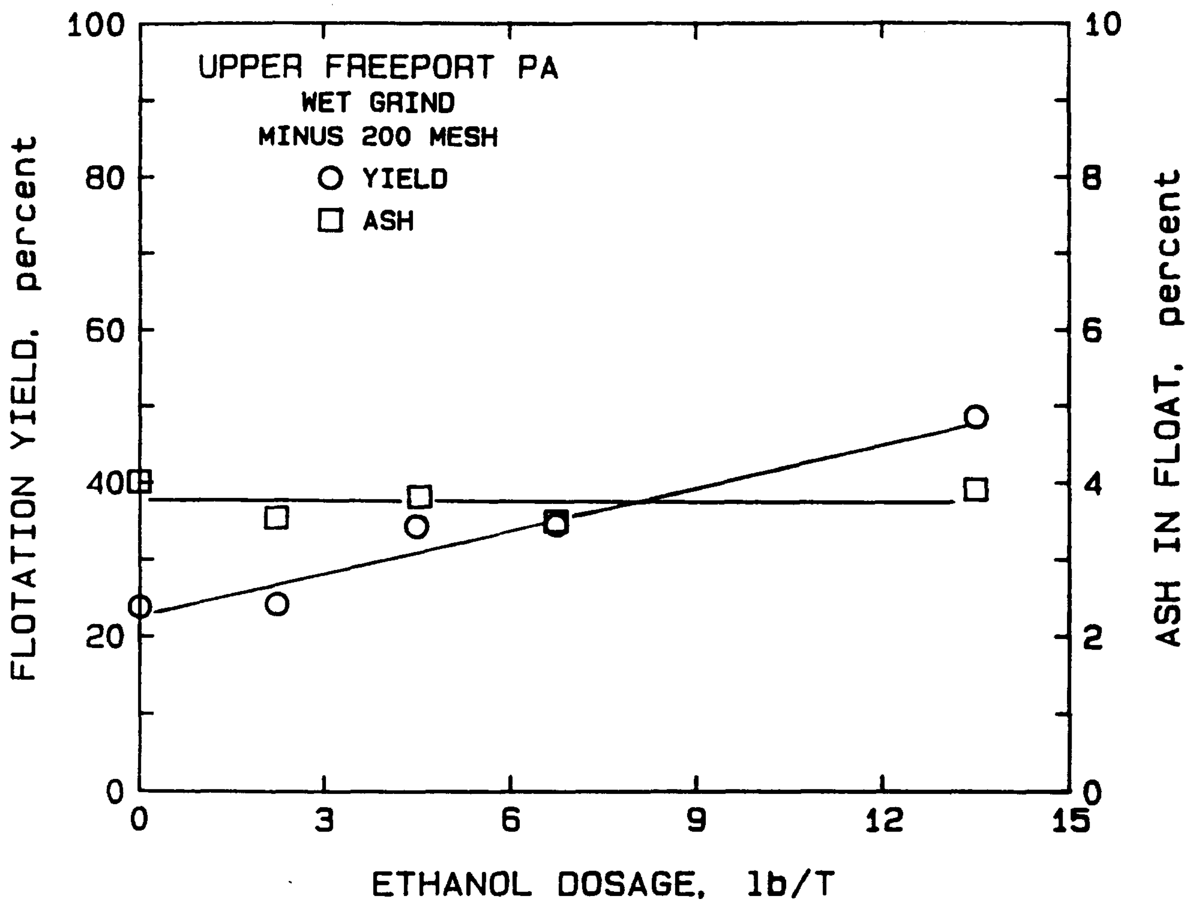

Figure 6.7 The effect of ethanol addition on the flotation performance of Upper Freeport PA coal.

7.0 WEATHERING STUDIES OF THE BASE COALS

The overall objective of the weathering study is to determine and understand the effect of the coal storage environment on its surface properties and, hence, on its flotation behavior. The procedure for generating the weathered samples has been discussed in detail in Task 2 of the Project Work Plan as well as in Section 2.0 of Quarterly Report No. 2.

For each of the three base coals (Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA) three size fractions, namely, +1 inch, 1 x 1/4 inch, 1/4 inch x 0, are weathered in inert (argon), covered and open conditions. The samples are collected at scheduled intervals. In all, a total of 15 increments of weathered samples are to be collected during the period starting from November 1988 and extending up to October 1989. The schedule provided for the collection of six biweekly samples for the first three months, followed by monthly sample collection for the remaining nine months. At the time of sampling, the minus 1/4 inch material is dry screened at 28 mesh, and both size fractions (that is, 1/4 inch x 28 mesh and minus 28 mesh) are re-inerted prior to shipping. The first ten increments of weathered samples were collected by the end of the past quarter (June 1989).

Progress on the experimental work involving the weathered samples was substantial during the past quarter. The completed test work consisted of surface and bulk characterization of the weathered samples, including proximate and sieve analyses. The surface hydrophobicity of these samples was assessed using the film flotation technique. Zeta potential measurements and Diffuse Reflectance Fourier Transform (DRIFT) spectra were also obtained for some selected samples. Since the weathering effect is more pronounced on smaller particles, laboratory-scale standard flotation tests of the smallest size fraction, namely, 28 mesh x 0 natural fines, and the ground sample of the next size interval (1/4 inch x 28 mesh) were carried out. The results are discussed in the following paragraphs of this section.

7.1 Characterization of Weathered Samples

Weathering is an important phenomenon in coal handling and processing. The impact of weathering on the behavior of coal ranges from the autoignition of coal piles in storage to alteration of the physical, chemical and surface characteristics. For example, weathering has been reported to decrease the surface area of coal (1), increase the moisture adsorption capacity (2,3), decrease the calorific value (4), increase the heat of wetting (5), decrease the mechanical strength (6), decrease the flotability (7), and reduce the coking power (8).

When exposed to atmospheric air, coals absorb and desorb moisture depending on the heat and humidity of the environment, and can react chemically with oxygen. While absorption of moisture leads to swelling, desorption leads to shrinkage. Repeated wetting and drying can result in fissuring and spalling. To what extent a given coal decrepitates depends on its rank and composition and the vagaries of the climatic conditions.

Chemisorption of oxygen on coal results in the formation of surface oxygen functional groups such as carboxyl, hydroxyl and carbonyl radicals. These oxygen functional groups reduce the hydrophobicity of the coal surface and thereby have a deleterious effect on the surface-based separation processes, such as flotation and oil agglomeration. In some instances weathering or oxidation may have beneficial effects. For example, when coal particles are to be heated on grates or under fluidized conditions as in gasification processes, agglomeration of the particles is undesirable. Since oxidation decreases the agglomerability, a preoxidation step is often included in the process scheme to prevent particles from adhering to each other. Most of the literature pertaining to the weathering aspects of coal research is limited to coal samples oxidized under controlled laboratory conditions, and little is known about the changes in the behavior of coal exposed to natural weathering, which has been the impetus for the current research effort.

7.1.1 Proximate Analysis of Weathered Samples

As a first step in characterizing the weathered samples, the proximate analysis (volatile matter, fixed carbon and ash) of the 28 mesh x 0 and 1/4 inch x 28 mesh fractions of the samples stored in inert (argon), covered and open modes was carried out using a LECO MAC 400 analyzer. The samples analyzed include those which were tested for flotation response. The results are tabulated in Table 7.1 for Illinois No. 6 coal, Table 7.2 for Pittsburgh No. 8 coal, and Table 7.3 for Upper Freeport PA coal.

The data given in Table 7.1 show that the weathered Illinois No. 6 coal samples from Increment 1 analyzed between 28.2% - 28.8% ash. The ash of the natural 28 M x 0 size fraction is considerably higher than the ash of the parent sample or of a pulverized representative sample from it. The higher ash in the screened fraction of the 28 M x 0 sample is probably due to a concentration of clays and mineral matter in the natural fines. The next higher size interval, 1/4 inch x 28 M, screened from weathering Increment 1 contained about 20 % ash. The lower ash content of the 1/4 inch x 28 M fraction is somewhat expected considering the overall mass balance of the parent sample whose ash content analyzed about 17%. Identical results have been obtained for other increments tested during this period for this coal.

Clearly the 28 mesh x 0 weathered samples differ from minus 28-mesh ground samples, since the former contain a higher concentration of the natural fine mineral matter. Not only is the weathering sample not compositionally representative but the particle size distribution of the two 28 M samples would be different as will be presented later in this section.

Table 7.2 gives the proximate analysis of the Pittsburgh No. 8 weathered samples for the 28 M x 0 size fraction from Increments 1 to 9 and for the 28 M x 1/4 inch size for Increments 1 and 8 for all three modes of storage. The ash content for coal stored in each of the three modes of storage are very close to each other. The higher ash content, 17 to 18%, of the

28 M x 0 size fraction as compared to 11% ash in the parent sample is again due to the concentration of mineral matter or clays in the natural fines. However, this effect is not as pronounced as that observed for the Illinois No. 6 coal. The ash content of the 1/4 inch x 28 M fraction for this coal analyzed 11.0%, which is close to the that of the parent sample.

In the case of Upper Freeport PA coal (Table 7.3), the ash content of the weathered samples (about 10 percent) is slightly lower than that of the research sample (about 12 percent). No difference between the ash contents of the 28 mesh x 0 and 1/4 inch x 28 mesh size fractions of the weathered samples was observed.

Even though weathering or oxidation is expected to release some of the volatile components from the coal (4), for the samples analyzed to date, no appreciable decrease in the volatile matter of the weathered samples has been observed for any of the three coals irrespective of the weathering mode possibly due to the weathering time and the average temperature during this time.

7.1.2 Sieve Analysis of the Weathered Samples

Weathering can occur by either mechanical (due to swelling) or chemical (due to oxidation) pathways. While chemical weathering alters the composition of the material, mechanical weathering can lead to size degradation. Since natural weathering is a combination of chemical and mechanical processes, the susceptibility of coal to weathering may be monitored by observing the size distribution of the weathered samples provided that the effect of weathering exceeds typical experimental or sampling variability or the alteration of the material which occurs during handling. Sieve analyses of the 28 mesh x 0 and 1/4 inch x 28 mesh fractions of the samples weathered under inert (argon), covered and open modes were obtained for each of the increments of the three base coals.

Table 7.1 Proximate analyses of weathered samples of Illinois No. 6 coal.

Increment	Size	Storage Mode	Moisture %	(ON DRY BASIS, %) Volatile Matter	Fixed Carbon	Ash
1	28 M x 0	Inert	2.83	34.0	37.2	28.8
	28 M x 0	Covered	3.10	33.7	37.6	28.7
	28 M x 0	Open	3.11	33.8	38.0	28.2
2	28 M x 0	Inert	3.11	33.1	37.1	29.8
	28 M x 0	Covered	3.26	33.1	37.4	29.5
	28 M x 0	Open	3.16	32.1	36.0	31.9
3	28 M x 0	Inert	3.16	33.6	37.6	28.8
	28 M x 0	Covered	3.32	33.5	37.3	29.2
	28 M x 0	Open	2.97	32.0	35.0	33.0
4	28 M x 0	Inert	3.06	33.8	37.3	28.9
	28 M x 0	Covered	3.14	34.0	37.6	28.4
	28 M x 0	Open	3.04	33.7	36.7	29.6
5	28 M x 0	Inert	3.34	33.5	37.3	29.2
	28 M x 0	Covered	3.86	33.9	37.3	28.8
	28 M x 0	Open	3.40	33.4	37.5	29.1
6	28 M x 0	Inert	5.58	33.2	36.9	29.9
	28 M x 0	Covered	5.68	33.7	37.9	28.4
	28 M x 0	Open	5.33	32.3	36.5	31.2
7	28 M x 0	Inert	4.54	33.9	38.2	27.9
	28 M x 0	Covered	4.63	33.6	37.8	28.6
	28 M x 0	Open	4.24	32.7	36.9	30.4
9	28 M x 0	Inert	2.80	34.3	37.5	28.2
	28 M x 0	Covered	3.30	34.8	37.1	28.1
	28 M x 0	Open	3.00	33.9	38.3	27.8
1	1/4" x 28M	Inert	5.73	35.2	45.0	19.8
	1/4" x 28M	Open	5.90	35.1	44.5	20.4
7	1/4" x 28M	Open	6.61	35.3	45.7	18.9

Table 7.2 Proximate analyses of weathered samples of Pittsburgh No. 8 coal.

Increment	Size	Storage Mode	Moisture %	(ON DRY BASIS, %) Volatile Matter	Fixed Carbon	Ash
1	28 M x 0	Inert	1.81	30.2	52.1	17.7
	28 M x 0	Covered	1.86	30.0	52.0	18.0
	28 M x 0	Open	1.86	30.2	51.9	17.9
2	28 M x 0	Inert	1.82	30.3	53.0	17.7
	28 M x 0	Covered	1.90	30.4	51.9	17.7
	28 M x 0	Open	1.95	30.4	51.9	17.7
4	28 M x 0	Inert	1.82	30.9	51.1	18.0
	28 M x 0	Covered	1.98	30.6	51.4	18.0
	28 M x 0	Open	2.0	31.2	50.0	18.8
6	28 M x 0	Inert	2.00	30.6	51.5	17.9
	28 M x 0	Covered	2.16	30.7	51.3	18.0
	28 M x 0	Open	2.35	30.6	50.5	18.9
8	28 M x 0	Inert	1.96	30.5	51.4	18.1
	28 M x 0	Covered	2.26	30.5	51.7	17.7
	28 M x 0	Open	2.15	30.3	51.8	17.9
9	28 M x 0	Inert	2.15	30.1	51.9	18.0
	28 M x 0	Covered	2.67	30.4	51.9	17.7
	28 M x 0	Open	2.62	30.4	51.9	17.7
1	1/4" x 28M	Inert	1.98	36.1	52.4	11.3
	1/4" x 28M	Open	2.00	36.13	52.7	11.2
8	1/4" x 28M	Inert	2.4	35.5	53.5	11.0
	1/4" x 28M	Open	2.4	36.2	53.0	10.8

Table 7.3 Proximate analyses of weathered samples of Upper Freeport PA coal.

				(ON DRY BASIS, %)		
Increment	Size	Storage Mode	Moisture %	Volatile Matter	Fixed Carbon	Ash
1	28 M x 0	Inert	0.88	25.7	64.4	9.9
	28 M x 0	Covered	0.86	25.6	64.6	9.8
	28 M x 0	Open	0.91	25.9	64.5	9.6
4	28 M x 0	Inert	0.94	25.3	65.1	9.6
	28 M x 0	Covered	1.06	25.2	64.8	10.0
	28 M x 0	Open	1.17	25.4	64.8	9.8
8	28 M x 0	Inert	1.00	25.8	64.5	9.7
	28 M x 0	Covered	0.99	25.8	64.5	9.7
	28 M x 0	Open	0.97	26.0	64.6	9.4
10	28 M x 0	Inert	1.02	25.3	64.9	9.8
	28 M x 0	Covered	1.06	25.5	64.7	9.8
	28 M x 0	Open	1.20	26.0	65.3	8.7
1	1/4" x 28M	Inert	0.91	26.5	64.6	8.9
	1/4" x 28M	Open	0.86	26.6	64.7	8.7
8	1/4" x 28M	Inert	0.94	27.3	63.7	9.1
	1/4" x 28M	Open	0.82	26.7	63.4	9.9

In Figures 7.1 to 7.4 the size distributions of the weathered samples of Illinois No. 6 coal are presented for Increments 1, 4, 6 and 7 (that is, 0.5, 2, 3 and 4 months of weathering), respectively. In the same plots the size distribution of the standard grind of the research sample of Illinois No. 6 coal is also presented for comparison. The most notable characteristic of the weathered samples is that the fines resulting from natural breakage or degradation are relatively coarser than those of the research sample ground in a rod mill even though the top sizes are nearly identical. The effect of the difference in the size distributions must be considered when comparing the floatation results of the weathered samples with the research samples.

The effect of the size degradation due to weathering can be elucidated from plots of the size distribution of the 28 mesh x 0 samples (Increments 1 through 7) stored under open conditions (Figure 7.5). Even though there appears to be no consistent size degradation, there may be a tendency for the breakdown of coarser particles, contributing to an increase of finer particles. This is better delineated by plotting the weight percent of material in a given size interval as a function of the weathering time. Figure 7.6 presents such plots for a number of size intervals (28 x 35 mesh, 48 x 65 mesh, 100 x 150 mesh and minus 150 mesh) for samples weathered up to 6 months (Increment 9) under open conditions. It is clear that there is a decrease in the amount of 28 x 35 mesh material as the weathering time increases, and an increase in the amount of minus 150 mesh material. However, there is only a nominal increase in material of intermediate size, namely, 48 x 65 mesh. Such a phenomenon might be ascribed to the increased friability of the weathered samples, due to the propagation of cracks and cleats in coal when the coal swells. This could result in a reduction of the mechanical strength of the coal, and hence in the generation of fine material. The probability of this type of behavior should be more pronounced in the case of large particles.

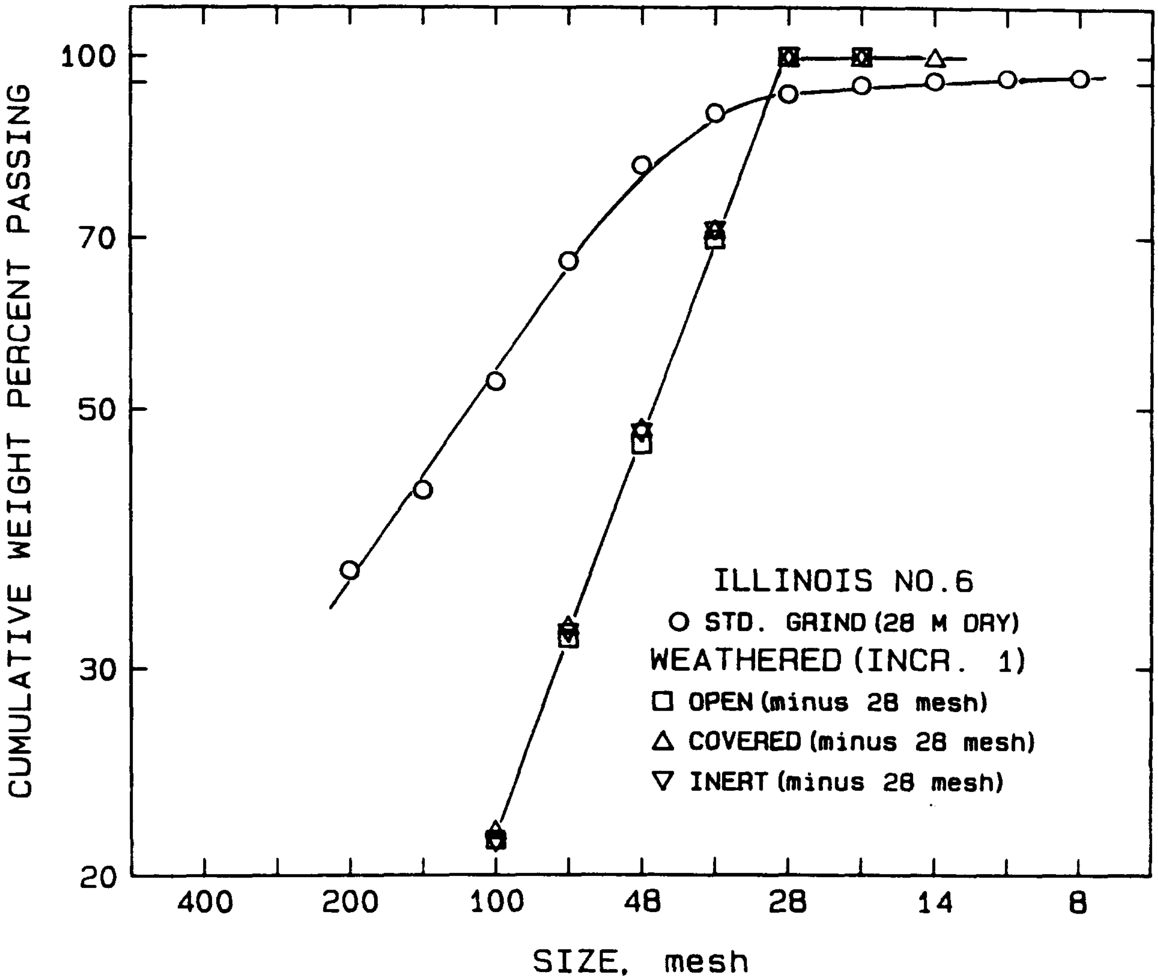

Figure 7.1 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and weathered sample Increment 1 (inert, covered and open) of Illinois No. 6 coal.

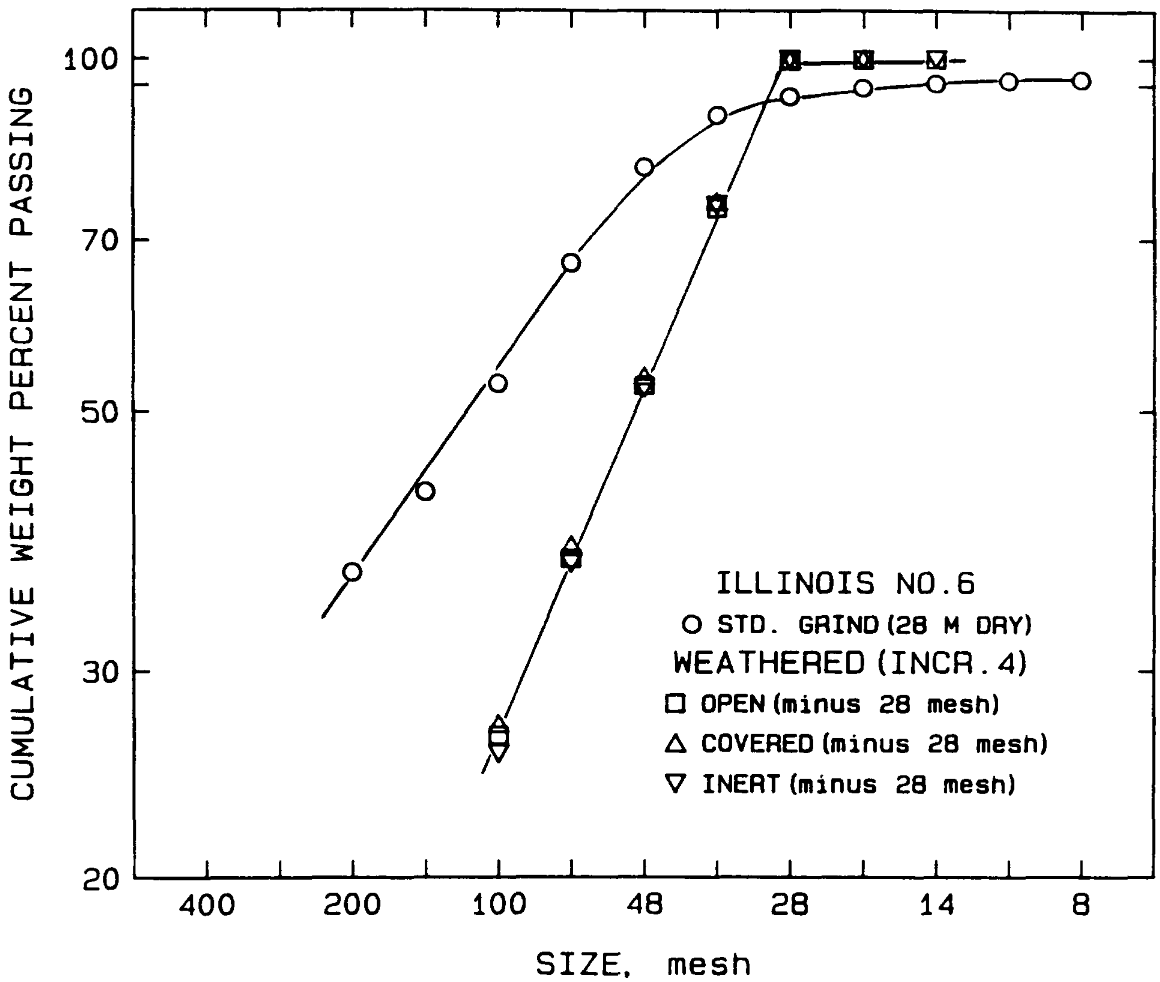

Figure 7.2 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and weathered sample Increment 4 (inert, covered and open) of Illinois No. 6 coal.

