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Hayri Erten

Missouri University of Science and Technology

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DETERMINATION OF WASHABILITY AND
FLOTATION CHARACTERISTICS OF IMPORTANT
MISSOURI COAL SEAMS

Dr. M. H. Erten
Department of Mining Engineering
UMR, Rolla, MO.

Abstract

Most of the 14 workable coal seams in the State of Missouri are considered to be high in ash and sulfur contents and need beneficiation in order to meet customer and Environmental Protection Agency requirements. If proper beneficiation methods can be designed, more Missouri coal will be mined and used within the state.

The objectives of this research are (1) to draw the washability curves of important Missouri coal seams in order to determine their washability characteristics, and (2) to determine the flotation characteristics of the same seams.

Tests conducted on 20 samples taken from Bevier, Croweburg, Mulky, and Summit seams showed important differences in ash, sulfur, and pyrite contents. Their washability and flotation characteristics were also quite variable.

1. INTRODUCTION

Missouri's estimated coal resource base is almost 50 billion tons. Nearly 900 million tons of this is classed as a recoverable reserve. Geologically Missouri coal seams occur in strata of Pennsylvanian age as do most of the coal seams of the Midwestern and Eastern United States. More than 30 coal seams have been identified in Missouri, but as shown in Figure 1, only 14 of them are of sufficient thickness and spatial extent to be considered sources of potentially mineable coal. However, most of the 14 workable coal seams are considered to be high in ash and sulfur contents and require beneficiation in order to meet customer and EPA requirements. Because of this fact, the State's yearly production is less than seven

million tons, which is about one-third of its annual consumption. If better resource evaluation is made and more suitable beneficiation methods are designed to remove excess ash and sulfur, more Missouri coal will be utilized within the state for direct or indirect combustion purposes. Coal beneficiation will not only remove part of the sulfur in the coal, it will also upgrade the coal by removing loose shale or rock existing in the coal.

Presently, the power plants use very efficient scrubbers to remove SO₂ from the flue gases. With such high-efficiency SO₂ removal systems available, it is not necessary to remove all the sulfur contained in a coal obtained from a seam by physical washing. However, coal preparation techniques that remove pyritic

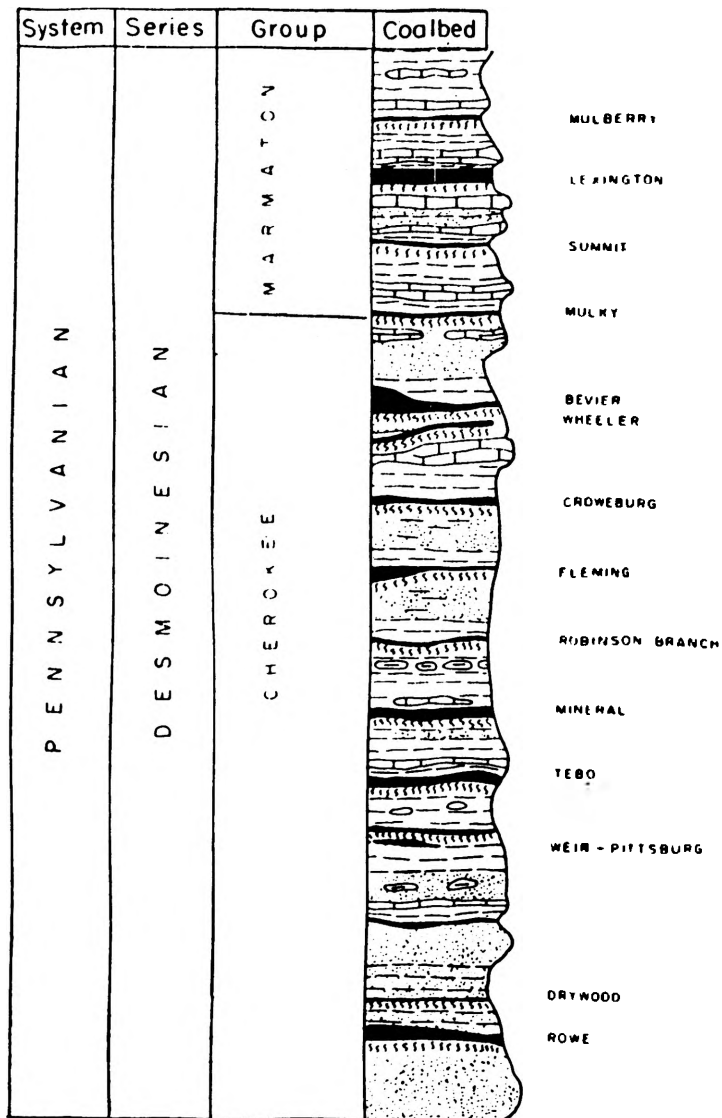


Figure 1. Stratigraphy of the Principal Coal Seams of Missouri (After Robertson¹, 1971)

sulfur cheaply can be used as a primary means of upgrading coals before burning them in a power plant. Before a decision can be taken about the coal preparation method to be applied, the washability and flotation characteristics of the coal seams must be determined.

