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SURFACE ELECTROCHEMICAL CONTROL FOR FINE COAL AND PYRITE SEPARATION

DOE Project No. DE-AC22-89PC89758

Technical Progress Report
January 1, 1990 - March 31, 1990

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Technical Progress Report
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This is the third technical progress report prepared in accordance with the reporting requirements of DOE Project No. DE-AC22-89PC89758. It covers the work performed for the period January 1, 1990 to March 31, 1990 and includes the test results on floatability of three selected coal samples. The on-going work includes the floatability evaluation of three typical U.S. coal samples, the flotation behavior of coal-pyrite and the electrochemical properties of coal-pyrite surfaces. Results obtained on surface electrochemical properties of mineral and coal pyrite and on the flotation behavior of coal pyrite will be presented in the next technical progress report.

FLOATABILITY EVALUATION OF THREE COAL SAMPLES

Research methods often used to assess floatability include such measurements as contract angles, bubble particle pick up, reagent adsorption and zeta-potential. Other tests include the critical surface tension for flotation, film flotation techniques, and large and small laboratory flotation cell tests. Test results using laboratory flotation cells are more directly valuable than other methods for process design and are more straightforward for visualizing actual industrial application. For this reason, the three coal samples were tested using a standard laboratory floatability evaluation method reported previously[1].

The three coal samples which cover a range of pyritic sulfur content and rank were selected for study. The sample preparation procedures and character of the samples have been presented in previous technical progress report.

The proximate analysis and ultimate analysis of the three coal samples analyzed are shown in Table 1 and Table 2.

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[1] W. Hu, R. Jin and D.M. Bodily, 1987, Floatability Evaluation of Fine Coal, SME, AIME Transaction Vol, 282, pp. 1910-1915.

Table 1. Proximate Analysis of Three Coal Samples

Coal Sample	Moisture %	Proximate Analysis (Dry Basis) V.M. %	F.C %	Ash %	Pyr.S. %
Illinois HVCb	11.05	36.43	47.38	16.19	2.56
Pittsburgh H	3.63	35.63	52.31	12.06	2.82
Freeport MVb	2.23	25.60	62.26	12.14	1.62

Table 2. Ultimate Analysis and Heat Value of Three Coal Samples

Heating Value Coal Sample	C%	H%	N%	S%	O%	Btu/Lb
Illinois HVCb	66.48	4.56	1.12	5.19	6.46	11,814
Pittsburgh HVCb	72.06	4.85	1.32	3.95	5.76	12,815
Upper Freeport MVb	77.25	4.64	1.32	2.24	2.41	13,601

EXPERIMENTAL

The feed for each flotation test was wet-ground in a laboratory rod mill (8-in dia. x 10-in length) to obtain a product of 95% finer than 200 mesh. An automated flotation cell adapted by the coal preparation laboratory of the Department of Energy was used for its ease of operation and reproducibility. Flotation test feed weight for each test was 125 g. Conditioning time for pulping was 4 min., for pulp level adjustment and pH measurement time 1 min., and for frother conditioning time 3 min. Flotation time required to attain steady state was 5 min. For the kinetic study, froth removal intervals were: 15 sec., 30 sec., 30 sec., 60 sec.,and 150 sec., and the accumulated time was 300 sec. Three series of tests were carried out as follows:

1. Flotation with distilled water. (This is considered as the coal's natural propencity for flotation.)

2. Flotation with frother only. (This is taken as the measurement of the coal

sample's floatability and selectivity under conventional froth flotation conditions, a criterion originally suggested by Prof. Hu and used by him in China.)

3. Salt flotation. (Salt flotation of coal has been used in China, the USSR and recently in the U.S. to evaluate the relative floatability and selectivity of coal and pyrite under an indifferent environment.)