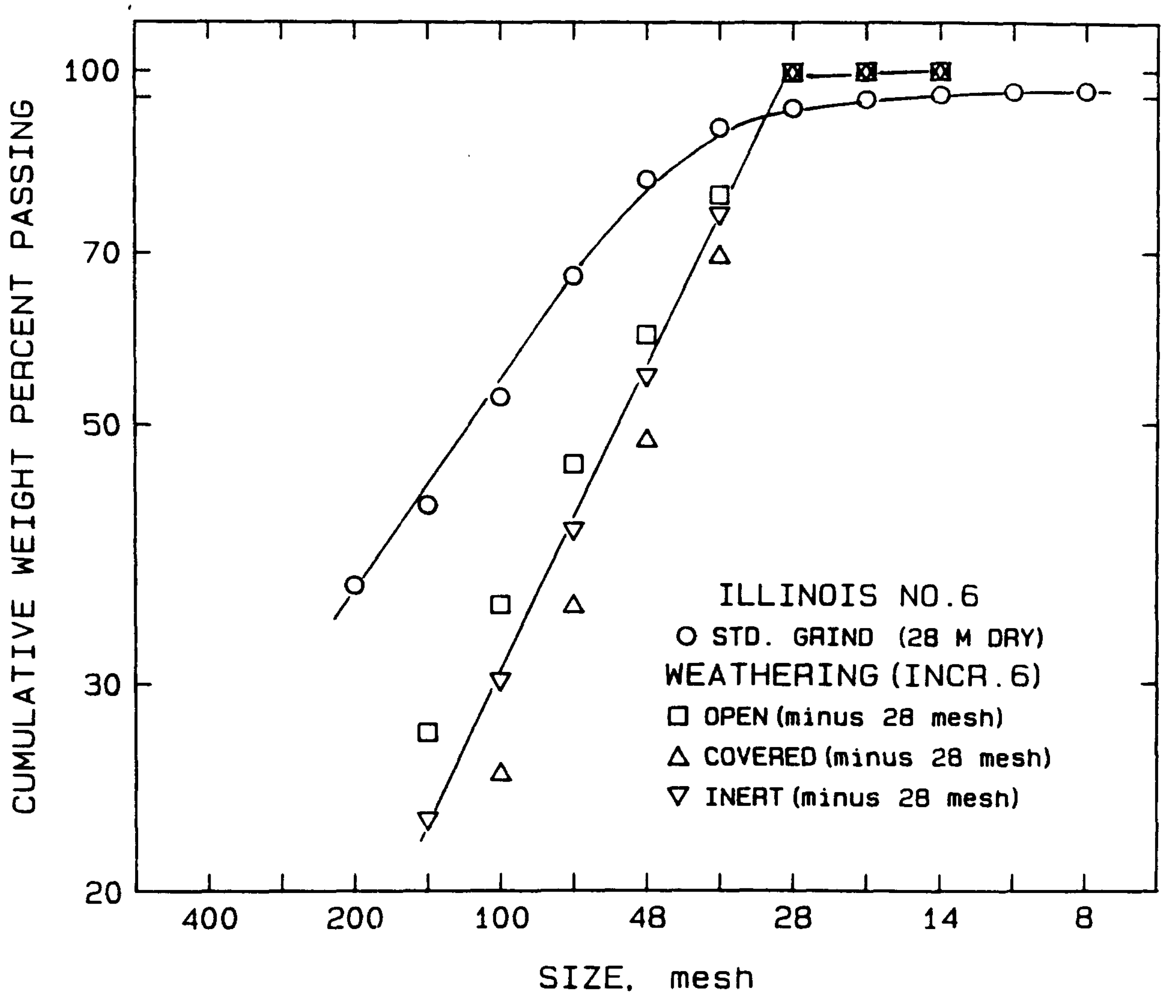

Figure 7.3 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and weathered sample Increment 6 (inert, covered and open) of Illinois No. 6 coal.

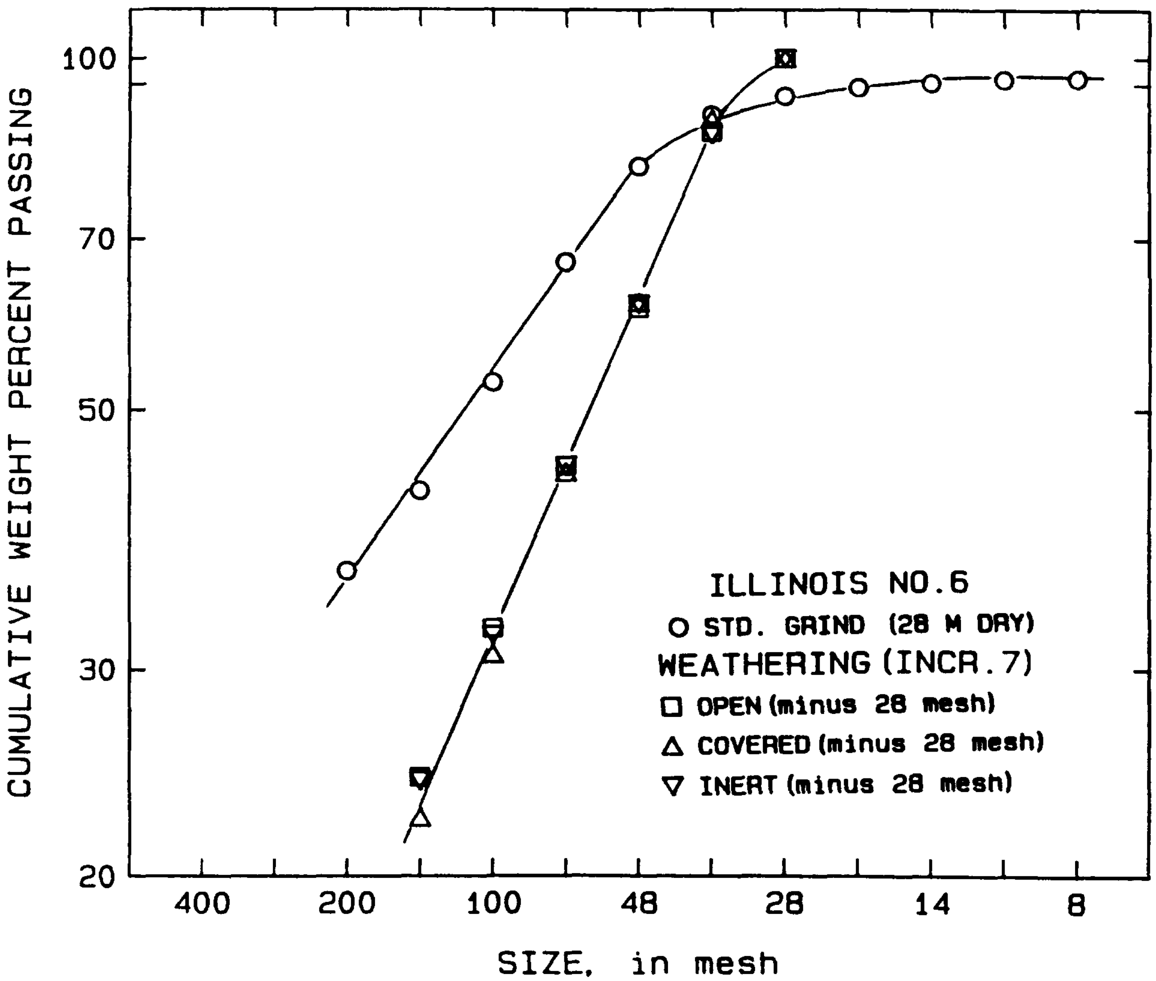

Figure 7.4 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 7 of the weathered sample (inert, covered and open) of Illinois No. 6 coal.

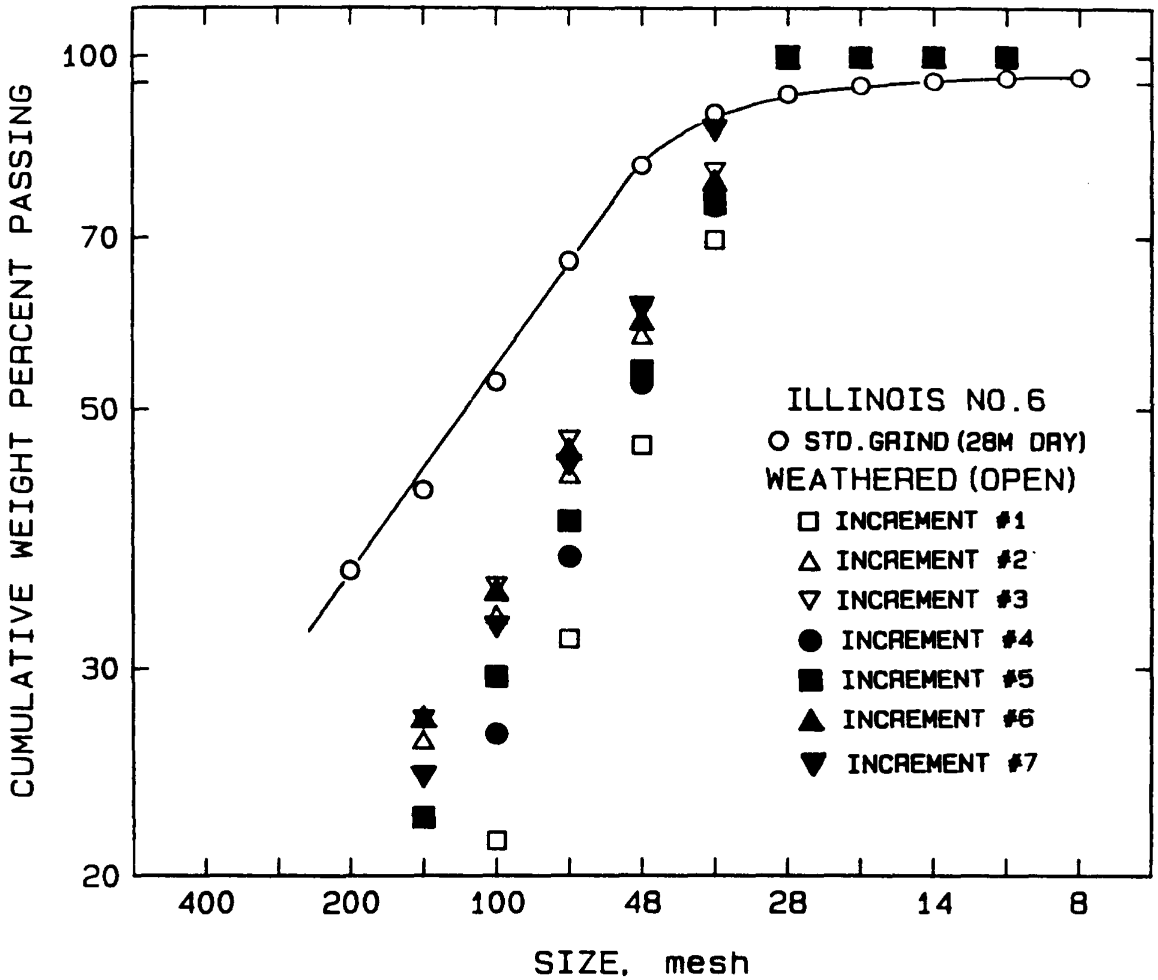

Figure 7.5 Effect of weathering on the size distribution of samples stored under open mode for Increments 1 through 7 of Illinois No. 6 coal.

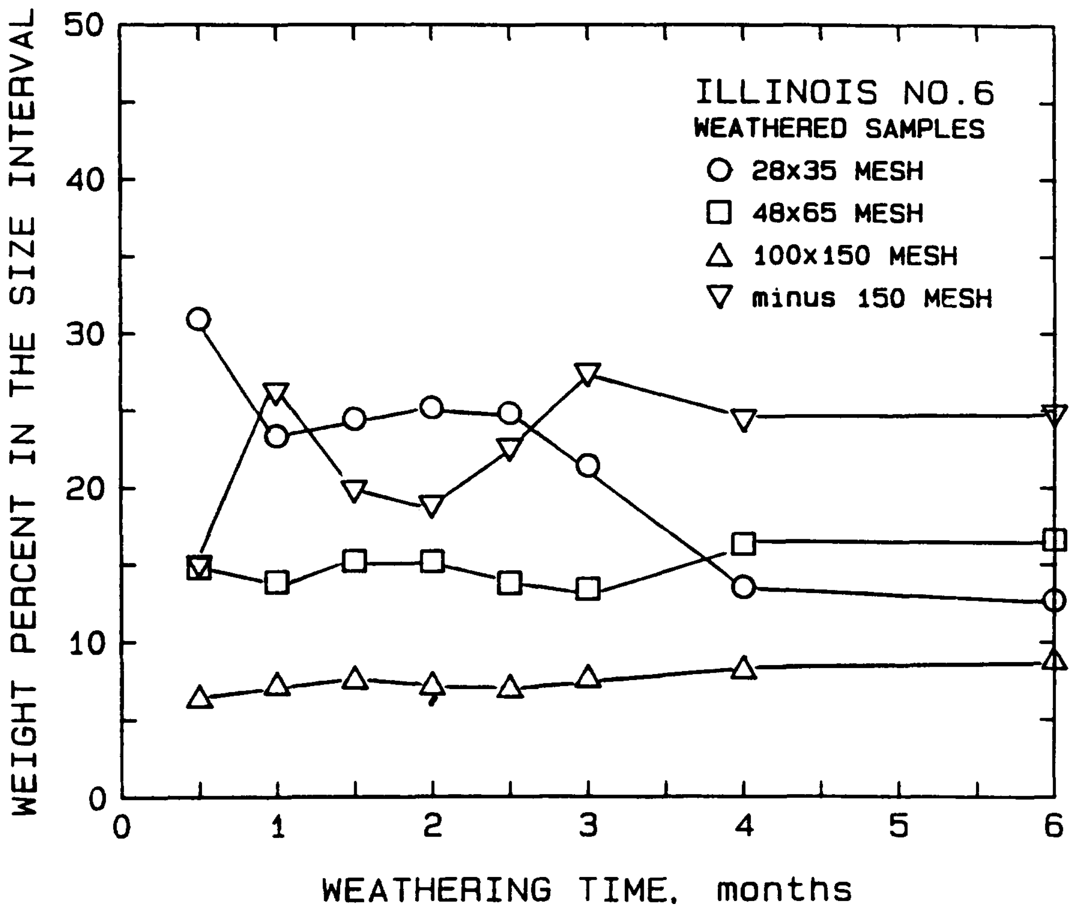

Figure 7.6 Effect of weathering time on the weight of material in different size intervals obtained by sieving the minus 28 mesh material of Illinois No. 6 coal weathered under open mode.

As a second step in understanding the effect of weathering on size degradation, sieve analyses of representative samples of the next larger size interval, namely 1/4 inch x 28 mesh material, were carried out. In Figure 7.7 the cumulative size distributions of the samples taken from Increments 1 (inert and open), 7 (open) and 9 (open) are presented. It is interesting to observe that in comparison to the inerted sample of the first increment (15 days of weathering), there is a progressive increase in the amount of fine particles. Because the minus 28 mesh material in this sample had been sieved out by the sampling laboratory, this effect is probably a result of increased friability of the coal and the spalling off of the material during sieve analysis.

The size distributions of the Pittsburgh No. 8 and Upper Freeport PA coals weathered under similar conditions, but at different locations, were also determined. As examples, the size distributions of samples from Increments 1 and 8 weathered under inert (argon), covered and open modes are presented in Figures 7.8 and 7.9. Unlike Illinois No. 6 coal, the size distributions of the weathered samples of Pittsburgh No. 8 coal are not appreciably different from that of the corresponding standard grind sample with the weathered samples being only slightly finer. Even in the case of next size interval (1/4 inch x 28 mesh), no size degradation is observed with weathering time (Figure 7.10). The weight percent of the material in various size intervals is plotted as a function of the weathering time in Figure 7.11. It appears that Pittsburgh No. 8 coal does not physically degrade due to weathering for the period studied so far.

Similar analyses were also carried out with the weathered samples of Upper Freeport PA coal. A comparison of the size distributions show that the weathered samples are coarser than that of the corresponding research sample ground in the rod mill (Figures 7.12 and 7.13). With respect to the weathering effect, a slight increase was observed in the amount of fines contained in the weathered 1/4 inch x 28 mesh material (Figure 7.14).

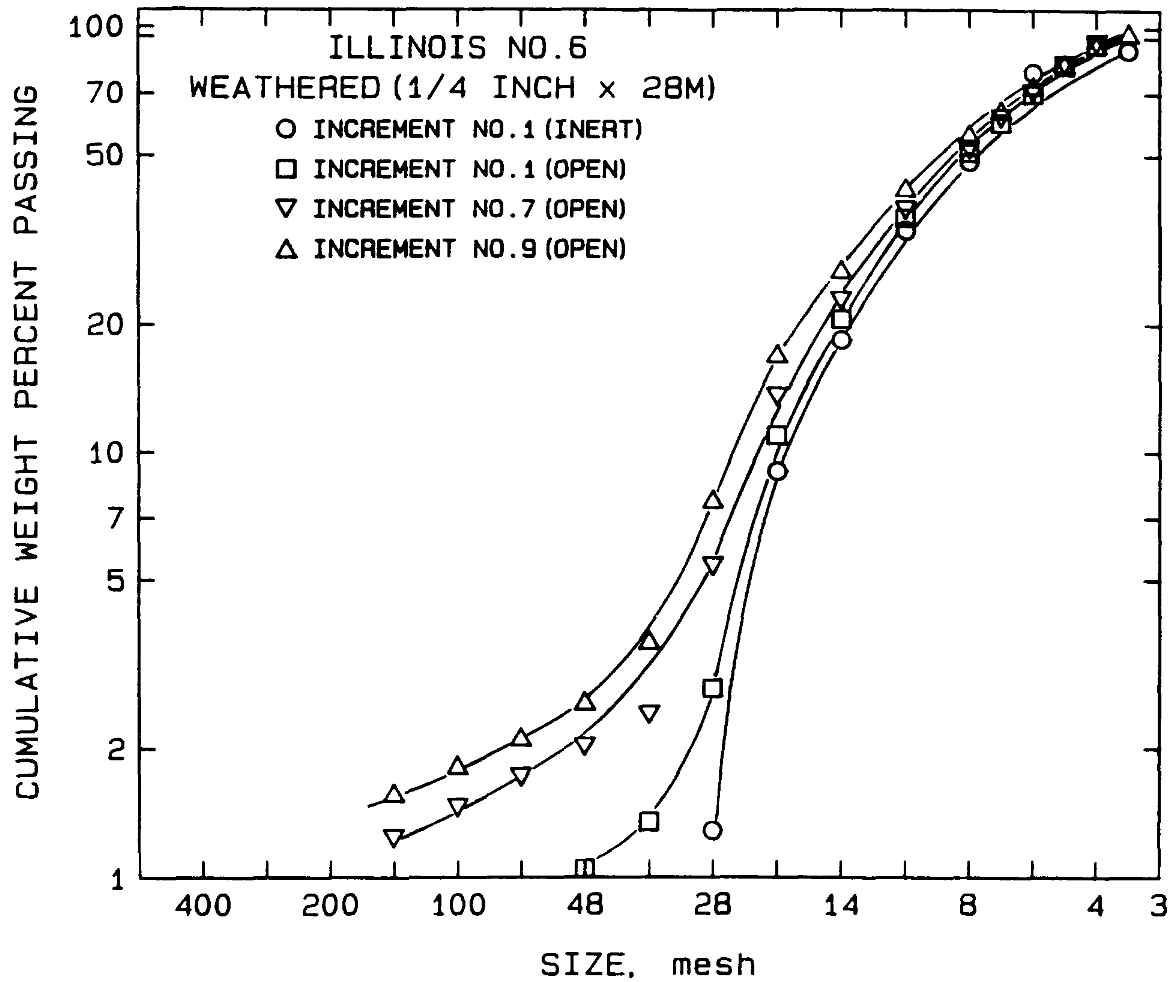

Figure 7.7 Effect of weathering on the size distribution of the 1/4 inch x 28 mesh Illinois No. 6 weathered coal samples (inert and open samples of Increment 1, and open samples of Increment 7 and 9).

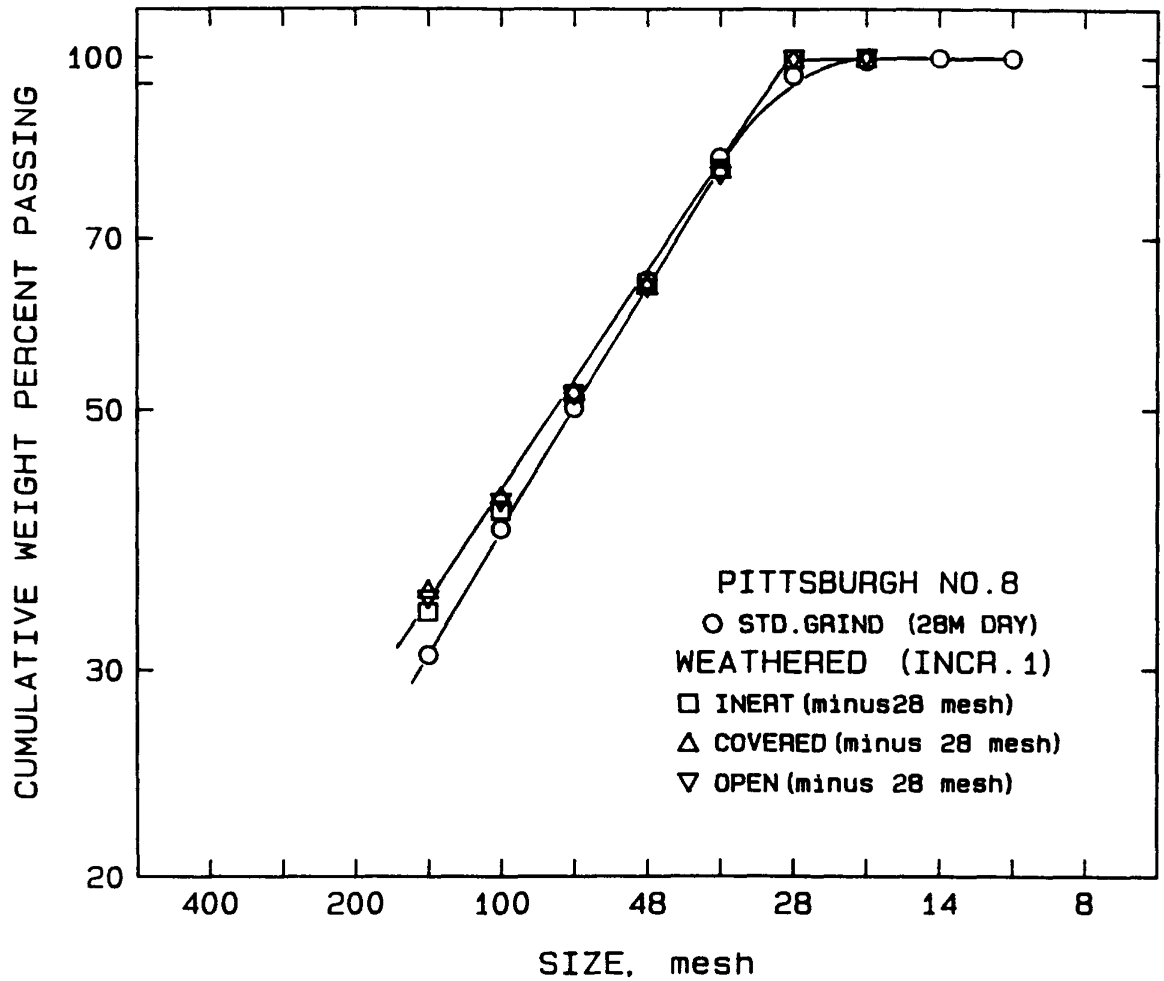

Figure 7.8 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 1 of the weathered sample (inert, covered and open) of Pittsburgh No. 8 coal.