In order to investigate the possibilities mentioned above, research has been initiated which uses the facilities of the coal preparation laboratory of the Mining Engineering Department of UMR, and this paper presents the preliminary findings of this research.

In the course of this study it is expected that during a period of two years, a total of 200 samples will be taken from different localities in the State of Missouri. So far various tests have been conducted on 20 channel samples.

Aims of the analysis program are:

1. To draw the washability curves of Missouri coal seams in order to determine their washability characteristics.
2. To determine flotation behavior of Missouri coal seams.

The basic data obtained as a result of these studies will be helpful in the long term technical evaluation of Missouri coal seams, especially when gasification, liquefaction, or coal-oil mixture uses are contemplated. The tests will also establish the percentage of sulfur that may have to be removed during or after direct burning to meet the EPA or other governmental requirements.

2. EXPERIMENTAL PROCEDURES

2.1 SAMPLING PLAN

A channel sample of 50 to 100 lb was taken from each coal seam either at the outcrop or at the faces of operating mines. Some drill core samples will also be obtained from places not suitable for direct sampling.

The sampling was the responsibility of the Division of Geology and Land Survey of the Missouri Department of Natural Resources. The funds and personnel necessary for sampling were provided by the above agency through a grant obtained from the Ozarks Regional Commission.

2.2 WASHABILITY STUDIES

After crushing each sample to minus one-inch size, two 5-kilogram sub-samples were prepared, one being

used for determination of moisture, ash, and sulfur contents of the coal, and the other for determining its washability characteristics.

The washability sample was first screened through a 16-mesh screen in order to separate fines that are difficult to treat in zinc chloride solution used for washability determinations. Normally, for washability tests, minus 16-mesh fraction is treated in heavy organic liquids that result in better separation of different specific gravity fractions of fine coal. For the first 20 Missouri coal samples tested, the minus 16-mesh fraction averaged about 10.5 percent of the bulk samples, and, for this reason, at the present time, only moisture, ash, and sulfur analyses and flotation tests were conducted on these fine coal samples.

The plus 16-mesh fraction of the washability sample was subjected to "float-and-sink" tests in zinc chloride solutions of 1.30, 1.40, 1.50, 1.60, and 1.70 specific gravities in order to determine the washability characteristics of the seam represented by the sample. With the use of this data, the ash and sulfur distribution in various gravity fractions is studied and the standard washability curves are drawn.

2.3 FLOTATION TESTS

Most coal washeries use froth flotation in cleaning slurries containing -0.5 mm size coal. Since most of the coal seams investigated are not yet being worked, it is impossible for us to obtain slurry samples from them. For this reason the preliminary flotation tests were conducted on special samples prepared from different seams. During the flotation tests, maximum removal of impurities, especially that of sulfur, will be attempted by using various flotation reagents. Regular flotation procedure as well as reverse flotation method of sulfur elimination will be applied. Frothers and collectors such as pine oil, MIBC (methyl isobutyl carbinol), cresylic acid, kerosene, and fuel oil will be used in various quantities. Some froth may be re-floated for better sulfur elimination. Reverse flotation, i.e., depressing of coal and floating of pyrite will also be tried on some froths obtained in regular flotation. Lime, potassium permanganate, starch, and stannous chloride will be used as depressing agents for pyrite or coal.

For all flotation tests, a pulp density of ten percent (by weight) will be adapted. The experiments will be conducted with a Galliger Model LA-500 batch-type flotation machine using 1.5-liter cell.

3. TEST RESULTS

3.1 PROXIMATE ANALYSIS OF COAL SAMPLES

Table 1 shows moisture, ash, and sulfur analyses and BTU contents of coal samples investigated. Moisture contents were determined with a Brabender apparatus, and the ash analyses were conducted with the standard ASTM procedures. The sulfur analyses were made with a Leco sulfur analyser. The calorific values (Btu's) were determined with a Parr adiabatic oxygen bomb calorimeter. Btu values are calculated on moisture-free basis.