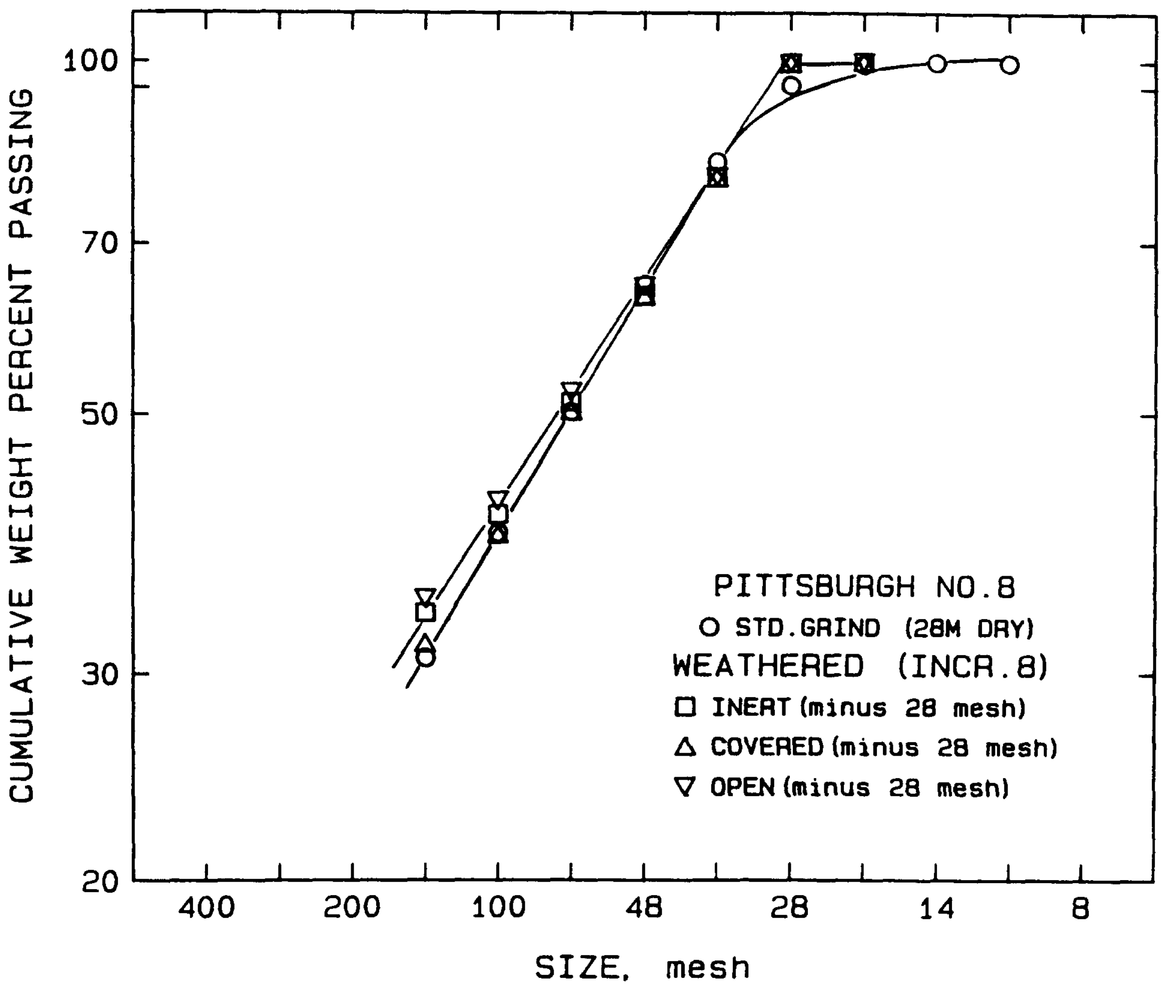

Figure 7.9 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 8 of the weathered sample (inert, covered and open) of Pittsburgh No. 8 coal.

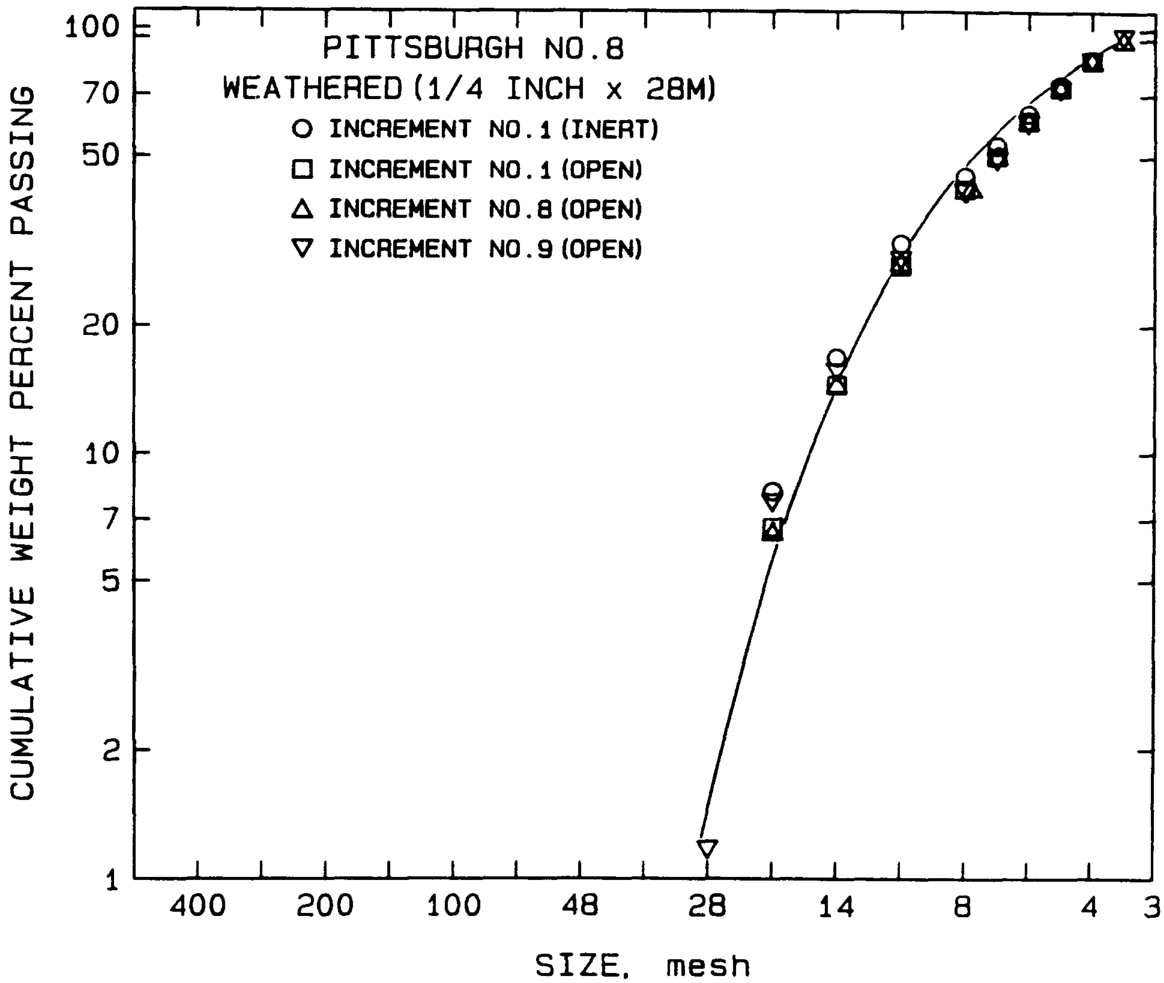

Figure 7.10 Effect of weathering on the size distribution of the 1/4 inch x 28 mesh weathered samples (inert and open samples of Increment 1, and open samples of Increment 8 and 9) of Pittsburgh No. 8 coal.

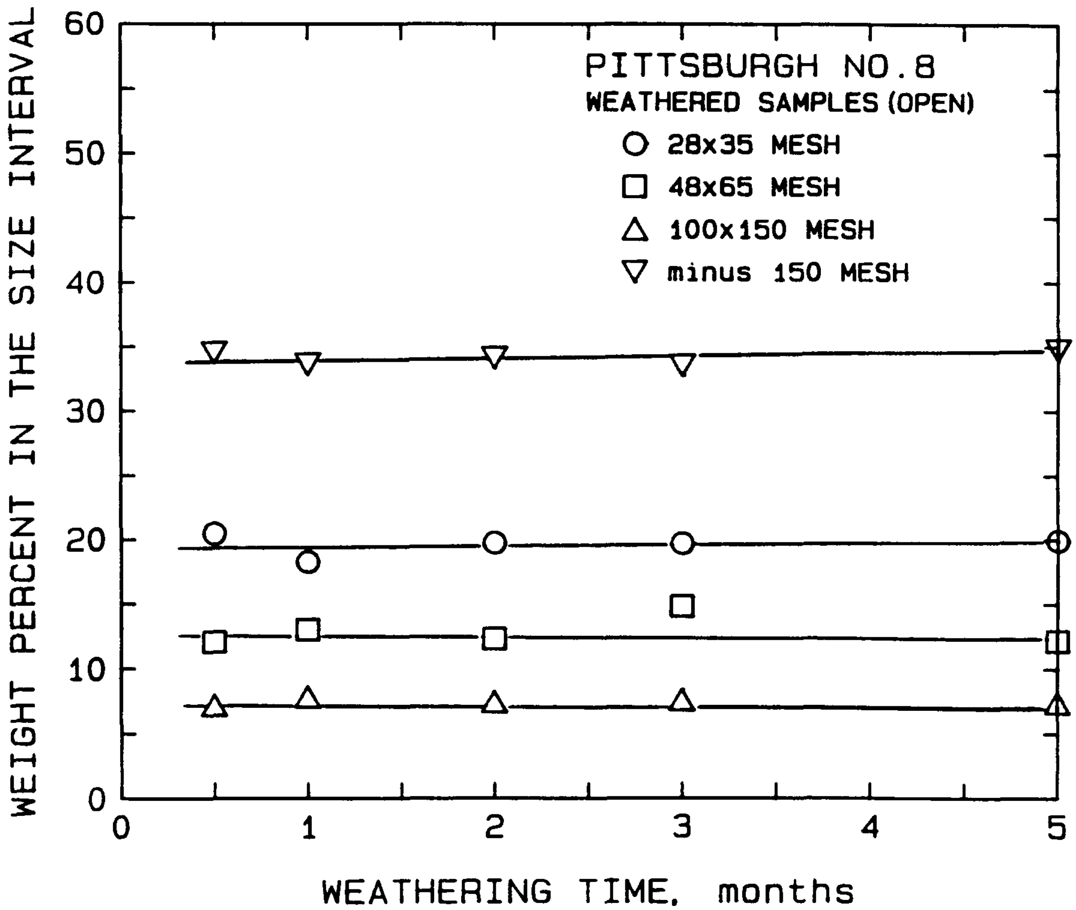

Figure 7.11 Effect of weathering time on the weight of material in different size intervals obtained by sieving the minus 28 mesh material of Pittsburgh No. 8 coal weathered under open mode.

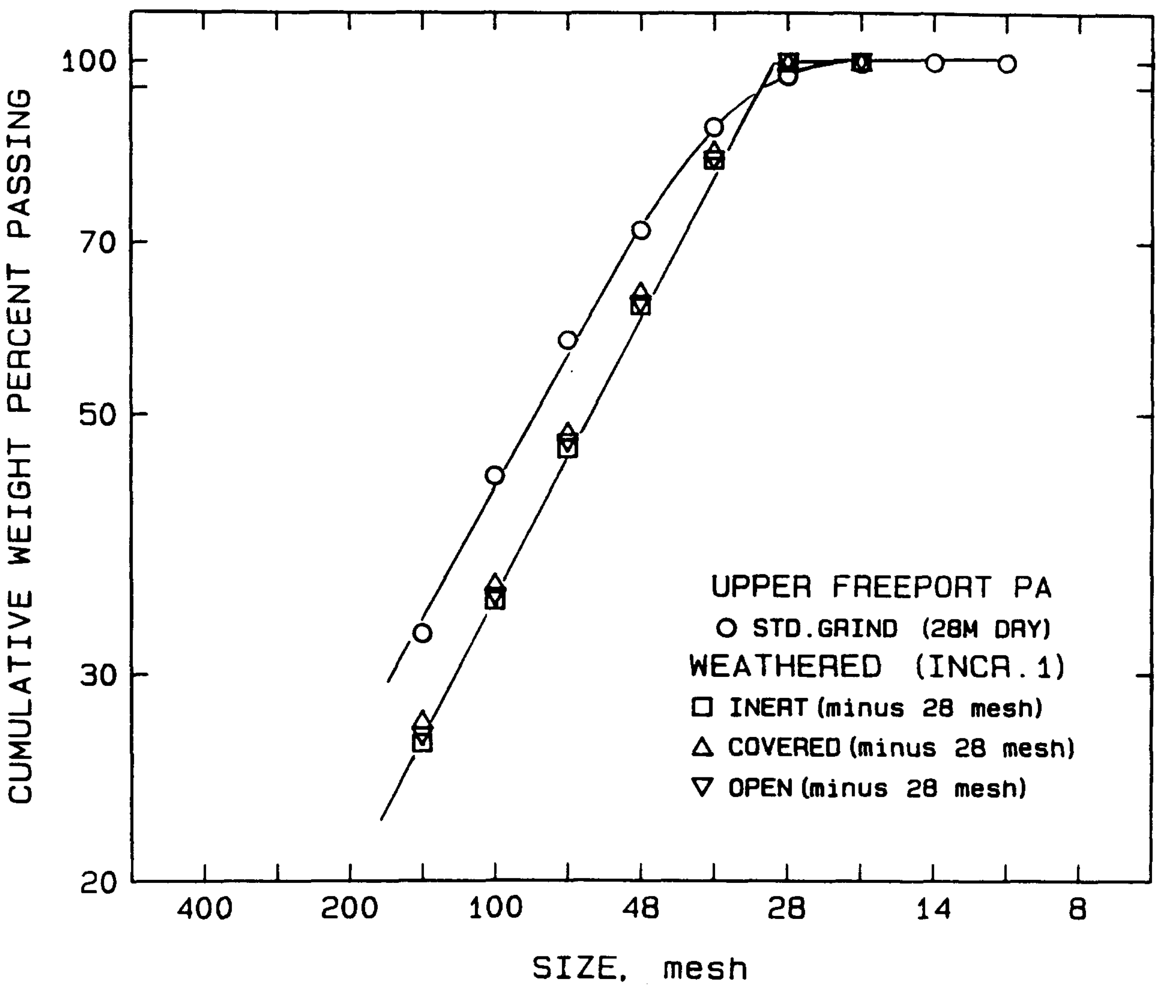

Figure 7.12 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 1 of the weathered sample (inert, covered and open) of Upper Freeport PA coal.

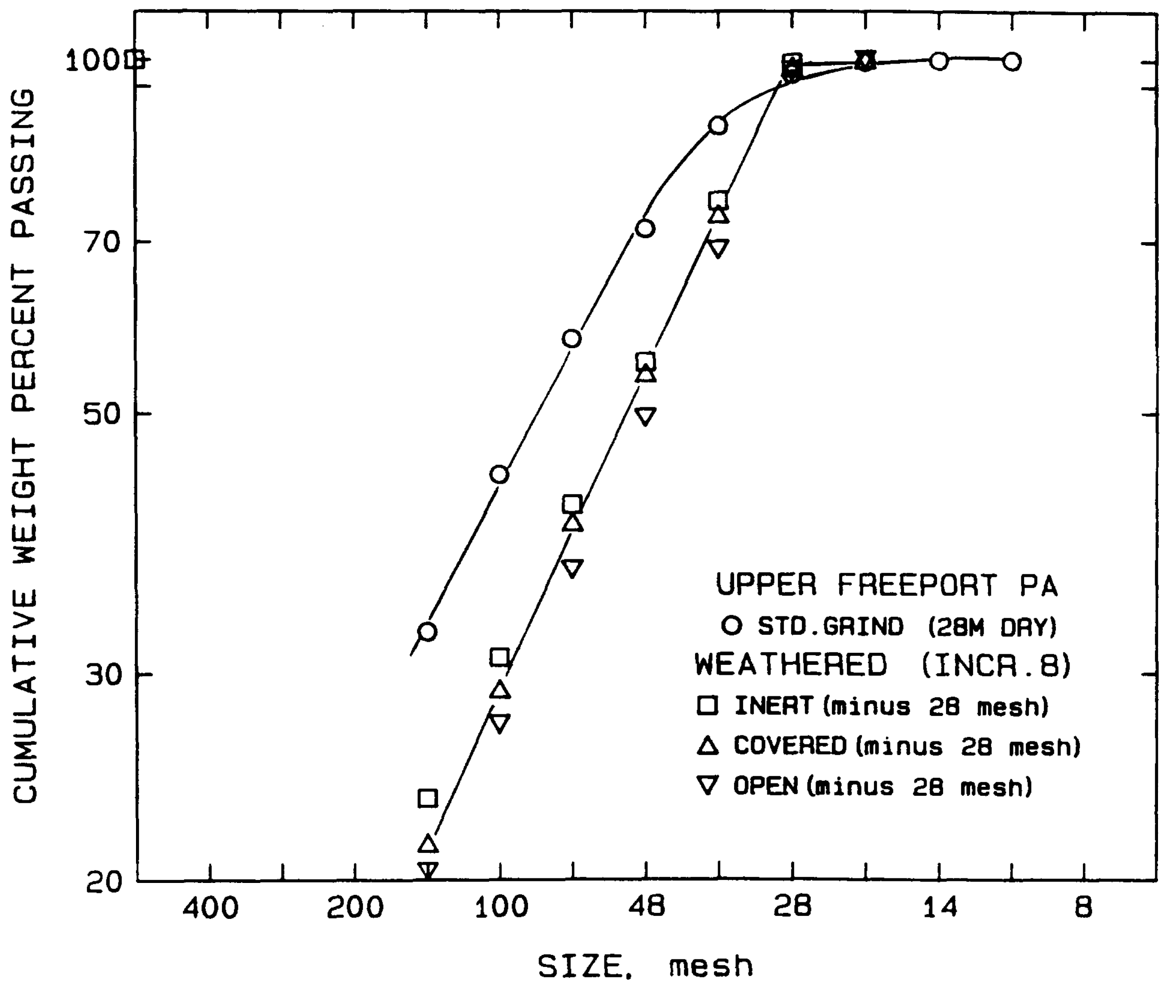

Figure 7.13 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 8 of the weathered sample (inert, covered and open) of Upper Freeport PA coal.

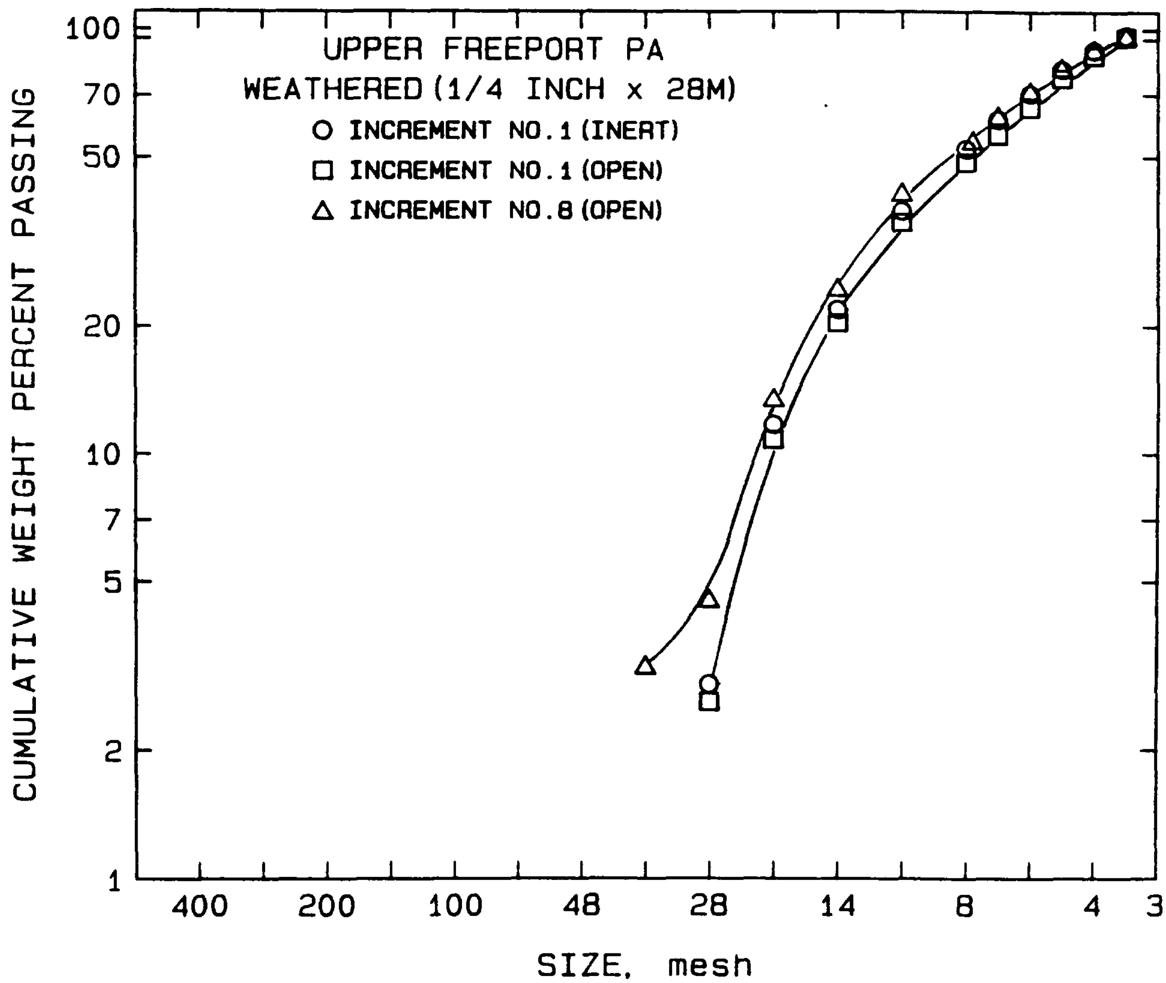

Figure 7.14 Effect of weathering on the size distribution of the 1/4 inch x 28 mesh weathered inert and open samples of Increment 1, and open sample of Increment 8 from Upper Freeport PA coal.

In summary, size degradation due to weathering affects Illinois No. 6 more than Upper Freeport PA coal. Weathering does not appear to have any effect on the size degradation of Pittsburgh No. 8. This effect, even in the extreme, is slight and is probably not as significant as the associated surface chemical effects of weathering.

7.1.3 Assessing the Hydrophobicity of Weathered Samples

The hydrophobic nature of particle surfaces is of utmost importance in surface-based separation processes. In this part of the report, the effect of weathering on the hydrophobicity of coal particles assessed by film flotation in aqueous methanol solutions is reported. In order to eliminate the effect of particle size, the 28 mesh x 0 size fraction material was sieved in an inert atmosphere in a glove box in order to obtain 100 x 150 mesh coal particles for the film flotation tests. The 100 x 150 mesh size fraction of each sample studied was dried under vacuum at room temperature to reduce the effect of moisture on film flotation and stored in glass bottles in an argon-filled glove box.

Figure 7.15 presents the film flotation partition curves of Increment 3 of Illinois No. 6 coal stored under inert, covered and open modes. It appears that both the inerted and covered samples have similar hydrophobic character, as compared with the sample weathered under open conditions. As part of this program, we have been concerned with the effect of fine particles adhering to larger particles, the so-called slime coating phenomenon. Since in flotation processes, the particulates are conditioned under turbulent conditions, slime coatings on particles are removed from the solid surface. However, there is no turbulence in film flotation, and hence, any small particles attached to the 100 x 150 mesh particles will probably remain attached. Therefore, in order to correlate the film flotation results with the actual laboratory flotation, the coal samples used for film flotation were deslimed by washing with running distilled water and then dried under

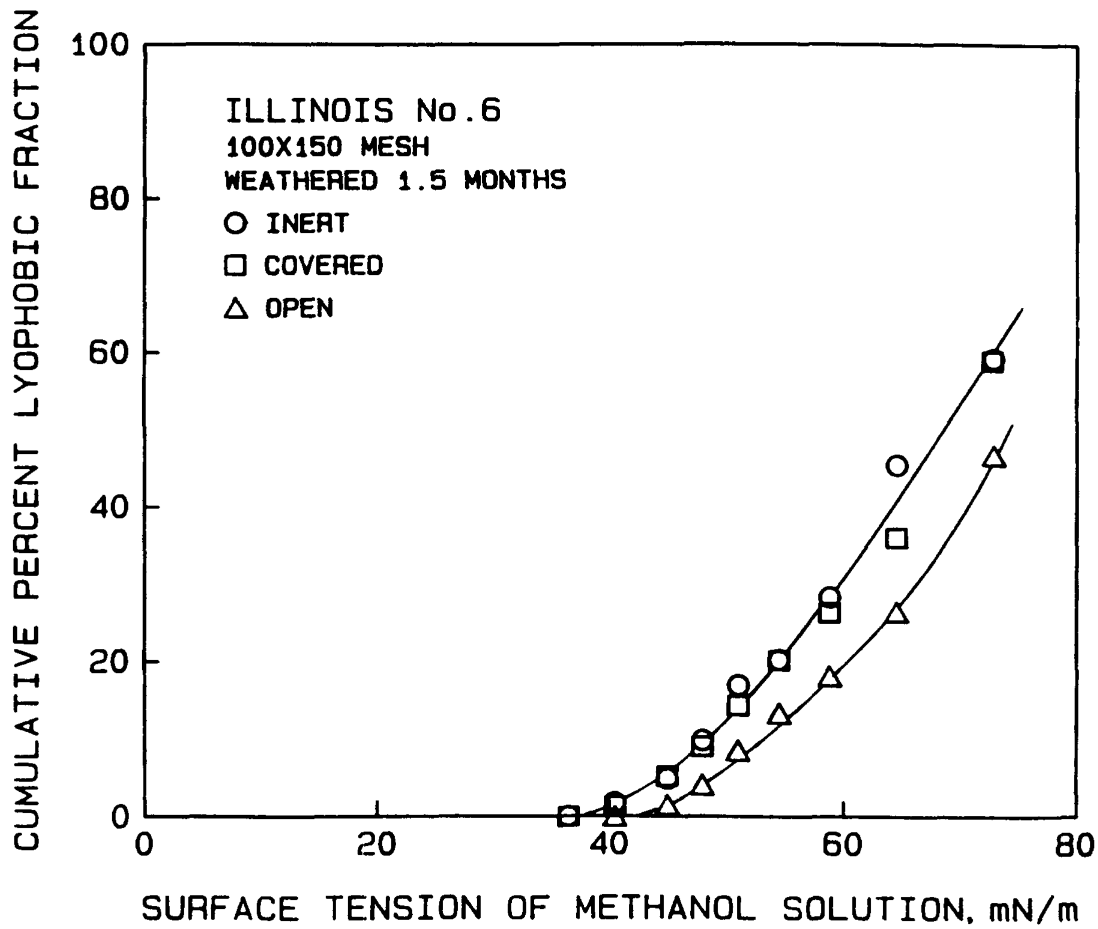

Figure 7.15 Film flotation partition curves of as-received samples of Illinois No. 6 (Increment 3) weathered under inert, covered and open conditions.

inert conditions to prevent further oxidation. In Figure 7.16 the film flotation partition curves of the deslimed sample (Increment 3) of Illinois No. 6 coal are shown. The results in Figure 7.16 indicate that the inert sample is more hydrophobic than either the covered or the open samples. A comparison of the proximate analysis of the 100 x 150 mesh sieved and deslimed samples indicate that the ash content of the sieved samples is higher than that of the deslimed sample. The higher ash content is indicative of the potential for slime coating by the ash-containing minerals, with its effect on the apparent hydrophobicity of the coal particles as shown is Figure 7.15. Therefore, subsequent testing by the film flotation technique was carried out using deslimed samples.

Further study of the effects of the time of weathering on the hydrophobicity of the coal was delineated by performing additional film flotation tests with Illinois No. 6 weathered under open conditions. In Figure 7.17, the film flotation partition curves of the samples weathered for 0.5, 1.5 and 4 months under open conditions are presented. The shift in the partition curve is not as significant between Increment 1 (15 days of weathering) and Increment 3 (1.5 months of weathering) as between Increment 3 and Increment 7 (4 months of weathering). The coal surface has become appreciably more hydrophilic as indicated by the shift in the partition curve. Similar testing is in progress with the weathered samples of Pittsburgh No. 8 and Upper Freeport PA coals. The results will be discussed in the next quarterly report.

7.1.4 Zeta Potential Measurements of Weathered Samples

To establish possible differences in the surface charge characteristics of coal samples weathered under various conditions for different lengths of time, zeta potential measurements were carried out for the initial increments of Illinois No. 6 and Pittsburgh No. 8 coal samples. For this purpose, the minus 200 mesh material was sieved from the 28 mesh x 0 fraction of the

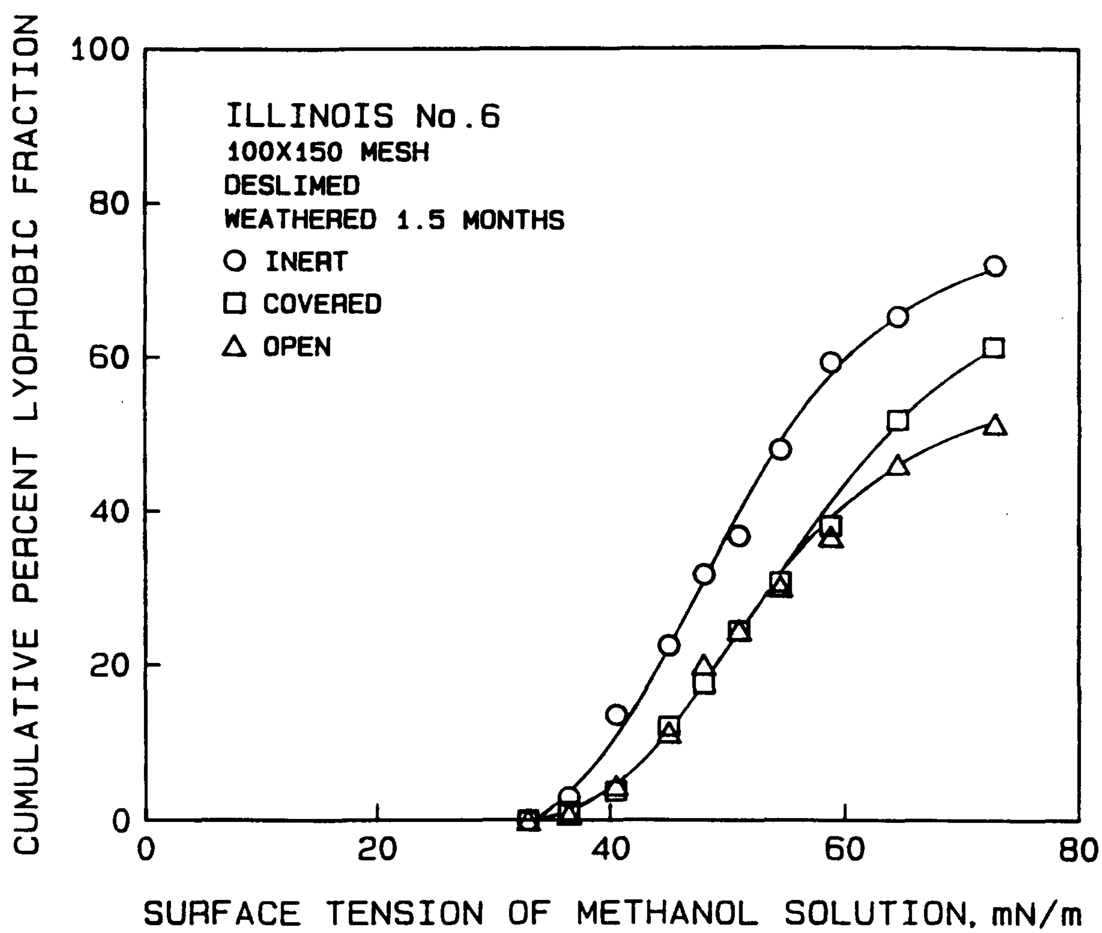

Figure 7.16 Film flotation partition curves of deslimed samples of Illinois No. 6 coal (Increment 3) samples weathered under inert, covered and open conditions.

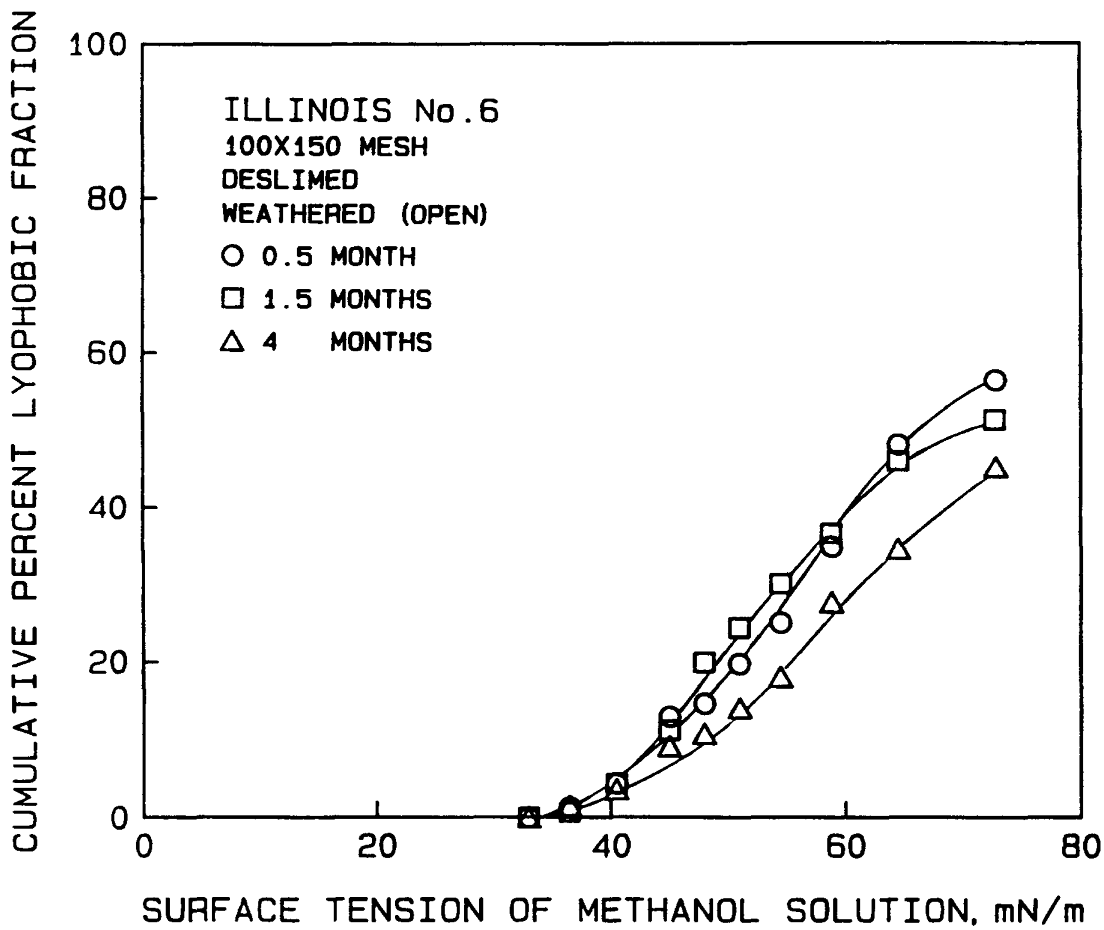

Figure 7.17 Film flotation partition curves of Illinois No. 6 coal samples weathered under open mode for 0.5, 1.5 and 4 months.

weathered samples. The samples were conditioned at different pHs for seven hours inside a glove box and an aliquot of the suspension was taken for measuring zeta potentials with the Laser Zee Meter. While the difference between the samples from different modes of storage is not significant, a marked difference in the zeta potentials of the research and weathered samples can be seen from the results given in Figure 7.18. The weathered samples exhibit more negative potentials below pH 4.5 and less negative potentials above pH 4.5 than the unweathered samples. These shifts may result from either oxidation effects or the high amount of ash-forming minerals (about 30 percent) present in the weathered samples.

The zeta potential results for the weathered samples of Pittsburgh No. 8 coal are presented in Figure 7.19. For the increments tested (up to Increment 4), there seems to be no significant difference in the zeta potentials of samples stored under the different modes. Zeta potential measurements are difficult in the case of coal because of the chemical and physical heterogeneity of the surface and any attempt to separate the mineral matter from the coal would probably alter the surface of the naturally weathered coals. Because of the complexities involved, only select coal samples subjected to prolonged weathering will be characterized using this technique.

7.1.5 Diffuse Reflectance Infrared Fourier Transform (DRIFT) Spectroscopy of Weathered Samples

Infrared techniques have been used for many years to study the effect of oxidation on the formation of different functional groups on the surface of coal particles. A variation of conventional FTIR (Fourier Transform Infrared) is the DRIFT technique, which has the advantage of using powdered samples and can be used for characterizing opaque material. In the present context DRIFT spectroscopy was carried out on some of the weathered samples of Illinois No. 6 and Pittsburgh No. 8 coal.

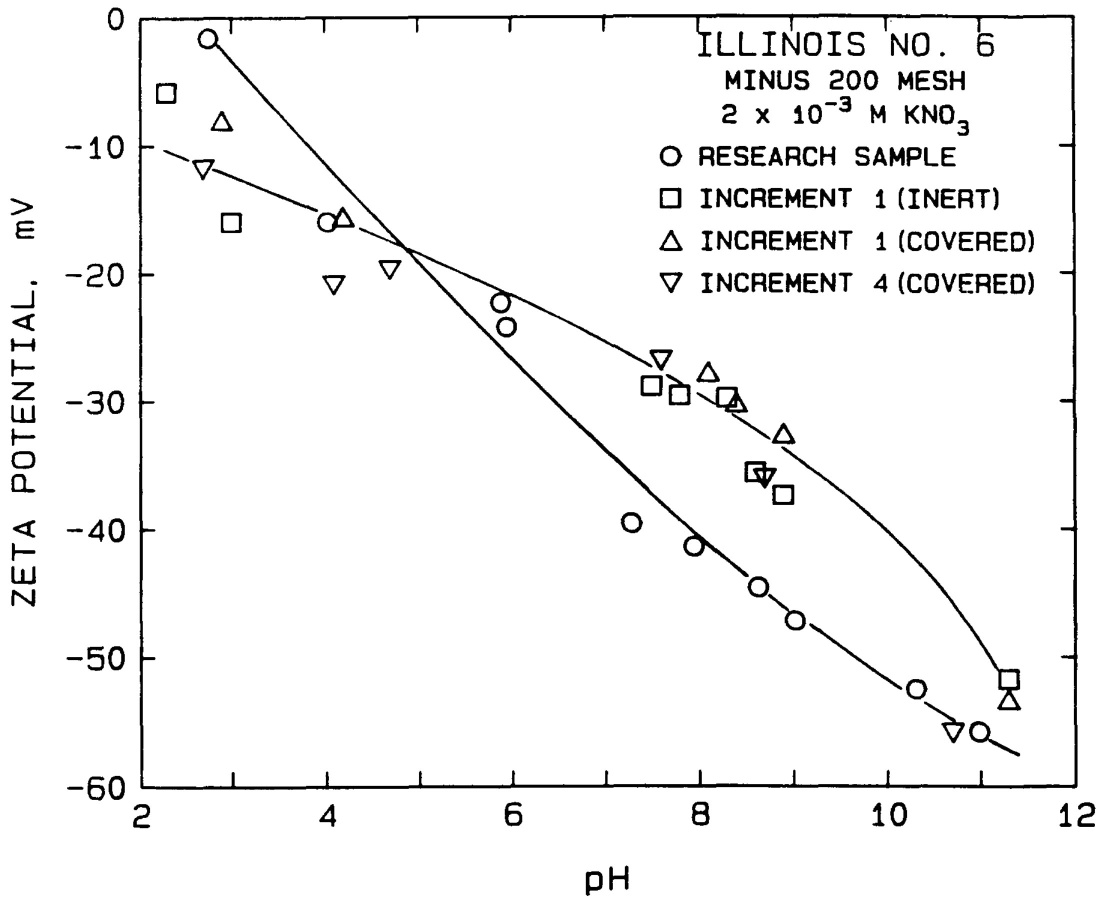

Figure 7.18 Zeta potential vs pH of Illinois No. 6 coal research and weathered samples.

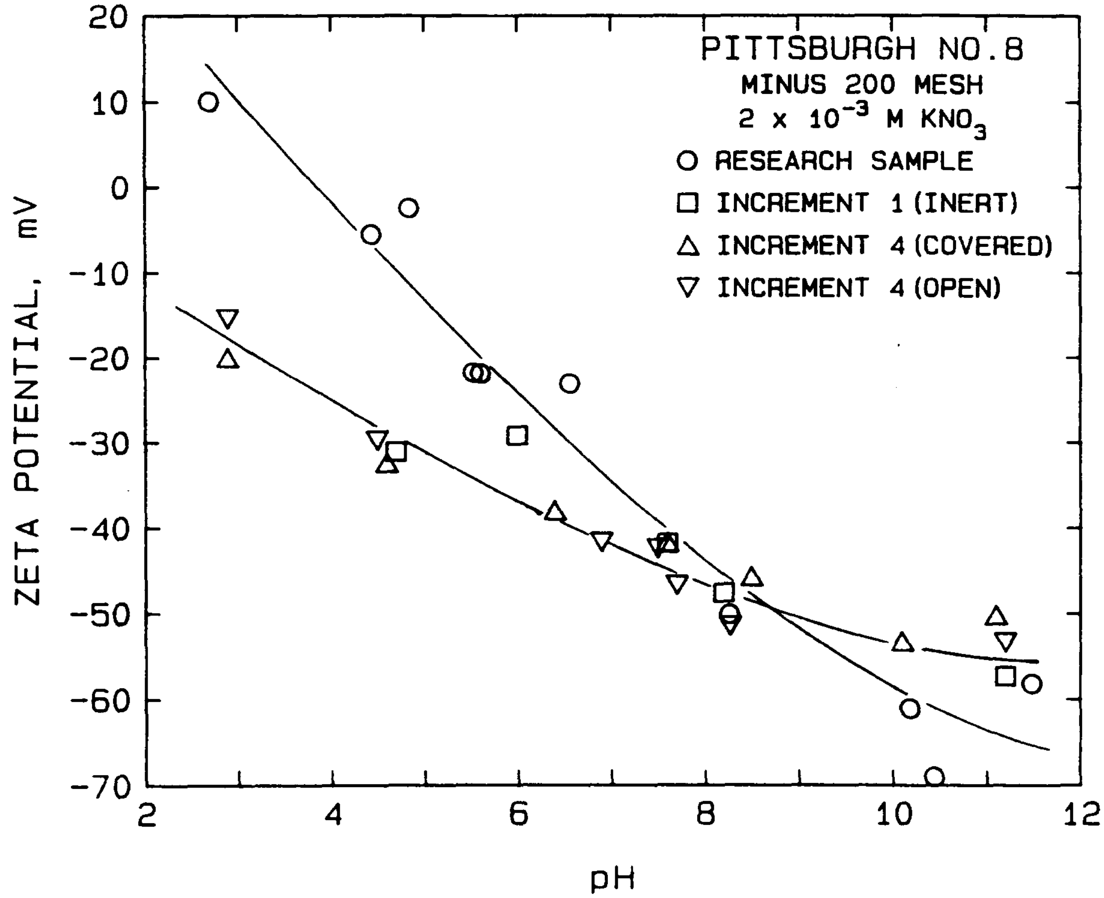

Figure 7.19 Zeta potential vs. pH for research and weathered samples of Pittsburgh No. 8 coal.

Preliminary DRIFT analysis of the minus 400 mesh fraction and 80 x 100 mesh samples showed that there is no difference between the two spectra. However, for the purpose of the current study, an 80 x 100 mesh fraction was used to enable direct comparison with wettability and microflotation studies. The diffuse reflectance spectra of different weathered samples of Illinois No. 6 and Pittsburgh No. 8 coals are presented in Figures 7.20 and 7.21. These results can be compared with the spectra of the respective research samples in Figures 7.22 and 7.23. Examination of the results shows that while the DRIFT spectra of the different weathered samples are similar, there are some differences between the spectra of the weathered samples and the research sample.