Table 1. Proximate Analysis of Samples

Sample No.	Name of Seam	Name of County	% Moisture (Total)	% Ash (Dry)	% S (Total)	Btu Value (Dry)
1	Croweburg	Howard	11.50	17.90	4.87	10,763
2	Bevier	Chariton	28.93	15.10	1.93	8,274
3	Croweburg	Chariton	11.96	27.52	2.77	9,392
4	Mulky	Randolph	10.55	25.17	5.02	9,920
5	Bevier	Chariton	19.35	23.73	2.12	8,280
6	Uncorrelated	Dade	9.76	10.37	0.95	11,152
7	Bevier-Wheeler	Howard	15.25	12.87	5.32	11,929
8	Summit	Howard	11.42	15.73	3.51	11,402
9	Bevier-Wheeler	Howard	13.30	19.22	6.40	10,887
10	Bevier-Wheeler	Howard	7.68	15.37	5.09	11,620
11	Wheeler	Randolph	16.69	24.47	8.02	10,136
12	Bevier	Randolph	18.41	12.53	3.69	10,839
13	Mulky	Randolph	12.24	13.75	6.76	10,982
14	Croweburg	Randolph	9.72	14.86	3.87	11,708
15	Summit	Howard	14.41	17.83	4.54	10,816
17	Croweburg	Henry	29.17	18.78	3.05	8,193
18	Mineral	Vernon	5.09	16.38	6.30	11,880
19	Bevier	Randolph	12.61	13.72	5.20	11,660
20	Croweburg	Vernon	4.55	18.52	4.93	12,075
21	Wheeler	Randolph	9.76	17.38	6.78	11,062

Table 2 shows reclassification of seams according to their moisture ash, and sulfur content.

Table 2. Grouping of Samples According to Moisture, Ash, and Sulfur Contents

Sample No.	Name of Seam	Name of County	% Moisture (Total)	% Ash (Dry)	% S (Dry)	Thickness, Inches
1	Croweburg	Howard	11.50	21.12	4.87	10-19
3	"	Chariton	11.96	27.52	2.77	20
14	"	Randolph	9.72	14.86	3.87	-
17	"	Henry	29.17	18.78	3.05	-
20	"	Vernon	4.55	18.52	4.93	12
2	Bevier	Chariton	28.93	15.10	1.93	14
5	"	Chariton	19.35	23.73	2.12	24
12	"	Randolph	18.41	12.53	3.69	-
19	"	Randolph	12.61	13.72	5.20	29
4	Mulky	Randolph	10.55	25.17	5.02	14
13	"	Randolph	12.24	13.75	6.76	-
7	Bevier-Wheeler	Howard	15.25	12.87	5.32	4-18
9	"	Howard	13.30	19.22	6.40	40
10	"	Howard	7.68	15.37	5.09	39
11	Wheeler	Randolph	16.69	24.47	8.02	-
21	"	Randolph	9.76	17.38	6.78	5-11
6	Uncorrelated	Dade	9.76	10.37	0.95	4-26
15	Summit	Howard	14.41	17.83	4.54	5-18
8	"	Howard	11.42	15.73	3.51	2-21
18	Mineral	Vernon	5.09	16.38	6.30	18

3.2 SUMMARY OF WASHABILITY TEST RESULTS

Assuming the coals to be washed at 1.40 specific gravity, which is the usual separation density employed in most coal washeries, the floats and sinks and their ash and sulfur analysis are given in Table 3.

Table 3. Separation of Samples at 1.40 Specific Gravity (+16 Mesh Coals)

Sample No.	Name of Seam	+1.40 Analyses			-1.40 Analyses		
		%Float	%Ash	% S	%Sink	%Ash	%S
1	Croweburg	82.43	9.03	4.41	17.54	42.67	7.10
3	"	89.27	13.95	2.17	10.73	27.13	2.82
14	"	90.70	5.81	3.34	9.30	55.32	17.97
17	"	71.88	11.45	2.28	28.12	37.52	5.01
20	"	79.26	5.94	2.47	20.74	44.64	13.28
2	Bevier	61.27	9.44	1.75	38.73	25.43	2.22
5	"	83.11	8.24	2.11	16.89	23.23	2.16
12	"	93.10	5.98	2.53	6.90	54.70	11.57
19	"	83.68	8.19	2.85	16.32	44.54	14.94
4	Mulky	68.70	6.05	3.37	31.30	70.30	12.38
13	"	81.63	10.82	2.90	18.37	46.72	9.68
7	Bevier-Wheeler	83.86	9.20	4.77	16.14	45.23	10.64
9	"	70.05	8.94	3.14	29.95	41.54	13.31
10	"	73.89	9.29	3.40	26.11	41.10	10.13
11	Wheeler	64.09	11.28	6.00	35.91	47.40	11.81
21	"	46.70	9.13	5.60	53.30	22.85	7.69
6	Uncorrelated	94.75	4.95	1.02	5.25	34.31	0.91
15	Summit	78.81	11.37	2.76	21.19	44.20	8.40
8	"	88.03	11.44	2.92	11.97	43.27	7.22
18	Mineral	79.22	12.45	3.35	20.78	50.93	17.26

In Table 3, +1.40 analyses belong to geometrical averages of coals floating in 1.30 and 1.40 specific gravity zinc chloride solutions. On the other hand, -1.40 analyses represent the geometrical averages of coals floating in 1.40, 1.50 and 1.70 specific gravity solutions and those totally sinking in 1.70 specific gravity solution.