The spectra of the weathered samples of Illinois No. 6 coal (Figure 7.22) shows the presence of a peak corresponding to the carbonyl group (1655 cm^{-1}) indicating a certain degree of oxidation of even coal that had been stored under inert atmosphere. The presence of the hydroxyl group peak (3695 cm^{-1}) may be due to clays which make up the ash. Unlike Illinois No. 6 coal, the weathered sample of Pittsburgh No. 8 coal does not exhibit the presence of carbonyl groups, showing that Pittsburgh No. 8 coal is less susceptible to oxidation by weathering.

7.2 Effect of Weathering on Hallimond Tube Flotation

Microflotation tests in triply distilled water were carried out using a Hallimond tube flotation cell. The minus 28 mesh weathered sample was dry sieved to obtain 80 x 100 mesh material for these tests. The pH was maintained between 5.0 and 5.4 using HNO_3 and KOH. The test results with Illinois No. 6 and Pittsburgh No. 8 coals are given in Figures 7.24 and 7.25. Flotation response of the corresponding research samples is also shown in these figures. These figures show that the research samples give much higher flotation yields than the weathered samples, which may be ascribed to the difference in the ash content between the research and weathered samples.

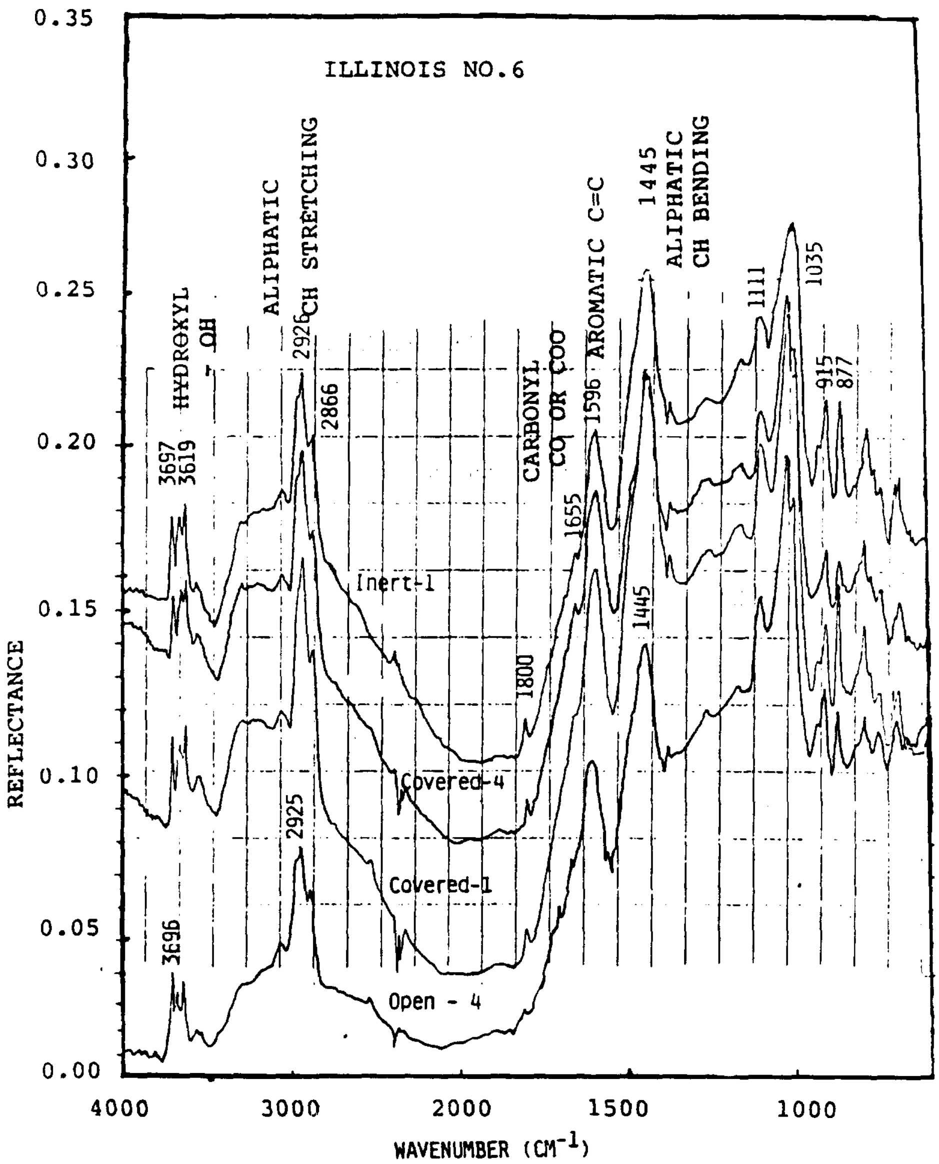

Figure 7.20 DRIFT spectrum of the weathered samples of Illinois No. 6 coal.

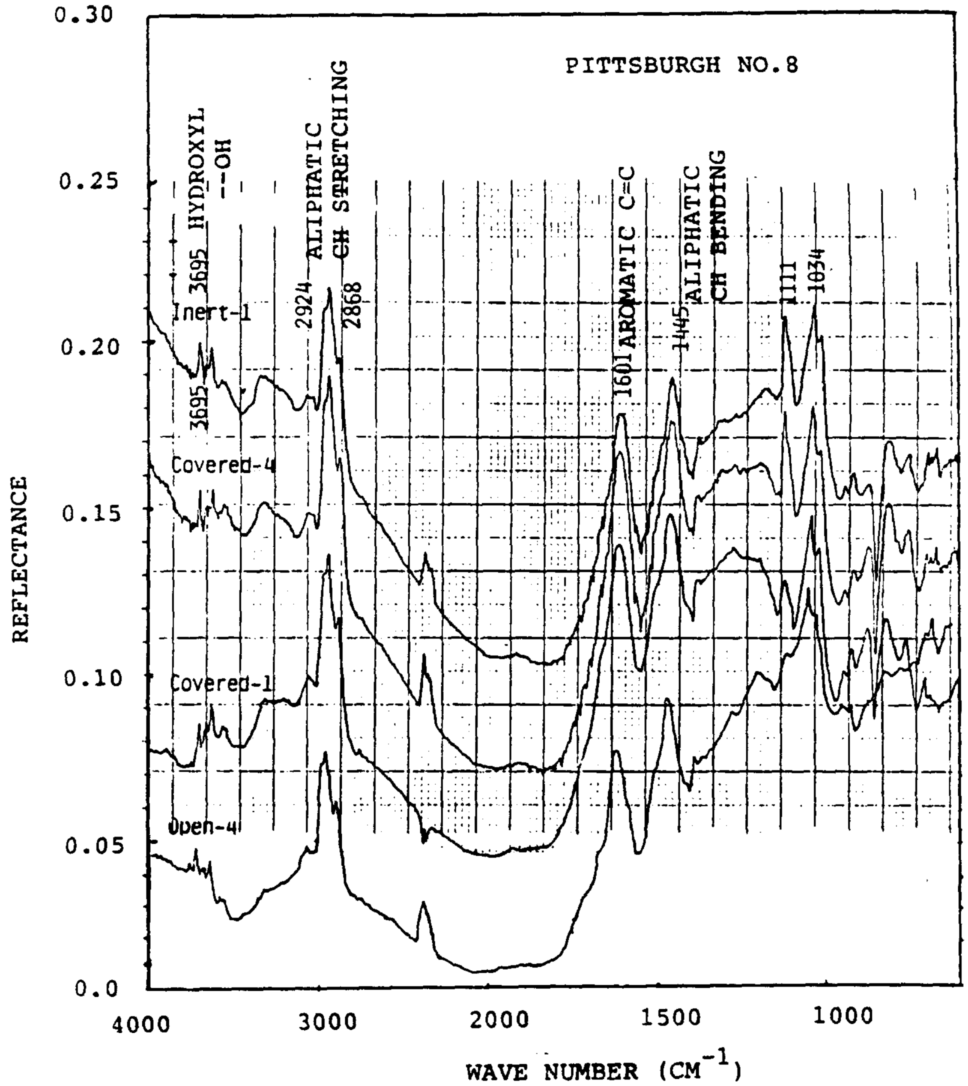

Figure 7.21 DRIFT spectrum of the weathered samples of Pittsburgh No. 8 coal.

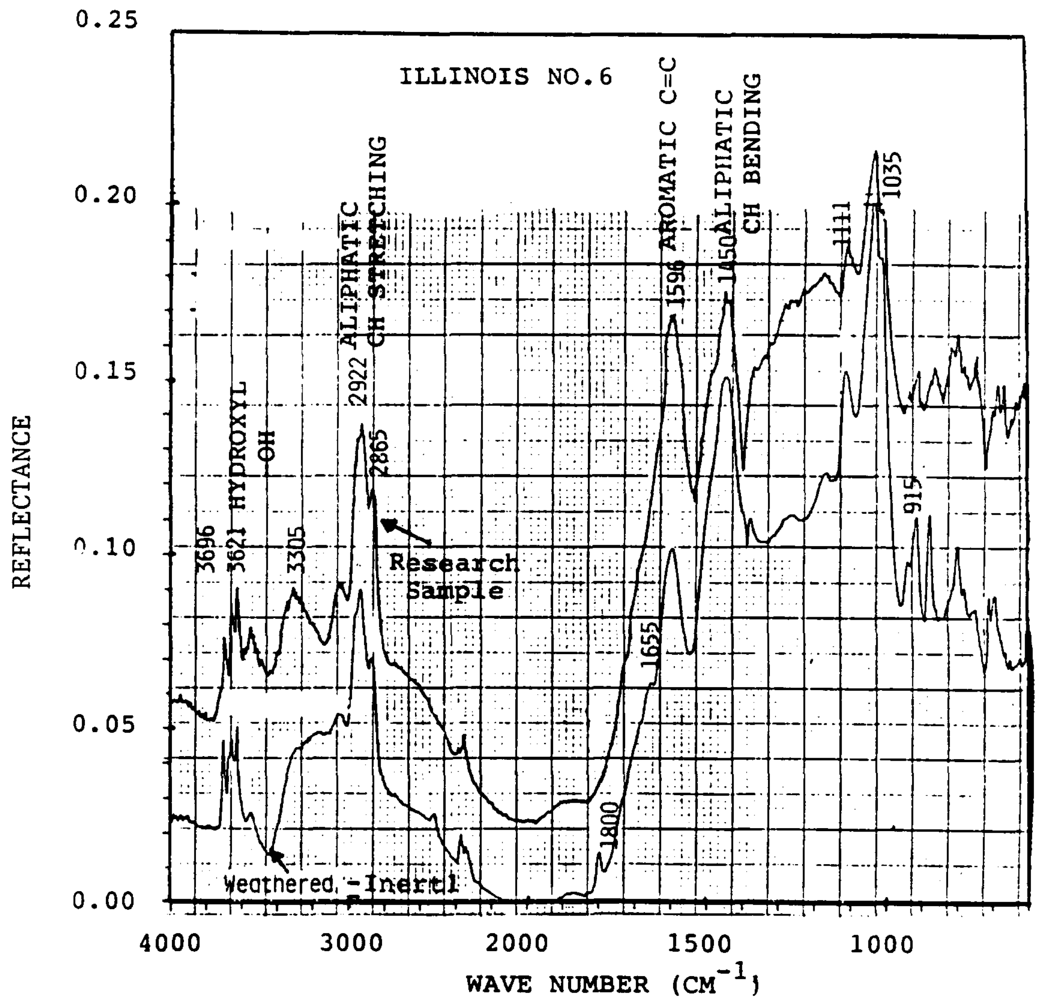

Figure 7.22 Comparison of the DRIFT spectra of the research and the weathered samples of Illinois No. 6 coal.

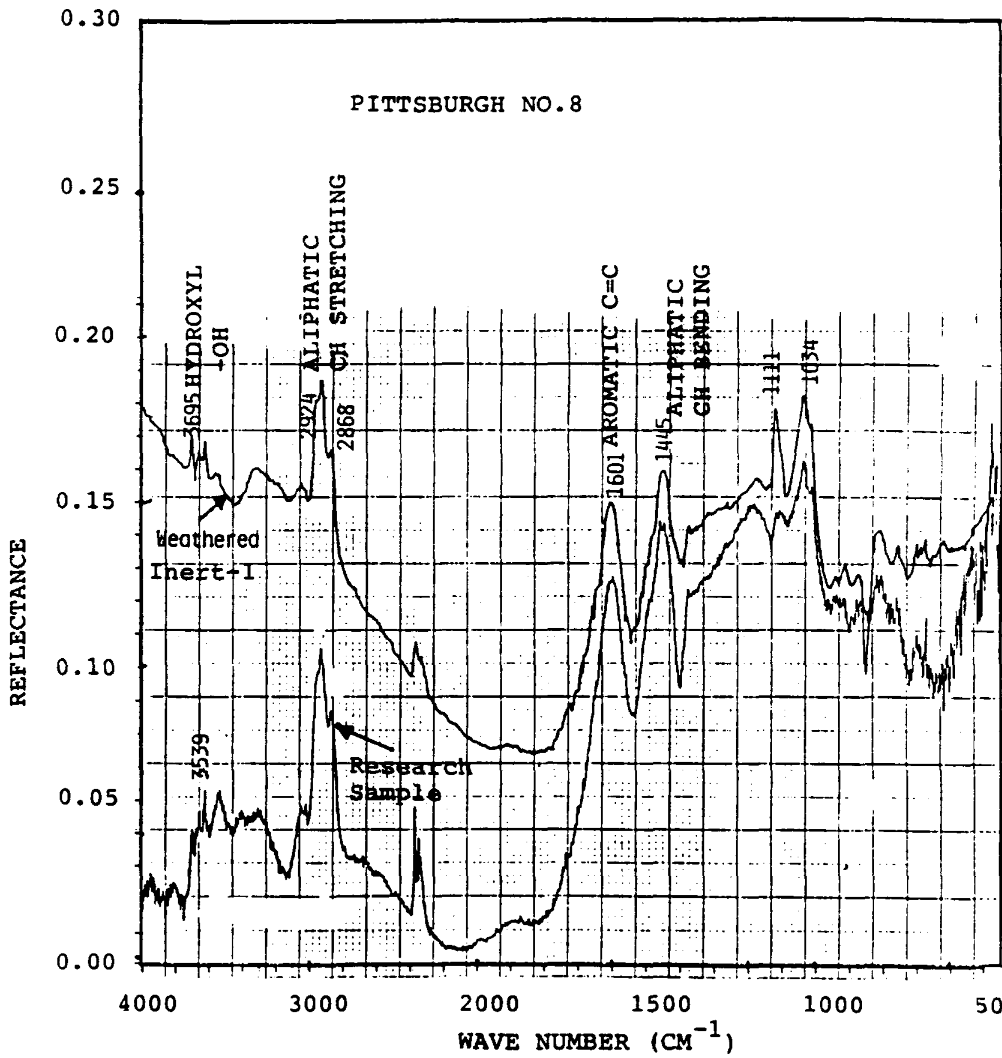

Figure 7.23 Comparison of the DRIFT spectra of the research and the weathered samples of Pittsburgh No. 8 coal.

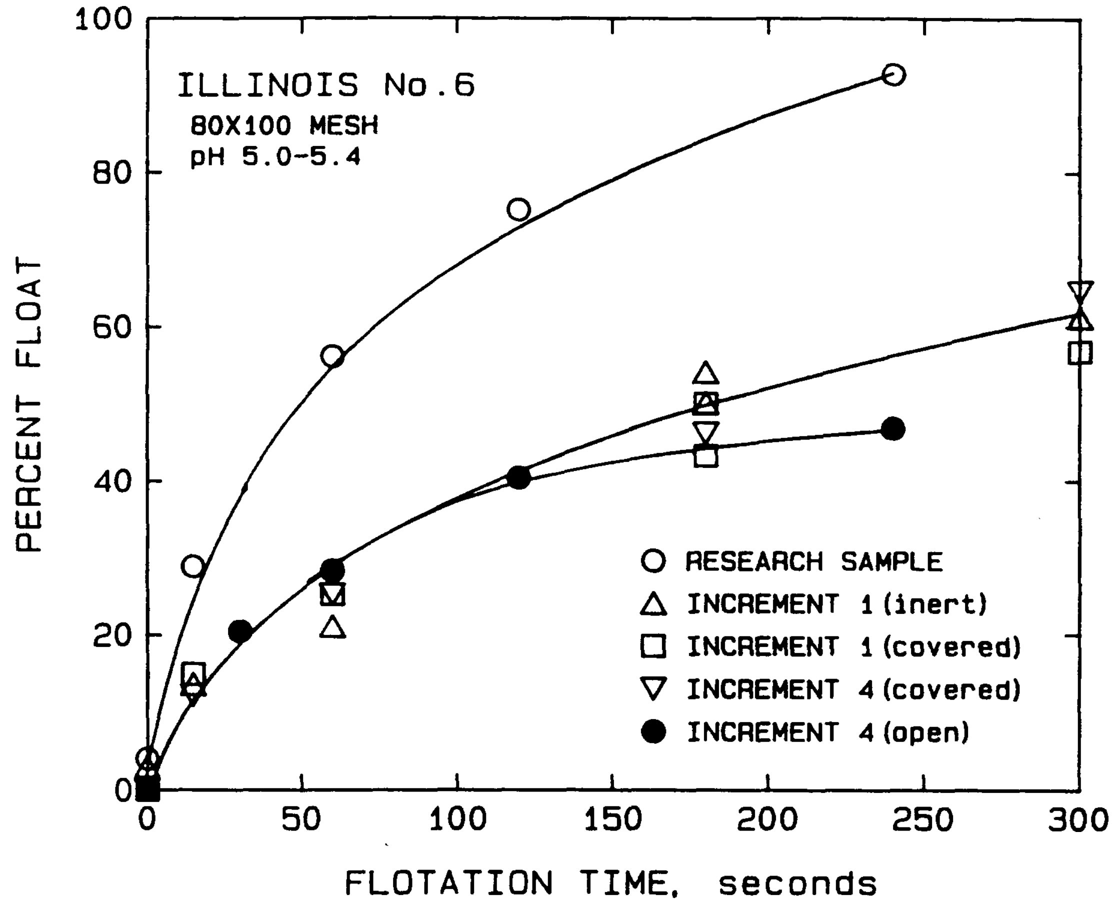

Figure 7.24 Effect of weathering on Hallimond tube flotation of Illinois No. 6 coal.

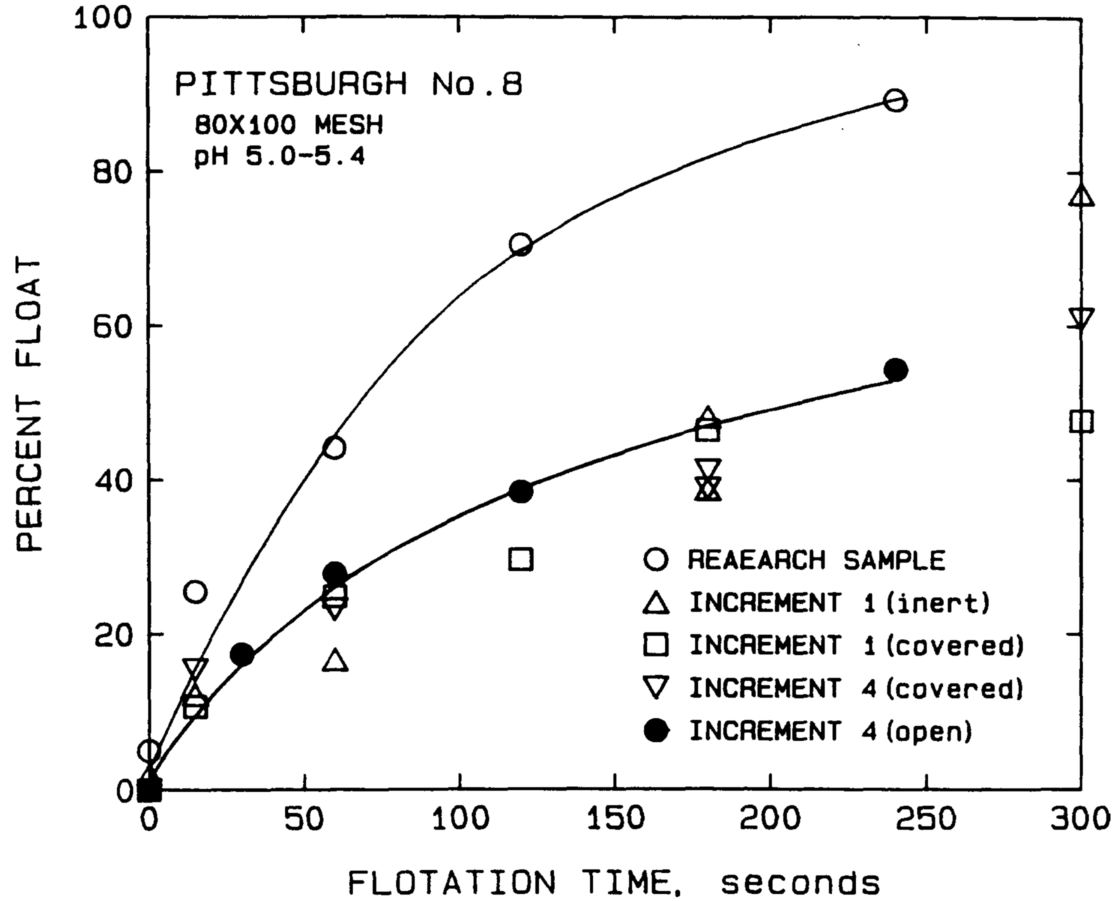

Figure 7.25 Effect of weathering on Hallimond tube flotation of Pittsburgh No. 8 coal.

However, neither the weathering time nor mode seems to affect the flotation response for the samples tested (up to Increment 4). This will be examined further by performing flotation tests of select samples of other increments.

7.3 Flotation Studies of the Weathered Samples

The main emphasis in studying the surface characteristics of the weathered samples is to delineate their behavior in surface-based separation processes such as flotation. Such an approach will facilitate a better understanding of the factors responsible for any difference in the flotation response resulting out of either the weathering mode (inert, covered and open) or the weathering time. In the case of coal, flotation response is a function of its rank, particle size distribution, mineral matter content and the state of oxidation. The Standard Flotation test procedure, established as a part of Task 4 in the Work Plan (see Section 6 of Quarterly Report No. 2), has been used to study the effect of weathering on the flotation response. The collector and frother dosages used correspond to those for the standard dry ground (95 percent passing 28 mesh) research sample of different coals.

In the last quarterly report, the results of the first five increments were discussed. It was observed that the samples stored under inert conditions gave uniformly higher yields than did the samples stored under either covered or open modes. The flotation yields decreased with weathering time irrespective of the storage mode. During in the present quarter, additional flotation experiments were carried out using the weathered samples of all three research coals.

In Figure 7.26 the flotation tests results of Illinois No. 6 coal weathered for different lengths of time are presented. The samples tested include coal weathered for up to six months (Increment 9). The results clearly show that the samples stored under an inert environment give higher yields than the samples stored either open or covered. The flotation yield of the inerted

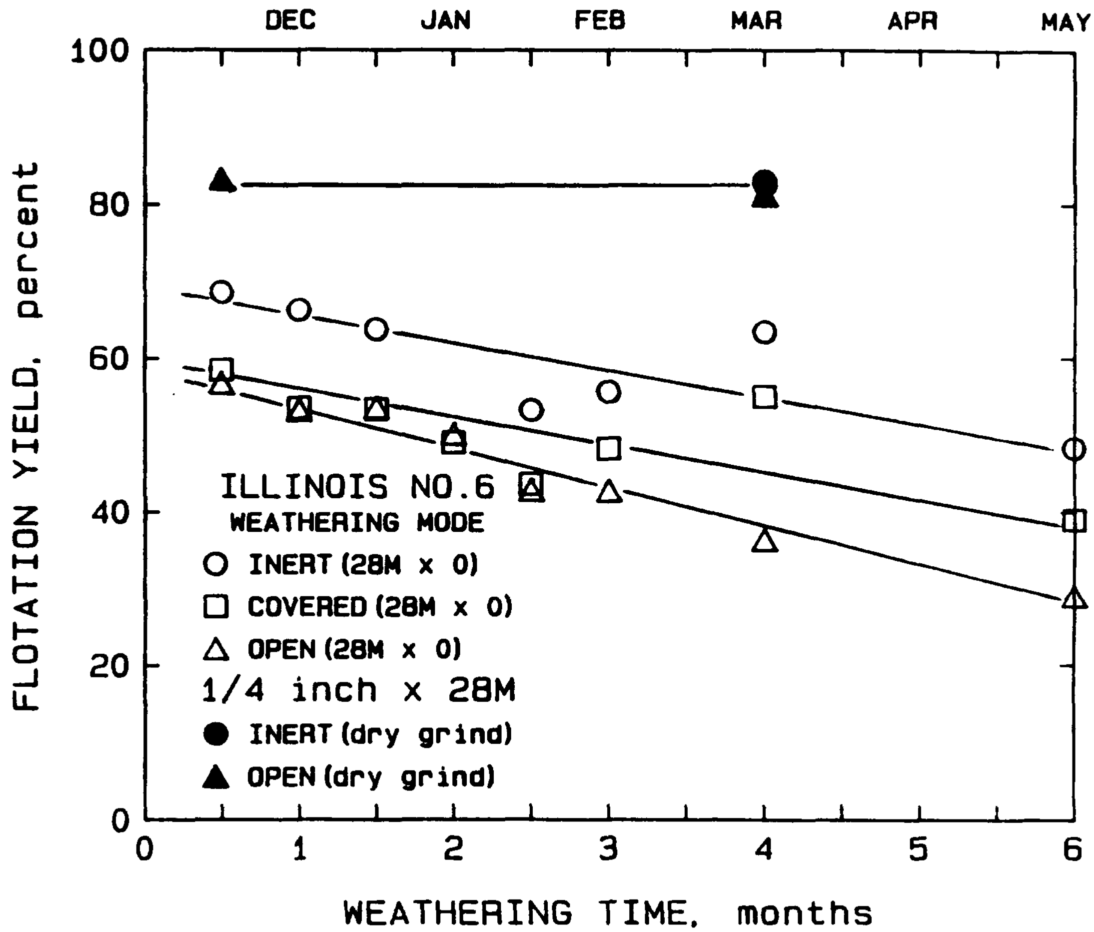

Figure 7.26 Effect of weathering time on the flotation yields of Illinois No. 6 coal stored under inert, covered and open modes.

sample was found to decrease from 68.5 percent for Increment 1 (15 days of weathering) to about 48 percent for Increment 9. This decrease with time might be ascribed to oxygen trapped in the pores of the coal, which could not have been eliminated by purging the sample in a drum with inert gas. The corresponding yields for the samples stored under open conditions decrease from about 56 percent to about 29 percent after the six months of weathering.

In comparison to the standard flotation yield obtained for the research sample (about 75 percent yield for the 28 mesh sample), the yields for the inerted weathered samples are less even for the first increment. This difference may be attributed to the higher ash content in the weathered samples (30 percent) in comparison to the research sample (17.3 percent) and the difference in the particle size distribution of the standard ground sample and the weathered samples. The effect of the difference in the ash content on the flotation behavior can be minimized by comparing the combustible recoveries of the research sample (about 79 percent) and the first increment of the inerted weathered sample (about 83 percent), which are reasonably close to each other. However, since the ash content of the weathered samples collected at different times are consistent, the effect of weathering can be delineated by comparing the flotation response of the weathered samples to the inert sample of Increment 1. Such a comparison shows that the order of the effect of weathering is, as expected, open > covered > inert. The decrease in flotation yields can be attributed to the increased hydrophilicity of the weathered coal surface as seen in the shifts of the film flotation partition curves (Figure 7.15).

To determine the effect of the particle size at which coal is weathered has on flotation, the weathered 1/4 inch x 28 mesh material from Increment 1 (open and inert) and Increment 7 were dry ground for the time used in the standard grinding procedure to produce a minus 28 mesh product to be tested under the standard flotation conditions. The results are presented in Figure 7.26 along with the results of the weathered 28 mesh x 0 material. The flotation yield is

Figure 7.26 along with the results of the weathered 28 mesh x 0 material. The flotation yield is about 82 percent irrespective of the weathering time or the storage mode. When the coal is comminuted, new surface is generated, resulting in good flotation of coarser material that had been weathered for 4 months. This aspect will be further examined by monitoring the flotation behavior of future increments.

The flotation yields of the weathered samples of Pittsburgh No. 8 coal are presented in Figure 7.27. As in the case of Illinois No. 6 coal, the inerted samples exhibit uniformly higher yields than do samples from either the covered or open modes. As would be expected, the effect of weathering time is more pronounced in the case of the open samples than for the inerted samples. The yields of the inerted material decrease slightly from 62 percent for Increment 1 to 59.5 percent for Increment 9. Whereas in the case of samples stored under open condition, the yield decreases from 55 to 27 percent over a six month period. As shown in the same figure, the flotation yields of the ground 1/4 inch x 28 mesh weathered gives a much higher yield (about 94 percent), even after five months of weathering under either the inert or open modes of storage.

Similar experiments have been completed with Upper Freeport PA coal samples. With the experience gained through testing most of the increments of the other two coals, the testing procedure was modified and only select samples were floated in this case. The flotation results of 28 mesh x 0 samples of Increments 1, 4 and 8 (inert, covered and open) and 1/4 inch x 28 mesh samples of Increments 1 and 8 (inert and open) are presented in Figure 7.28. The effect of weathering on the flotability of the open samples is greater than that for the inerted or the covered samples. There is also a large decrease on the yield of the open sample between the fifth and seventh months (corresponding to April and June) probably due to the onset of warmer temperatures. The 1/4 inch x 28 mesh samples stored under similar conditions do not indicate any weathering effect with time or mode of weathering.

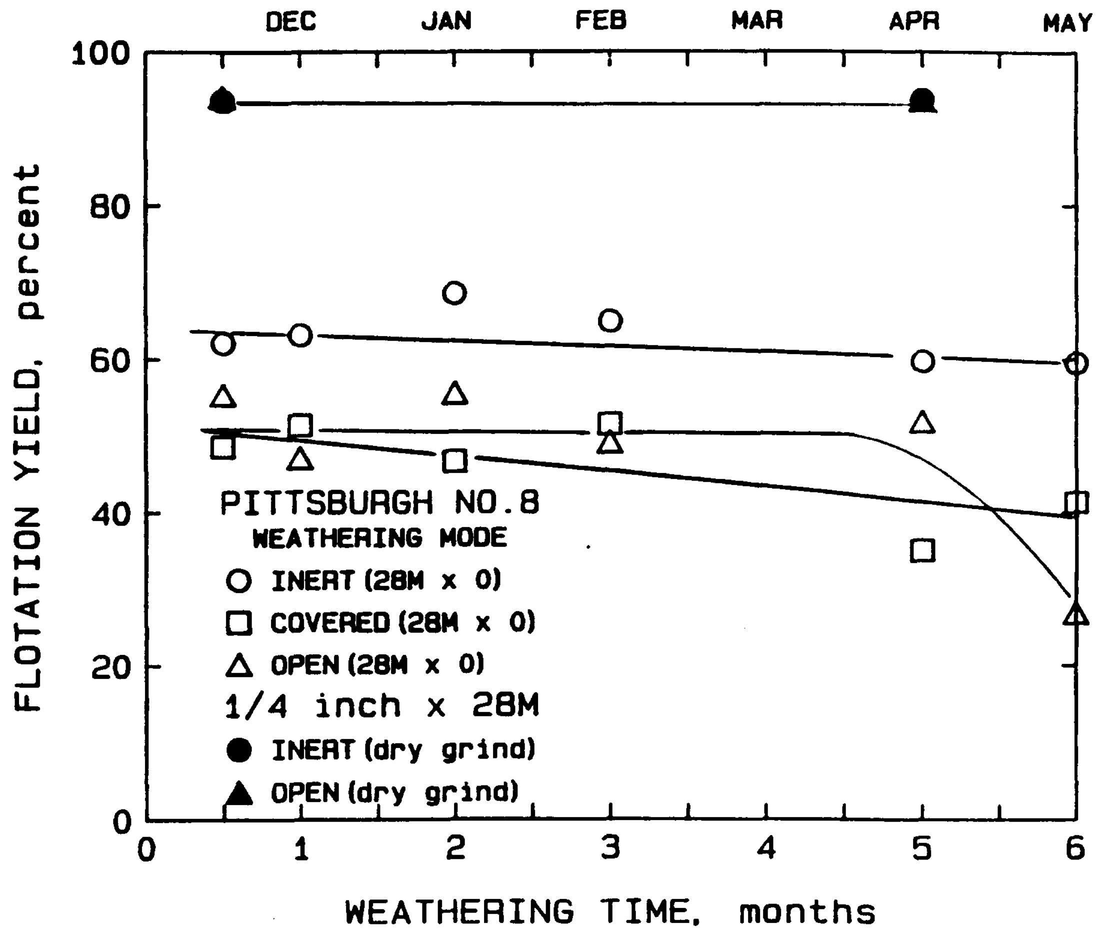

Figure 7.27 Effect of weathering time on the flotation yields of Pittsburgh No. 8 coal stored under inert, covered and open modes.

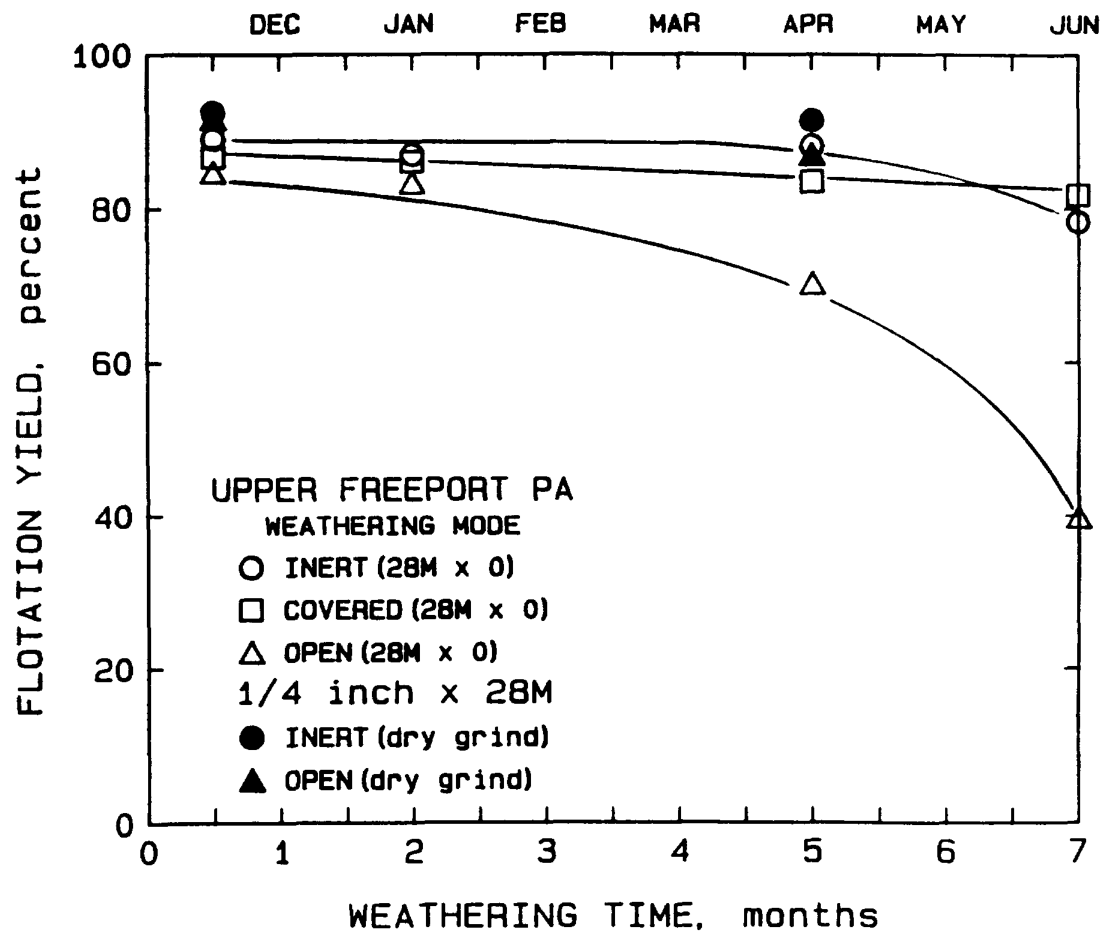

Figure 7.28 Effect of weathering time on the flotation yields of Upper Freeport PA coal stored under inert, covered and open modes.

Better insight into the comparative behavior of the three coals can be obtained by plotting the flotation results of the samples stored under the open mode. Figure 7.29 is such a plot which shows that the Upper Freeport PA coal is relatively more refractory to atmospheric oxidation than either Illinois No. 6 or Pittsburgh No. 8 coals.

In summary, weathering has a definite effect on the flotabilities of all three coals tested. Illinois No. 6 coal is more susceptible to weathering than either Pittsburgh No. 8 or Upper Freeport coals. Assessment of wettability of the weathered samples using film flotation and diffuse reflectance spectroscopy also shows similar trends. The tests done so far also indicate that the flotability of coal weathered as larger particles is not affected by the mode or the time of weathering once the material is ground. Apparently grinding the sample prior to flotation generates enough fresh surfaces that are not oxidized resulting in better flotation yields.

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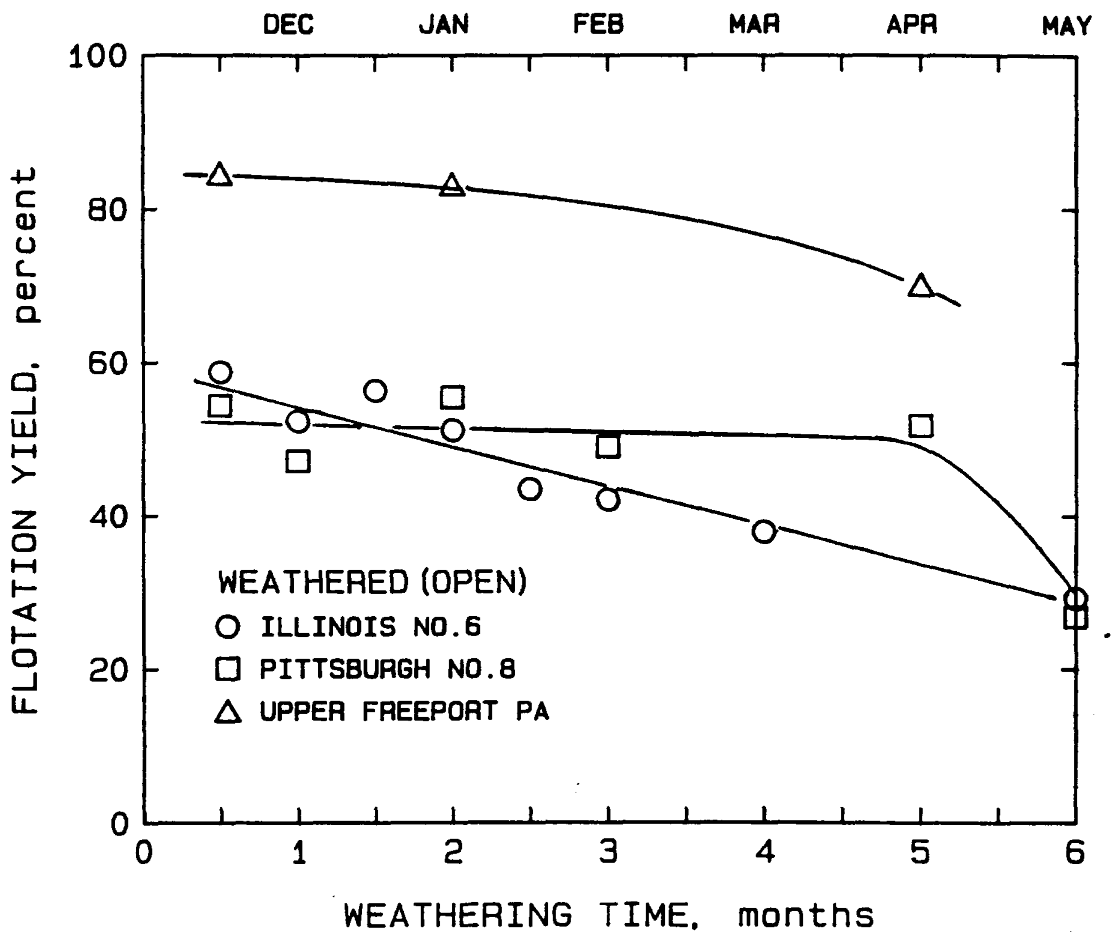

Figure 7.29 Comparison of the effect of weathering on the combustibles recovered by flotation of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals weathered under open condition.

APPENDIX

The tables given below provide tabulations of the data used in tables and figures included in the text and have identical numbering to provide additional information to the reader.

Table 2.1 Statistical Analysis of the composite values of ash and sulfur for Pittsburgh No. 8 sample.

Composite Size	Ash %	Tot. S %	Pyr. S %	Btu/lb %	200 M fines %
4" x 0	11.5	3.90	2.96	13000	1.8
1-1/2" x 0	12.0	3.91	2.94	12950	2.8
1/8" x 0	12.0	3.93	2.78	12950	5.5
28 M x 0	12.0	3.89	2.77	12850	24
200 M x 0	12.0	3.90	2.78	12750	100
Mean	11.9	3.91	2.85	12900	
Std. Dev.	0.22	0.01	0.08	91	
Variance	0.05	0.0001	0.0007	8198	
SD/Mean	0.018	0.003	0.029	0.007	
95% CI/Mean	0.27	0.013	0.102	112	

Table 2.2 Comparison of the Floats at 1.3 an specific gravity for Pittsburgh No. 8 sample.

	FLOAT AT 1.3					FLOAT AT 1.6			
Size	Wt. %	Ash %	Tot. S %	Btu/lb	Size	Wt. %	Ash %	Tot. S %	Btu/lb
4" x 0	18.3	4.34	2.12	14200	4" x 0	91.6	7.48	3.33	13700
1 1/2" x 0	31.8	4.57	2.28	14250	3/2" x 0	90.4	7.18	3.24	13800
1/8" x 0	46.4	3.34	1.85	14400	1/8" x 0	88.8	6.28	2.79	13900
28 M x 0	51.1	2.66	1.59	14400	28 M x 0	87.3	5.29	2.21	14000
200 M x 0	53.9	2.08	1.35	14500	200 M x 0	86.8	4.26	1.48	14100

Table 2.3 Statistical Analysis of Composite Values of Ash and Sulfur for Upper Freeport PA coal.

Composite Size	Ash %	Tot. S %	Pyr. S %	Btu/lb	200 M fines %
4" x 0	13.02	2.15	1.54	13450	1.76
1 1/2" x 0	12.78	2.16	1.44	13500	1.7
1/8" x 0	12.21	2.25	1.58	13600	5.12
28 M x 0	12.37	2.19	1.34	13400	21.36
200 M x 0	11.94	2.35	1.42	13500	100
Mean	12.46	2.22	1.46	13500	
Std. Dev.	0.39	0.08	0.09	71	
Variance	0.15	0.006	0.007	5112	
95% CI	0.48	0.093	0.106	89	
SD/Mean	0.031	0.034	0.059	0.005	

Table 2.4 Comparison of the floats at 1.3 and 1.6 gravity for the Upper Freeport PA sample.

Size	FLOAT AT 1.3 Wt. %	Ash %	Tot. S %	Btu/lb	Size	FLOAT AT 1.6 Wt. %	Ash %	Tot. S %	Btu/lb
4" x 0	34.3	3.62	1.23	15050	4" x 0	91.2	9.16	1.50	14100
1 1/2" x 0	35.1	3.71	1.20	15050	1 1/2" x 0	91.2	8.98	1.49	14150
1/8" x 0	42.3	3.29	1.12	15200	1/8" x 0	89.9	8.07	1.31	14350
28 M x 0	44.9	2.32	0.89	15200	28 M x 0	85.4	6.50	1.02	14500
200 M x 0	54.9	1.87	0.79	15250	200 M x 0	81.0	3.75	0.82	14600

Table 2.5 Statistical analysis of composite values of ash and sulfur for Illinois No. 6 sample.

Composite Size	Ash% %	Tot. S% %	Pyr. S% %	Btu/lb	200 M fines %
4" x 0	16.4	4.94	2.95	11850	0.3
1 1/2" x 0	15.9	4.87	2.90	11800	0.2
1/8" x 0	16.1	4.86	2.97	11750	4.2
28 M x 0	15.6	4.72	2.88	11800	6.7
200 M x 0	15.9	4.46	2.68	11800	100
Mean	16.0	4.77	2.88	11800	
Std. Dev.	0.26	0.17	0.1	35	
Variance	0.07	0.03	0.01	1207	
SD/Mean	0.017	0.036	0.036	0.003	

Table 2.6 Comparison of the floats at 1.3 and 1.6 specific gravity for the Illinois No. 6 sample.

Size	FLOAT AT 1.3 Wt. %	Ash %	Tot. S %	Btu/lb	Size	FLOAT AT 1.6 Wt. %	Ash %	Tot. S %	Btu/lb
4" x 0	20.7	4.40	2.98	13800	4" x 0	87.2	10.70	3.71	12780
1 1/2" x 0	22.0	4.10	2.92	13730	1 1/2" x 0	87.5	10.44	3.59	12690
1/8" x 0	34.5	3.34	2.75	13820	1/8" x 0	81.5	8.63	3.14	13010
28 M x 0	1.8	1.31	2.45	14170	28 M x 0	84.9	8.35	3.02	13010
200 M x 0	5.1	1.89	2.50	14120	200 M x 0	82.3	6.87	2.48	13320
200 M x 0*	34.5	2.85	2.38		200 M x 0*	81.2	6.26	2.41	

* Results of QA/QC Test done at Gould Energy Laboratories

Table 2.7 Comparison of proximate and sulfur analyses of base coals.

Coal	Moisture %	PROXIMATE ANALYSIS (DRY BASIS) V. Matter %	F. Carbon %	Ash %	Tot. S %
WASHABILITY SAMPLE					
Illinois No. 6	6.34	36.1	46.0	17.9	5.81
Pittsburgh No. 8	1.89	35.1	52.6	12.3	4.15
Upper Freeport PA	0.82	25.4	62.1	12.5	2.29
RESEARCH SAMPLE PREPARED AT UTAH					
Illinois No. 6	4.23	36.0	47.4	16.6	5.27
Pittsburgh No. 8	2.03	36.1	53.0	10.8	4.19
Upper Freeport PA	0.94	26.2	61.4	12.4	2.23
RESEARCH SAMPLE PREPARED AT BERKELEY					
Illinois No. 6	9.5	36.2	46.3	17.5	5.73
Pittsburgh No. 8	2.32	35.7	52.5	11.8	4.28
Upper Freeport PA	1.0	26.2	61.8	12.0	2.38

Table 2.8 Comparison of the elemental analyses* of the base coals.