3.3 FLOTATION TEST RESULTS

3.3.1 -60 Mesh size samples

Flotation test results shown in Table 4 were obtained by floating part of three bulk samples which were crushed and pulverized to -60 mesh size for ash and sulfur analyses of the main samples. Although the usual size for flotation test is -28 mesh, we have used -60 mesh samples in order to increase the possibility of pyrite separation during the flotation.

Table 4. Flotation Test Results of -60 Mesh Samples

Sample No. & Name	Froth I			Froth II			Tailing	
	%	%	%	%	%	%	%	%
	Recov.	Ash	S	Recov.	Ash	S	Ash	S
6 (uncorrel.)	91.3	5.65	0.81	-	-	-	31.93	1.24
10 (Bevier-W)	62.4	9.65	3.66	11.5	13.40	4.46	37.00	8.16
12 (Bevier)	72.6	6.68	3.09	-	-	-	27.93	4.95

In table 4, Froth I was obtained during the first three minutes and Froth II during the next three minutes. In floating samples 6 and 12, the froths looked so clean that they were mixed and analysed as a single froth. In all cases, 20 drops of pine oil and 20 drops of kerosene were used as frother and collector.

Table 5 shows the flotation test results of three -16 mesh samples which were leftovers from the washability samples as -16 mesh screenings. Before flotation, the samples were pulverized to -30 mesh (about 0.5 mm) in order to comply with the usual flotation practice.

Table 5. Flotation Test Results of -30 Mesh Samples

Sample No. & Name	Froth I			Froth II			Tailing	
	% Rec.	% Ash	% S	% Rec.	% Ash	% S	% Ash	% S
6 (uncorrel.)	14.1	21.7	0.87	73.6	25.8	0.94	31.4	1.21
10 (Bevier-W)	58.3	10.6	4.28	26.7	16.8	5.43	63.8	7.63
12 (Bevier)	81.8	7.7	3.03	9.0	23.5	3.05	27.6	5.02

4. CONCLUSIONS

From the preliminary studies conducted on 20 bulk samples, the following conclusions are drawn.

1. As it is indicated in Table 1 and 2, Missouri coal seams generally contain high ash and sulfur. This is in accordance with the results given in References 2, 3, and 4. However, Bevier seam in Chariton County (samples #2 and #5) shows about 2% sulfur which is lower than those in other counties. Also, an uncorrelated seam in Dade County (sample #6) contains less than 1% sulfur. The latter sample was taken from an outcrop and showed a thickness of 4 to 26 inches. Since this seam is very low in ash and sulfur and its calorific value is fairly high (11,152 Btu/lb), it may form an important source of coal production if its thickness increases in depth. For this reason it should further be investigated during the rest of the project.

2. Since many of the samples were taken from outcrops, they showed high moisture.

3. Table 3 shows that at 1.40 specific gravity separation, most of the samples will show high recovery with acceptable ash contents, but, except for sample # 6, their sulfur contents will still be too high for direct combustion in the power plants not equipped with SO₂ scrubbers. The table also indicates that the sulfur analyses of -1.40 fractions are considerably higher than those of +1.40 fractions. This is an indication that in addition to organic sulfur, Missouri coal contain important quantity of pyritic sulfur.⁴

4. The preliminary flotation test results shown in Tables 4 and 5 indicate that -60 mesh samples prepared from the original bulk samples are better suited to flotation than -16 mesh fractions of the same samples. However, except for sample #6, in both cases, the froths obtained are high in sulfur and further tests should be carried out in order to investigate the possibility of obtaining cleaner froths.

5. ACKNOWLEDGEMENTS

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RESUME

Dr. M. H. Erten is Acting Chairman and Professor of Mining Engineering at the University of Missouri at Rolla, Missouri.

He received his BSc in Mining Engineering from the University of Minnesota in 1944, and MA (1945) and PhD (1946 in Metallurgy) from Stanford University in California.

His industrial experience includes iron mining, coal mining, and designing of a one-million ton/year steel plant built at Iskenderun, Turkey.

Between 1962 and 1977, Dr. Erten was a Professor and Chairman of the Mining Engineering Department of the Middle East Technical University at Ankara Turkey, and joined the Mining Engineering Department of UMR on a permanent basis in 1979.

Dr. Erten has published about 20 papers in various journals and international symposia.