Coal	Moisture %	ELEMENTAL ANALYSIS (DRY BASIS) Carbon %	Hydrogen %	Nitrogen %	Sulfur %	Oxygen %
WASHABILITY SAMPLE						
Illinois No. 6	6.34	63.9	5.29	1.01	5.81	6.07
Pittsburgh No. 8	1.89	71.0	5.01	1.23	4.15	
Upper Freeport PA	0.82	76.1	4.76	1.34	2.29	3.00
RESEARCH SAMPLE PREPARED AT UTAH						
Illinois No. 6	4.23	63.9	5.04	1.39	5.27	7.88
Pittsburgh No. 8	2.03	72.4	5.06	1.47	4.19	6.07
Upper Freeport PA	0.94	76.3	4.66	1.45	2.23	3.02
RESEARCH SAMPLE PREPARED AT BERKELEY						
Illinois No. 6	9.5	63.8	5.65	1.24	5.73	6.10
Pittsburgh No. 8	2.23	71.0	5.12	1.45	4.28	6.40
Upper Freeport PA	1.00	75.6	4.70	1.45	2.38	3.85

* The analysis work was done at Berkeley

Table 2.9 Analysis of Select sink-float fractions.

Grind	Size Fraction	Gravity Fraction	ORIGINAL DATA Ash %	Tot. S %	Pyr. S %	QA/QC DATA Ash %	Tot. S %	Pyr. S %
ILLINOIS NO. 6								
28 M	28 x 200 M	1.60 x 1.80	35.8	5.78	4.27	35.8	6.19	5.98
28 M	200 M x 0	1.40 x 1.60	11.7	2.54	0.77	11.7	2.42	0.69
28 M	200 M x 0	+1.80	60.1	16.5	15.66	62.9	16.97	15.05
PITTSBURGH NO. 8								
28 M	28 x 200 M	-1.30	2.8	1.66	0.4	2.9	1.8	0.33
28 M	28 x 200 M	1.40 x 1.60	17.7	5.78	4.66	17.4	6.6	4.43
28 M	200 M x 0	1.35 x 1.40	4.8	1.46	0.36	4.7	1.66	0.34
UPPER FREEPORT PA								
28 M	28 x 200 M	-1.30	2.9	0.92	0.61	3.1	0.96	0.20
28 M	200 M x 0	1.35 x 1.40	4.4	0.79	0.48	4.5	0.83	0.09
200 M	200 M x 0	1.35 x 1.40	5.9	0.90	0.14	4.3	0.85	0.08

Table 2.10 Ash and total sulfur of 28 M x 0 screened fraction of Pittsburgh No. 8 weathering samples reconstituted from flotation product analysis.

Incr. No.	Date	INERT Ash %	Tot. S %	COVERED Ash %	Tot. S %	OPEN Ash %	Tot. S %
1	11/22/88	17.19	6.42	17.14	6.37	17.13	6.21
2	12/07/88	16.92	6.04	NA	NA	17.91	5.92
4	01/05/89	17.12	6.65	17.18	6.81	18.06	6.63
6	02/10/89	17.63	6.53	16.99	5.96	17.7	6.30
8	04/06/89	17.42	5.86	17.10	6.55	17.31	6.43
Mean		17.26	6.3	17.09	6.42	17.62	6.3
Std. Dev.		0.25	0.3	0.07	0.31	0.35	0.24

Table 2.11 Ash and total sulfur of 28 M x 0 screened fraction of Illinois No. 6 weathering samples reconstituted from flotation product analysis.

Incr. No.	Date	INERT Ash %	Tot. S %	COVERED Ash %	Tot. S %	OPEN Ash %	Tot. S %
1	12/07/88	26.93	4.98			26.72	4.84
2	12/20/88	28.11	4.48			30.48	4.90
3	01/04/89	27.67	4.95			30.02	4.62
4	01/17/89	27.65	5.02			26.87	5.14
5	01/31/89	28.05	4.56			28.07	4.74
6	02/14/89	28.48	4.94				
7	03/13/89	24.17	4.97			29.36	4.79
Mean		27.3	4.84			28.6	4.84
Std. Dev.		1.35	0.21			1.46	0.16

Table 2.12 Ash and total sulfur of 28 M x 0 screened fraction of Upper Freeport PA weathering samples reconstituted from flotation product analysis

Incr. No.	Date	INERT Ash %	INERT Tot. S %	COVERED Ash %	COVERED Tot. S %	OPEN Ash %	OPEN Tot. S %
1	11/30/88	9.10	3.25	9.82	3.45	9.46	3.44
4	01/16/89	9.36	2.83	9.48	2.90	9.85	2.82
8	04/07/89	9.43	2.88	9.36	3.11	9.68	2.85
Mean		9.29	2.94	9.54	3.15	9.67	3.04
Std. Dev.		0.14	0.23	0.21	0.23	0.17	0.29

Table 3.1 Air/water advancing contact angles (in degrees) on the three base coals measured by the sessile-drop method on surfaces prepared by by different methods.

Coal sample	Dry-polished in Argon	Wet-polished in Air	Pellet
Illinois No. 6	70	44	41
Pittsburgh No. 8	82	60	66
Upper Freeport PA	81	62	69

Table 3.2 Air/water contact angles (in degrees) on the three base coals measured by the captive-bubble method on surfaces wet-polished in air.

Coal Sample	Advancing	Receding	Equilibrium
Illinois No. 6	42	23	25
Pittsburgh No. 8	61	45	49
Upper Freeport PA	61	42	50

Table 3.3 Air/water contact angles (in degrees) on the three base coals calculated from film flotation results and advancing contact angles measured by the sessile-drop and the captive-bubble methods on on surfaces wet-polished in air.

Coal Sample	Film Flotation	Sessile-Drop	Captive-Bubble
Illinois No. 6	52	44	42
Pittsburgh No. 8	63	60	61
Upper Freeport PA	63	62	61

Table 3.4 The change in contact angle (in degrees) of the three base coals as a function of the time after placing a water drop on a pellet surface (using the sessile-drop method)

Coal Sample	ELAPSED TIME, MINUTES								
	0	0.5	1	2	5	10	15	25	40
Illinois No. 6	46	32	24	7					
Pittsburgh No. 8	68	-	66	65	62	59	49	41	15
Upper Freeport PA	71	69	-	68	65	55	47	32	10

Table 4.1 Reproducibility of standard flotation tests of Pittsburgh No. 8 coal.

	Institution	Yield %	Ash %	Pyr. S %
28 Mesh, Dry	Columbia	81.3	6.9	1.99
	Berkeley			
	Utah	84.3	6.3	1.48
28 Mesh, Wet	Columbia	86.5	7.5	2.09
	Berkeley	89.8	8.0	
	Utah	85.3	6.6	1.57
200 Mesh, Dry	Columbia	75.6	6.0	1.37
	Berkeley			
	Utah	75.0	6.3	1.22
200 Mesh, Wet	Columbia	76.1	4.9	1.2
	Berkeley	77.8	5.8	1.2
	Utah	74.7	5.1	1.16

Table 4.2 Reproducibility of standard flotation tests of Upper Freeport PA coal

	Institution	Yield %	Ash %	Pyr. S %
28 Mesh, Dry	Utah	81.8	6.7	0.69
	Columbia	79.8	7.4	0.68
	Berkeley			
28 Mesh, Wet	Utah	75.5	6.4	0.63
	Columbia	79.5	7.5	0.77
	Berkeley	89.3	7.8	
200 Mesh, Dry	Utah	59.1	6.3	0.67
	Columbia	67.1	7.3	0.63
	Berkeley	74.1	7.2	
200 Mesh, Wet	Utah	68.2	5.3	0.51
	Columbia			
	Berkeley	81.3	6.5	0.67

Table 4.3 Reproducibility of standard flotation tests of Illinois No. 6 coal.

	Institution	Yield %	Ash %	Pyr. S %
28 Mesh, Dry	Berkeley	74.6	9.6	1.77
	Utah	75.0	9.1	1.03
	Columbia			
28 Mesh, Wet	Berkeley	88.0	11.0	1.94
	Utah			
	Columbia			
200 Mesh, Dry	Berkeley	60.4	8.8	1.36
	Utah			
	Columbia			
200 Mesh, Wet	Berkeley	77.0	8.5	1.57
	Utah			
	Columbia			

Table 4.4 Comparison between dodecane and kerosene as collectors in the flotation of wet ground 200 mesh coal

Coal	Collector	Yield, %
Illinois No. 6	Dodecane	74.6
	Dodecane	75.1
	Kerosene	80.3
	Kerosene	80.5
Pittsburgh No. 8	Dodecane	75.9
	Dodecane	77.6
	Kerosene	81.5
	Kerosene	78.4

Table 4.5 Flotation results of wet-ground Pittsburgh No. 8 coal obtained from Babcock and Wilcox (Kaiser Engineers) and the University of California.

		FLOTATION PRODUCT ANALYSIS		
Sample Source	Grind Size	Yield %	Ash %	Tot. S %
Kaiser	200 M	81.8	5.6	2.7
Berkeley		78.2	5.7	2.5
Kaiser	28 M	89.5	7.7	3.3
Berkeley		89.6	5.7	2.5

Table 5.2 Relative Grindabilities of the three base coals.

DRY GRIND	28 MESH	200 MESH
Illinois No. 6	1.0	1.0
Pittsburgh No. 8	1.7	1.6
Upper Freeport PA	2.8	2.7

WET GRIND	28 MESH	200 MESH
Illinois No. 6	2.4	3.2
Pittsburgh No. 8	2.7	2.9
Upper Freeport PA	4.6	4.2

Table 5.3 The effect of grinding atmosphere and flotation gas composition on the yields of 200 mesh feed.

	Grind. Method	AG/AF	AG/IF	IG/AF	IG/IF
Illinois No. 6	Dry	60.4	56.5		54.5
	Wet	75.3	78.3	81.3	82.6
Pittsburgh No. 8	Dry	86.9	85.9	77.8	80.8
	Wet	75.3	74.8	74.8	75.5
Upper Freeport PA	Dry	63.5	65.4	67.9	67.5
	Wet	75.0	75.9	75.4	74.9

AG/AF = air grind, air float
AG/IF = air grind, inert float

Table 5.4 Comparison of results of QA/QC tests of the standard grinding procedure for Illinois No. 6 coal.

Site	Grind	Grind. Method	Grind. Time	Percent Passing
Berkeley	28 M	Dry	8 min.	95.8
Columbia	28 M	Dry	8 min.	
Utah	28 M	Dry	8 min.	
Berkeley	28 M	Wet	3 min. 20 sec.	95.0
Columbia	28 M	Wet	3 min. 20 sec.	
Utah	28 M	Wet	3 min. 20 sec.	
Berkeley	200 M	Dry	48 min.	95.9
Columbia	200 M	Dry	48 min.	
Utah	200 M	Dry	48 min.	
Berkeley	200 M	Wet	15 min.	95.5
Columbia	200 M	Wet	15 min.	93.6
Utah	200 M	Wet	15 min.	

Table 5.5 Comparison of results of QA/QC tests of the standard grinding procedure for Pittsburgh No. 8 coal.

Site	Grind	Grind. Method	Grinding Time	Percent Passing
Berkeley	28 M	Dry	4 min. 50 sec.	95.8
Columbia	28 M	Dry	4 min. 50 sec.	95.0
Utah	28 M	Dry	4 min. 50 sec.	
Berkeley	28 M	Wet	3 min.	96.1
Columbia	28 M	Wet	3 min.	96.0
Utah	28 M	Wet	3 min.	
Berkeley	200 M	Dry	31 min.	96.0
Columbia	200 M	Dry	31 min.	94.0
Utah	200 M	Dry	31 min.	
Berkeley	200 M	Wet	18 min.	97.5
Columbia	200 M	Wet	18 min.	96.0
Utah	200 M	Wet	18 min.	

Table 5.6 Comparison of results of QA/QC tests of the standard grinding procedure for Upper Freeport PA coal.

Site	Grind	Grind. Method	Grinding Time	Percent Passing
Berkeley	28 M	Dry	3 min. 15 sec.	97.3
Columbia	28 M	Dry	3 min. 15 sec.	96.7
Utah	28 M	Dry	3 min. 15 sec.	95.0
Berkeley	28 M	Wet	1 min. 55 sec.	97.7
Columbia	28 M	Wet	1 min. 55 sec.	95.4
Utah	28 M	Wet	1 min. 55 sec.	95.0
Berkeley	200 M	Dry	21 min. 30 sec.	92.9
Columbia	200 M	Dry	21 min. 30 sec.	92.8
Utah	200 M	Dry	21 min. 30 sec.	95.0
Berkeley	200 M	Wet	11 min. 20 sec.	95.5
Columbia	200 M	Wet	11 min. 20 sec.	92.0
Utah	200 M	Wet	11 min. 20 sec.	94.9

Table 6.1 Effect of Surface modifier (2,n- butyl thiophene) addition on the flotation performance of wet 28 mesh grind from Pittsburgh No. 8 coal at pH 6.

REAGENT DOSAGE			FLOTATION PRODUCT ANALYSIS			PERCENT REJ.		
Coll. lb/T	Frother lb/T	Mod. lb/T	Yield %	Ash %	Pyr. S %	Ash %	Pyr. S %	EI
2.1	0.43	0.0	86.5	7.5	2.09	46.0	33.0	23*
2.1	0.43	0.0	84.3	6.6	2.01	53.6	37.2	27
2.1	0.43	0.7	88.1	7.7	1.45	43.5	52.7	45
2.1	0.43	1.4	88.5	7.9	1.7	41.7	44.3	37
0.65	0.43	1.4	85.6	7.0	1.58	50.1	49.9	40

* Standard flotation test; pH - 3.4

Table 6.2 Effect of Surface modifier (2,n- butyl thiophene) addition on the flotation performance of wet 200 mesh grind from Pittsburgh No. 8 coal at pH 6.

REAGENT DOSAGE			FLOTATION PRODUCT ANALYSIS			PERCENT REJ.		
Coll. lb/T	Froth. lb/T	Mod. lb/T	Yield %	Ash %	Pyr. S %	Ash %	Pyr. S %	EI
2.1	0.3	0.0	77.5	4.9	1.2	68.3	65.5	48.0
2.1	0.3	0.0	66.5	4.8	0.81	73.4	80.0	52.0
2.1	0.3	0.7	57.7	4.7	0.87	71.4	76.5	56.0
1.7	0.3	1.4	71.5	4.9	0.87	72.6	79.6	55.0

* standard flotation test; pH = 4.3

Table 6.3 Effect of Surface modifier (Aerosol OT) addition on the flotation performance of wet 200 mesh grind from Pittsburgh No. 8 coal at pH 6.

REAGENT DOSAGE			FLOTATION PRODUCT ANALYSIS			PERCENT REJ.		
Coll. lb/T	Froth. lb/T	Mod. lb/T	Yield %	Ash %	Pyr. S %	Ash %	Pyr. S %	EI
2.1	0.3	0.0	77.5	4.9	1.2	68.3	65.5	48.0
2.1	0.3	0.0	66.5	4.8	0.81	73.4	80.1	52.0
2.1	0.3	0.18	73.1	5.0	1.0	69.5	72.9	52.0
2.1	0.3	0.35	82.8	5.7	1.15	60.7	64.7	53.0
2.1	0.3	0.7	83.6	5.6	0.97	61.0	70.0	60.0
1.2	0.3	0.7	83.6	5.6	0.97	61.0	70.0	60.0

Table 6.4 Effect of Surface modifier (Aerosol OT) addition on the flotation performance of wet 200 mesh ground Upper Freeport PA coal.

REAGENT DOSAGE			FLOTATION PRODUCT ANALYSIS			PERCENT REJ.		
Coll. lb/T	Froth. lb/T	Mod. lb/T	Yield %	Ash %	Pyr. S %	Ash %	Pyr. S %	EI
0.26	0.23	0.00	69.5	5.3	0.5	69.0	73.0	49.0
0.26	0.23	0.05	76.9	6.2	0.55	63.3	70.0	54.0
0.00	0.23	0.05	68.7	5.8	0.47	69.3	75.0	49.0
0.26	0.23	0.09	83.3	7.3	0.64	53.5	58.4	47.0
0.00	0.23	0.09	78.5	6.6	0.58	60.1	65.0	49.0

Table 6.5 Effect of surface modifier (organic monomer) addition flotation performance of dry 200 mesh grind from Illinois No. 6 coal.

SURFACE MODIFIER	REAGENT DOSAGE			FLOTATION PRODUCT ANALYSIS		
	Coll. lb/T	Froth. lb/T	Mod. lb/T	Yield %	Ash %	Tot. S %
None	6.3	1.3	0.0	59.1	8.9	3.4
Styrene	6.3	1.3	3.2	62.8	8.7	3.7
	6.3	1.3	6.4	67.3	8.6	3.7
Ethyl Benzene	6.3	1.3	3.2	63.2	8.3	3.6
	6.3	1.3	6.4	65.5	9.1	3.6
Vinyl Acetate	6.3	1.3	4.8	62.9	8.9	3.7
Ethyl Acetate	6.3	1.3	4.8	67.6	8.8	3.6

Table 6.6 Effect of Surface modifier (organic monomer) addition on the flotation performance of 200 mesh grind from Pittsburgh No. 8 coal.

SURFACE MODIFIER	REAGENT DOSAGE			FLOTATION PRODUCT ANALYSIS		
	Coll. lb/T	Froth. lb/T	Mod. lb/T	Yield %	Ash %	Tot. S %
None	2.1	0.3	0.0	77.4	6.5	2.59
Styrene	2.1	0.3	4.8	83.7	6.6	2.78
Ethyl Benzene	2.1	0.3	4.8	84.5	7.0	2.85
Vinyl Acetate	2.1	1.3	4.8	84.9	6.7	2.78

Table 6.7 Effect of surface modifier (Organic monomer) addition on the flotation performance of dry 200 mesh grind from Upper Freeport PA coal.

SURFACE MODIFIER	REAGENT DOSAGE			FLOTATION PRODUCT ANALYSIS		
	Coll. lb/T	Froth. lb/T	Mod. lb/T	Yield %	Ash %	Tot. S %
None	0.52	0.23		74.1	7.2	1.41
Styrene	0.52	0.23	4.8	74.7	7.1	1.49
Ethyl Benzene	0.52	0.23	4.8	74.0	7.1	1.38
Vinyl Acetate	0.52	0.23	4.8	74.0	7.2	1.44

Table 7.1 Proximate Analysis of weathered samples of Illinois No. 6 coal

Incr.	Size	Storage Mode	Moisture	(ON DRY BASIS %) Volatile Matter	Fixed Carbon	Ash
1	28 M x 0	Inert	2.83	34.0	37.2	28.8
	28 M x 0	Covered	3.10	33.7	37.6	28.7
	28 M x 0	Open	3.11	33.8	38.0	28.2
2	28 M x 0	Inert	3.11	33.1	37.1	29.8
	28 M x 0	Covered	3.26	33.1	37.4	29.5
	28 M x 0	Open	3.16	32.1	36.0	31.9
3	28 M x 0	Inert	3.16	33.6	37.6	28.8
	28 M x 0	Covered	3.32	33.5	37.3	29.2
	28 M x 0	Open	2.97	32.0	35.0	33.0
4	28 M x 0	Inert	3.06	33.8	37.3	28.9
	28 M x 0	Covered	3.14	34.0	37.6	28.4
	28 M x 0	Open	3.04	33.7	36.7	29.6
5	28 M x 0	Inert	3.34	33.5	37.3	29.2
	28 M x 0	Covered	3.86	33.9	37.3	28.8
	28 M x 0	Open	3.40	33.4	37.5	29.1
6	28 M x 0	Inert	5.58	33.2	36.9	29.9
	28 M x 0	Covered	5.68	33.7	37.9	28.4
	28 M x 0	Open	5.33	32.3	36.5	31.2
7	28 M x 0	Inert	4.54	33.9	38.2	27.9
	28 M x 0	Covered	4.63	33.6	37.8	28.6
	28 M x 0	Open	4.24	32.7	36.9	30.4
9	28 M x 0	Inert	2.80	34.3	37.5	28.2
	28 M x 0	Covered	3.30	34.8	37.1	28.1
	28 M x 0	Open	3.00	33.9	38.3	27.8
1	1/4" x 28 M	Inert	5.73	35.2	45.0	19.8
	1/4" x 28 M	Open	5.90	35.1	44.5	20.4
7	1/4" x 28 M	Open	6.61	35.3	45.7	18.9

Table 7.2 Proximate Analysis of weathered samples of Pittsburgh No. 8 coal.

Incr.	Size	Storage Mode	Moisture	(ON DRY BASIS %) Volatile Matter	Fixed Carbon	Ash
1	28 M x 0	Inert	1.81	30.2	52.1	17.7
	28 M x 0	Covered	1.86	30.0	52.0	18.0
	28 M x 0	Open	1.86	30.2	51.9	17.9
2	28 M x 0	Inert	1.82	30.3	53.0	17.7
	28 M x 0	Covered	1.90	30.4	51.9	17.7
	28 M x 0	Open	1.95	30.4	51.9	17.7
4	28 M x 0	Inert	1.82	30.9	51.1	18.0
	28 M x 0	Covered	1.98	30.6	51.4	18.0
	28 M x 0	Open	2.00	31.2	50.0	18.8
6	28 M x 0	Inert	2.00	30.6	51.5	17.9
	28 M x 0	Covered	2.16	30.7	51.3	18.0
	28 M x 0	Open	2.35	30.6	50.5	18.9
8	28 M x 0	Inert	1.96	30.5	51.4	18.1
	28 M x 0	Covered	2.26	30.5	51.7	17.7
	28 M x 0	Open	2.15	30.3	51.8	17.9
9	28 M x 0	Inert	2.15	30.1	51.9	18.0
	28 M x 0	Covered	2.67	30.4	51.9	17.7
	28 M x 0	Open	2.62	30.4	51.9	17.7
1	1/4" x 28 M	Inert	1.98	36.1	52.4	11.3
	1/4" x 28 M	Open	2.00	36.1	52.7	11.2
8	1/4" x 28 M	Inert	2.40	35.5	53.5	11.0
	1/4" x 28 M	Open	2.40	36.2	53.0	10.8

Table 7.3 Proximate Analysis of weathered samples of Upper Freeport PA coal.

Incr.	Size	Storage Mode	Moisture	(ON DRY BASIS %) Volatile Matter	Fixed Carbon	Ash
1	28 M x 0	Inert	0.88	25.7	64.4	9.9
	28 M x 0	Covered	0.86	25.6	64.6	9.8
	28 M x 0	Open	0.91	25.9	64.5	9.6
4	28 M x 0	Inert	0.94	25.3	65.1	9.6
	28 M x 0	Covered	1.06	25.2	64.8	10.0
	28 M x 0	Open	1.17	25.4	64.8	9.8
8	28 M x 0	Inert	1.00	25.8	64.5	9.7
	28 M x 0	Covered	0.99	25.8	64.5	9.7
	28 M x 0	Open	0.97	26.0	64.6	9.7
10	28 M x 0	Inert	1.02	25.3	64.9	9.8
	28 M x 0	Covered	1.06	25.5	64.7	9.8
	28 M x 0	Open	1.20	26.0	65.3	8.7
1	1/4" x 28 M	Inert	0.91	26.5	64.6	8.9
	1/4" x 28 M	Open	0.86	26.6	64.7	8.7
8	1/4" x 28 M	Inert	0.94	27.3	63.7	9.1
	1/4" x 28 M	Open	0.82	26.7	63.4	9.9

Figure 5.1 Flotation kinetics of Illinois No. 6 coal wet ground to 200 mesh with the standard collector dosage added to the rod mill or flotation cell.

Coll. Addition	Flot. time (min)	Yield %	1-Yield
Cell	0.0	0.0	1.0
	0.5	28.9	0.77
	1.0	16.0	0.55
	2.0	21.6	0.34
	4.0	12.8	0.2
	5.0	2.3	0.19
Mill	0.0	0.0	1.0
	0.5	48.5	0.52
	1.0	21.7	0.3
	2.0	11.0	0.19
	4.0	4.3	0.15
	5.0	0.9	0.14

Figure 5.2 Flotation kinetics of Pittsburgh No. 8 coal wet ground to 200 mesh with the standard collector dosage added to the rod mill or flotation cell.

Coll. Addition	Flot. time (min)	Yield %	1-Yield
Cell	0.0	0.0	1.0
	0.5	21.6	0.78
	1.0	18.1	0.6
	2.0	24.7	0.36
	4.0	11.2	0.24
	5.0	3.2	0.21
Mill	0.0	0.0	1.0
	0.5	17.6	0.82
	1.0	13.1	0.69
	2.0	13.4	0.56
	4.0	11.1	0.45
	5.0	2.6	0.42

Figure 5.3 Flotation kinetics of Upper Freeport PA coal wet ground to 200 mesh with the standard collector dosage added to the rod mill or flotation cell.

Coll. Addition	Flot. time (min)	Yield %	1-Yield
Cell	0.0	0.0	1.0
	0.5	29.0	0.71
	1.0	20.4	0.51
	2.0	18.4	0.32
	4.0	10.1	0.22
	5.0		
Mill	0.0	0.0	1.0
	0.5	22.7	0.77
	1.0	17.2	0.6
	2.0	19.4	0.41
	4.0	13.3	0.27
	5.0	4.1	0.23

Figure 5.4 Initial flotation kinetics of Illinois No. 6 coal with different dodecane additions to the rod mill.

Coll. Dosage lb/T	Flot. time (min)	Yield %	1-Yield
0.8	0.5	21.0	0.79
	1.0	13.1	0.66
1.6	0.5	28.1	0.72
	1.0	14.8	0.57
3.2	0.5	40.0	0.6
	1.0	22.3	0.38
6.3	0.5	49.8	0.5
	1.0	24.4	0.26
12.7	0.5	60.8	0.39
	1.0	21.9	0.17

Figure 5.5 Initial flotation kinetics of Pittsburgh No. 8 coal with different dodecane additions to the rod mill.

Coll. Dosage lb/T	Flot. time (min)	Yield %	1-Yield
0.5	0.5	8.1	0.92
	1.0	6.6	0.85
1.1	0.5	13.9	0.86
	1.0	10.0	0.76
2.1	0.5	18.3	0.82
	1.0	14.5	0.67
4.2	0.5	30.6	0.69
	1.0	20.4	0.49
8.4	0.5	47.6	0.52
	1.0	25.3	0.27

Figure 5.6 Initial flotation kinetics of Upper Freeport PA coal with different dodecane additions to the rod mill.

Coll. Dosage lb/T	Flot. time (min)	Yield %	1-Yield
0.1	0.5	21.7	0.78
	1.0	19.2	0.59
0.3	0.5	20.7	0.79
	1.0	19.3	0.6
0.5	0.5	23.5	0.77
	1.0	20.0	0.57
2.1	0.5	26.8	0.73
	1.0	24.0	0.49
4.2	0.5	38.1	0.62
	1.0	29.9	0.32

Figure 5.7 Initial flotation rate constants for the three base coals as a function of the dodecane addition to the rod mill.

Coal	Collector added (lb/T)	k (min)
Illinois No. 6	0.8	0.43
	1.6	0.58
	3.2	0.98
	6.3	1.36
	12.7	1.78
Pittsburgh No. 8	0.5	0.16
	1.1	0.28
	2.1	0.4
	4.2	0.72
	8.4	1.3
Upper Freeport PA	0.1	0.52
	0.3	0.5
	0.5	0.56
	2.1	0.69
	4.2	1.1

Figure 6.1 Effect of pH on flotation of 28 M dry grind Pittsburgh No. 8.

pH	Float. Yield %	Pyr. S Rej. %	EI
3.4	81.3	40.0	25.9
6.0	82.9	43.7	32.3
6.0	83.5		
8.2	80.6	50.0	35.3
10.0	72.4	62.5	40.0

Figure 6.2 Effect of pH on the flotation of 28 M Wet grind Pittsburgh No. 8 coal using lime.

pH	Flot. Yield %	Pyr. S Rej. %	EI
3.7	86.5	33.0	23.3
6.0	84.3	37.2	26.3
6.0	86.3		
8.0	88.1	36.4	27.4
10.0	82.3	42.0	26.0

Figure 6.3 Effect of pH on 200 M Dry Grind Pittsburgh No. 8.

pH	Flot. Yield %	Pyr. S Rej. %	EI
4.6	75.6	62.0	42.7
6.0	64.4	72.0	41.3
6.0	63.0		
8.2	61.7	78.5	44.5
10.0	40.0	82.3	25.3

Figure 6.4 Effect of pH on 200 M wet grind Pittsburgh No. 8 coal.

pH	Flot. Yield %	Pyr. S Rej. %	EI
4.5	76.1	66.2	48.3
6.0	67.0	80.0	52.3
6.0	70.0		
8.2	63.9	80.0	49.1
10.0	40.3	73.0	29.1

Figure 6.5 Effect of pH on flotation of 200 M Wet Grind Upper Freeport PA coal.

pH	Flot. Yield %	Pyr. S Rej. %	EI
3.5	69.5	73.0	49.5
6.0	77.7	70.0	53.0
6.0	77.7		
8.0	76.5	75.0	57.0
10.0	76.5	73.7	56.0

Figure 6.6 Effect of Methanol on the flotation of 200 M wet grind Upper Freeport PA coal.

Methanol lb/T	Flot. Yield %	Flot. Ash %
0.0	23.8	4.0
2.25	24.7	4.1
2.25	24.7	4.1
6.75	26.5	4.0
13.5	26.5	3.8

Figure 6.7 Effect of Ethanol on the flotation of 200 M wet grind Upper Freeport PA coal.

Ethanol lb/T	Flot. Yield %	Flot. Ash %
0.0	23.8	4.0
2.25	24.2	3.5
4.5	34.3	3.8
6.75	34.5	3.5
13.5	48.5	3.9

Figure 7.1 Size Distribution of the standard grind (95% passing 28 mesh) and the weathered samples of Illinois No. 6 coal (Increment No. 1).

	CUMULATIVE WT. PERCENT PASSING			
Mesh	Std. Grin	Inert	Cover.	Open
270				
200	36.4			
150	42.7	14.4	13.9	14.9
100	52.7	21.3	21.9	21.4
65	66.9	32.2	32.7	31.8
48	80.8	47.7	48.2	46.6
35	89.4	70.9	71.2	69.8
28	92.8	99.9	99.8	99.9
20	94.5	100	99.9	100
14	95.3		100	
10	95.9			
8	96.1			

Figure 7.2 Size distribution of the standard grind (95% passing 28 mesh) and the weathered samples of Illinois No. 6 coal (Increment No. 4).

	CUMULATIVE WT. PERCENT PASSING			
Mesh	St. Grin.	Inert	Covered	Open
270				
200	36.4			
150	42.7	17.9	19.4	18.7
100	52.7	25.4	27	26.4
65	66.9	37	38.4	37.4
48	80.8	52.3	53.6	52.6
35	89.4	75	75.6	74.5
28	92.8	99.9	99.9	99.7
20	94.5	99.9	100	99.8
14	95.3	100		99.9
10	95.9			100
8	96.1			

Figure 7.3 Size distribution of the standard grind (95% passing 28 mesh and the weathered samples of Illinois No. 6 coal (Increment 6).

	CUMULATIVE WT. PERCENT PASSING			
Mesh	St. Grin.	Inert	Covered	Open
270				
200	36.4			
150	42.7	22.9	18.6	27.3
100	52.7	30.1	25.3	35
35	66.9	40.5	35.1	46.2
48	80.8	54.7	48.6	59.6
35	89.4	75.2	69.7	78.4
28	92.8	99.8	99.7	99.8
20	94.5	99.9	99.8	99.8
14	95.3	100	99.9	99.9
10	95.9		100	100
8	96.1			

Figure 7.4 Size distribution of the standard grind (95% passing 28 mesh) and the weathered samples of Illinois No. 6 coal (Increment 7).

	CUMULATIVE WT. PERCENT PASSING			
Mesh	St. Grin.	Inert	Covered	Open
270				
200	36.4			
150	42.7	24.3	21.7	24.7
100	52.7	33.2	30.9	33.6
65	66.9	46.1	44.3	46.3
48	80.8	63.1	61.6	63.0
35	89.4	86.5	25.8	27.2
28	92.8	99.8	99.8	99.9
20	94.5	99.8	99.9	100
14	95.3	100	100	
10	95.9			
8	96.1			

Figure 7.5 Size distribution of Increments 1 thru 7 of Illinois No. 6 samples weathered under open condition.

Mesh	CUMULATIVE WEIGHT PERCENT PASSING Incr.1	Incr.2	Incr.3	Incr.4	Incr.5	Incr.6	Incr.7
270							
200							
150	14.9	26.0	27.1	18.7	22.4	27.3	24.3
100	21.4	33.6	35.3	26.4	29.5	35.0	32.1
65	31.8	44.0	47.1	37.4	40.1	46.2	44.8
48	46.6	57.9	60.6	52.6	53.9	59.6	61.1
35	69.8	76.5	79.7	74.5	74.9	78.4	86.5
28	99.9	99.5	99.8	99.7	99.7	99.7	100.0
20	100.0	99.8	99.9	99.8	99.8	99.8	
14		99.9	100.0	99.9	99.9	99.9	
10		100.0		100.0	100.0	100.0	
8							

Figure 7.6 Effect of weathering time of the generation of material in different size intervals obtained by sieving the minus 28 mesh material of Illinois No. 6 coal weathered under open conditions.

Weathering Time, months	WEIGHT PERCENT IN SIZE INTERVAL 28 X 35 M	48 x 65 M	100 x 150 M	-150 M
0.5	30.1	14.8	6.5	14.9
1.0	23.0	13.9	7.6	26.0
1.5	20.1	13.5	8.2	27.1
2.0	25.2	15.2	7.7	18.7
2.5	24.8	13.8	7.1	22.4
3.0	21.4	13.4	7.7	27.3
4.0	13.5	16.3	8.3	24.3
6.0	12.7	16.7	8.9	24.7

Figure 7.7 Size distribution of the weathered samples (1/4 inch x 28M) of Illinois No. 6 coal.

Mesh	CUMULATIVE WEIGHT PERCENT PASSING Incr.1 (Inert)	Incr.1 (Open)	Incr.7 (Open)	Incr.9 (Open)
150	0.18	0.62	1.24	1.58
100	0.22	0.73	1.46	1.83
65	0.28	0.87	1.72	2.13
48	0.36	1.05	2.02	2.6
35	0.48	1.35	2.41	3.6
28	1.29	2.8	5.39	7.7
20	9.0	11.0	13.5	17.1
14	18.4	20.6	22.8	26.9
10	33.3	35.6	37.3	42.1
8	48.5	50.8	52.0	56.4
7		59.7	61.4	64.1
6	78.6	69.8	70.6	73.2
5	81.6	82.3	82.7	
4			92.5	91.7
3.5	88.8	91.5		97.8

Figure 7.8 Size distribution of the standard grind (95% passing 28 mesh) and the weathered samples of Pittsburgh No. 8 coal (Increment 1).

	CUMULATIVE WT. PERCENT PASSING			
Mesh	St. Grin.	Inert	Covered	Open
270	30.9	33.7	35.2	34.5
200	39.6	41.1	42.3	41.7
150	50.2	51.7	52.0	51.5
100	64.5	64.2	63.9	63.6
65	82.2	80.5	80.4	79.3
48	76.7	99.8	99.9	99.8
35	99.4	100.0	100.0	100.0
28	99.9			
20	99.9			
14	100.0			
10				
8				

Figure 7.9 Size distribution of the standard grind (95% passing 28 mesh) and the weathered samples of Pittsburgh No. 8 coal (Increment 8).

	CUMULATIVE WT. PERCENT PASSING			
Mesh	St. Grin.	Inert	Covered	Open
270	30.9	33.8	31.9	34.8
200	39.6	41.0	39.5	42.2
150	50.2	51.2	50.3	52.2
100	64.5	63.2	63.1	64.3
65	82.2	79.6	79.7	79.8
48	95.4	99.7	99.8	99.8
35	99.5	100.0	100.0	100.0
28	99.9			
20	99.9			
14	100.0			
10				
8				

Figure 7.10 Size distribution of the weathered samples (1/4 inch x 28M) of Illinois No. 6 coal.

	CUMULATIVE WT. PERCENT PASSING			
Mesh	Incr.1 (Open)	Incr.1 (Open)	Incr.8 (Open)	Incr.9 (Open)
150				
100				
65				
48				
35	0.2	0.12		
28	0.05	0.57	0.04	1.07
20	8.2	6.7	6.6	7.7
14	16.9	14.5	14.6	15.6
10	31.2	27.7	28.3	28.5
8	45	41.9	42.4	41.3
7	53.3	50.2	50.6	49.2
6	63.2	60.7	61.1	59.8
5	74.4	73	73.4	72.1
4	85.2	84.7	85.3	84.6
3.5			95.2	96

Figure 7.11 Effect of weathering time of the generation of material in different size intervals for Pittsburgh No. 8 coal weathered under open conditions.

Weathering Time, months	WEIGHT PERCENT IN SIZE INTERVAL 28 x 35 M	48 x 66 M	100 x 150 M	-150 M
0.5	20.4	12.1	7.2	34.5
1.0	18.2	13.1	7.8	33.6
1.5				
2.0	19.7	12.3	7.4	34.1
2.5				
3.0	19.7	14.8	7.6	33.5
4.0				
5.0	19.9	12.3	7.4	34.8

Figure 7.12 Size distribution of the standard grind (95% passing 28 mesh) and the weathered samples of Upper Freeport PA coal (Increment 1).

Mesh	CUMULATIVE WT. PERCENT PASSING St. Grin.	Inert	Covered	Open
270				
200				
150	32.5	26.2	27.4	26.0
100	44.3	34.7	35.9	34.9
65	57.8	46.7	48.3	47.2
48	71.7	61.8	63.7	62.0
35	87.8	82.4	84.0	82.4
28	97.3	99.8	99.9	99.9
20	99.6	100	100	100
14	99.9			
10	100			
8				

Figure 7.13 Size distribution of the standard grind (95% passing 28 M) and the weathered samples of Upper Freeport PA coal (Increment 8).

Mesh	CUMULATIVE WT. PERCENT PASSING St. Grin.	Inert	Covered	Open
200				
150	32.5	23.5	21.5	20.4
100	44.3	31.0	29.1	27.1
65	57.8	41.7	40.4	36.7
48	71.7	55.3	54.1	49.5
35	87.8	77.9	73.9	68.9
28	97.3	99.3	98.5	98.0
20	99.6	100	100	100
14	99.9			
10	100			
8				

Figure 7.14 Size distribution of the weathered samples (1/4 inch x 28M) of Illinois No. 6 coal.

Mesh	CUMULATIVE WT. PERCENT PASSING Incr.1 (Inert)	Incr.1 (Open)	Incr.8 (Open)
150			
100			
65			
48			
35	0.64	0.66	3.17
28	2.9	2.6	4.5
20	11.7	10.8	13.5
14	21.8	20.3	24.6
10	37.3	35.0	41.4
8	52.3	48.6	54.8
7	61.3	56.4	62.7
6	70.5	65.2	71.9
5	80.7	76.8	81.7
4	89.6	86.9	90.2
3.5	97.0	96.1	97.1

Figure 7.15 Film flotation results of weathered (inert, covered and open conditions) samples of Illinois No. 6 coal (Increment 3).

γ_{LV} mN/m	Inert	Cover.	Open
36.5	0	0	0
40.5	1.8	1.3	0
45.0	4.9	5.2	1.5
48.0	9.8	9.0	4.1
51.0	18.9	16.3	8.5
54.5	20.2	20.1	13.3
58.8	28.3	26.3	18.1
64.5	55.4	36.0	26.4
72.7	59.2	58.9	46.7

Figure 7.16 Film flotation results of weathered (inert, covered and open) samples of Illinois No. 6 coal (Increment 3) deslimed using 1-D water.

γ_{LV} mN/m	Inert	Cover.	Open
33.0	0.0	0.0	0.0
36.5	2.9	1.1	0.7
40.5	13.5	3.7	4.4
45.0	22.4	12.0	11.2
48.0	30.1	17.5	19.9
51.0	36.6	24.3	24.4
54.5	47.8	30.6	30.0
58.8	59.1	37.9	36.5
64.5	65.1	51.6	45.9
72.7	71.8	61.2	51.2

Figure 7.17 Effect of weathering time on the film flotation partition curves of Illinois No. 6 coal samples weathered under open mode for 0.5, 1.5 and 4 months.

OPEN TIME, MONTHS		
0.5	1.5	4
0.0	0.0	0.0
1.3	0.7	1.5
4.3	4.4	3.6
12.9	11.2	9.1
14.6	19.9	10.7
19.7	24.4	14.0
25.0	30.0	18.0
34.8	36.5	27.6
48.0	45.9	34.5
56.3	51.2	45.1

Figure 7.18 Effect of weathering on the zeta potential of Illinois No. 6 coal

RESEARCH SAMPLE		INCR. 1 (INERT)		INCR. 1 (COVERED)		INCR. 4 (COVERED)	
pH	ε,mV	pH	ε,mV	pH	ε,mV	pH	ε,mV
2.75	-1.6	2.3	-5.8	2.9	-8.0	2.7	-11.8
4.03	-16.0	3.0	-16.0	4.2	-15.6	4.1	-20.9
5.89	-22.3	7.5	-28.9	8.1	-27.8	4.7	-19.7
5.95	-24.2	7.8	-29.6	8.4	-30.2	7.6	-26.9
7.28	-39.6	8.3	-29.8	8.9	-32.6	8.7	-36.1
7.94	-41.4	8.6	-35.6	11.3	-53.3	10.7	-55.9
8.63	-44.6	8.9	-37.4				
9.02	-47.2	11.3	-51.7				
10.31	-52.5						
10.98	-55.8						

Figure 7.19 Effect of weathering on the zeta potential of Pittsburgh No. 8 coal

RESEARCH SAMPLE		INCR. 1 (INERT)		INCR. 4 (COVERED)		INCR. 4 (OPEN)	
pH	ε,mV	pH	ε,mV	pH	ε,mV	pH	ε,mV
2.7	10.0	2.8	-18.0	2.9	-20.0	2.9	-15.4
4.44	-5.6	4.7	-31.1	4.6	-32.4	4.5	-29.8
4.84	-2.5	6.0	-29.2	6.7	-38.0	6.9	-41.7
5.53	-21.8	7.6	-41.8	7.6	-41.7	7.5	-42.4
5.61	-21.90	8.2	-47.6	8.5	-45.4	7.7	-46.7
6.56	-23.10	11.2	-57.3	10.1	-53.3	8.2	-51.5
8.26	-50.15			11.1	-50.2	11.2	-53.4
10.18	-61.1						
10.44	-69.0						
11.48	-58.2						

Figure 7.24 Effect of weathering on Hallimond tube flotation of Illinois No. 6 coal.

Float. Time seconds	Research Sample	PERCENT FLOAT Incr. 1 (Inert)	Incr. 1 (Covered)	Incr. 4 (Covered)	Incr. 4 (Open)
0	4.0	3.0	0.0	0.0	0.0
15	28.8	13.6	14.9	12.2	
30					20.4
60	56.1	21.1	25.2	25.2	28.3
120	75.1				40.4
180		54.1, 50.1	43.3, 50.0	46.2	
240	92.8				46.8
300		61.2	56.8	64.6	

Figure 7.25 Effect of weathering on Hallimond tube flotation of Pittsburgh No. 8 coal.

Float. Time seconds	Research Sample	PERCENT FLOAT Incr. 1 (Inert)	Incr. 1 (Covered)	Incr. 4 (Covered)	Incr. 4 (Open)
0	5.0	2.0	0.0	0.0	0.0
15	25.5	12.2	10.7	15.4	17.4
60	44.0	16.7	24.6	23.2	27.8
120	70.5	--	29.6	--	38.4
180	--	38.7, 48.1	46.4	41.0, 38.8	--
240	89.3	--	--	--	54.3
300	--	77.2	47.7	61.0	--

Figure 7.26 Effect of weathering time on the flotation yields Illinois No. 6 coal stored under inert, covered and open conditions.

Weathering Time, months	FLOTATION YIELDS, PERCENT 28 M x 0 Inert	Covered	Open	1/4 inch x 28M Inert	Open
0.5	68.6	58.5	56.7	83.3	83.3
1.0	66.3	53.5	53.2		
1.5	63.8	53.4	53.6		
2.0		49.0	50.1		
2.5	53.5	43.7	42.9		
3.0		48.3	42.8		
4.0	63.45	55.0	36.4		
5.0					
6.0	48.3	39.0	29.1		81.4

Figure 7.27 Effect of weathering time on the flotation yields Pittsburgh No. 8 coal stored under inert, covered and open conditions.

FLOTATION YIELD, PERCENT

Weathering Time, months	28 M X 0 Inert	Covered	Open	1/4 inch x 28 M Inert	Open
0.5	62.1	48.6	55.3	93.6	94.1
1.0	63.1	51.4	47.0		
1.5					
2.0	68.5	46.7	55.5		
2.5					
3.0	64.8	51.5	49.1		
4.0					
5.0	59.6	35.0	51.8	93.8	93.7
6.0	59.5	41.4	26.9		

Figure 7.28 Effect of weathering time on the flotation yields of Upper Freeport PA coal stored under inert, covered and open conditions.

FLOTATION YIELDS, PERCENT

Weathering Time, months	28 M x 0 Inert	Covered	Open	1/4 inch x 28 M Inert	Open
0.5	89.1	86.7	84.7	92.5	91.7
2.0	87.0	86.1	83.3		
5.0	88.2	83.5	70.2	91.4	87.0
7.0	78.3	81.8	39.8		

Figure 7.29 Effect of weathering time on the flotation yields of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals stored under open conditions.

FLOTATION YIELDS, PERCENT

Weathering Time, months	Illinois No.6	Pittsburgh No.8	Upper Freeport PA
0.5	58.8	54.4	84.7
1.0	52.4	65.1	
1.5	56.4		
2.0	51.2	55.5	83.3
2.5	43.5		
3.0	42.1	49.0	
4.0	37.5		
5.0		51.8	70.2
6.0	29.2	26.9	