In the assessment of flotation test results, the Yield% and Ash% of cleaned coal are generally determined in conventional practice. Since the yield is only a weight measurement and the ash itself is not the main separation goal in coal desulfurization research, additional evaluation criteria of floatability and selectivity and associated terminology are proposed as follows:

AC	Ash (%) in the cleaned coal or concentrate
AF	Ash (%) in the feed
AR	Ash rejection; (%) of total ash in the tails
BTUR	BTU recovery (%); [BTU(Conc)/BTU(feed)] X 100
C	Concentrate, (wt) cleaned coal or float product
CMR	Combustible material recovery (%)
EI	Efficiency Index for pyrite separation
F	Feed (wt)
PSC	Pyritic sulfur (%) in concentrate or cleaned coal
PSR	Pyritic sulfur (P.S.) rejection; (%) of total P.S. in tails
PST	Pyritic sulfur (%) in tailing
y	Yield (%) of clean coal or float product; y = (C/F) X 100

$$CMR = y\left(\frac{100-AC}{100-AF}\right)$$

$$PSR = (100 - y)\left(\frac{PST}{PSF}\right)$$

EI includes a matrix made up of matrix elements E_{ij}, where i refers to specific components and j to products. The two ith components in this analysis refer to combustible material (i = 1) and pyritic sulfur (i = 2). The two jth products are concentrate (j = I) and tails (j = II). From the definitions listed above it is apparent thet E_{1I} = CMR and E_{2II} = PSR. From mass balance considerations, E_{1II} = 100 - CMR and E_{2I} = 100 - PSR. Since the matrix elements of EI are measured in % the Efficiency Index is represented by the matrix

$$EI = \frac{1}{100}\begin{pmatrix} E_{1I} & E_{1II} \\ E_{2I} & E_{2II} \end{pmatrix} = CMR + PSR - 100$$

TEST RESULTS AND DISCUSSION

Natural Floatability and Selectivity

The three coal samples were wet-ground to finer than 200 mesh, conditioned with distilled water, in the absence of added reagents. The test results show that only Upper Freeport coal has natural floatability (combustible material recovery is 26%) with good pyritic sulfur rejection 96.0% (pyritic sulfur in feed is 1.62% reduced to 0.27% in clean coal), but the separation efficiency is only 22 because of the low yield. Both Illinois and pittsburgh coal samples did not display natural floatability.

Flotation with Frother (MIBC) Only

Test results for flotation in the presence of of MIBC only are summarized in Table 3 and illustrated in Figures 1-1, 1-2, 1-3. It is evident that the yield and combustible material recovery increases with an increase in frother (MIBC) concentration. The flotation kinetics of the naturally floatable coal (Upper Freeport) and the effect of frother concentration are reported in Table 4 and illustrated in Figures 2-1, 2-2 and 2-3. The results show that the Upper Freeport coal has a rapid flotation rate, Pittsburgh coal a medium rate, and Illinois coal a very slow rate of flotation.

Salt Flotation Tests

Salt flotation test results are presented in Table 5 and shown in Figures 3-1, 3-2 and 3-3. It is apparent that salt has a significant effect on coal flotation. Kinetic data are shown in Table 6 and illustrated in Figures 4-1, 4-2 and 4-3. The relative rank of floatability (evaluated by yield and combustible material recovery) and selectivity (evaluated by pyritic sulfur rejection and separation efficiency) are:

Upper Freeport MVb > Pittsburgh HVAb > Illinois HVCb

Table 3
Natural Floatability and Effect of MIBC on Floatability and Selectivity

Flotation Condition: Wet-Ground, -200 mesh

Collector: 0
Flotation Time: 5 min.

MIBC (g/Mt)	Results & Product Analyses						
	Yield (%)	Ash (%)	PSC (%)	AR (%)	PSR (%)	CMR (%)	EI (%)
Upper Freeport MVb: Flotation pH: 3.7							
0	23.8	4.01	0.27	92.1	96.0	26.0	22.0
50	27.5	4.71	0.27	89.3	95.4	29.8	25.2
250	84.1	6.10	0.41	57.7	78.7	89.8	68.5
500	90.0	6.87	0.44	49.1	75.6	95.3	70.9
1000	94.7	8.16	0.69	36.2	59.7	98.9	58.6
Pittsburgh HVAb: Flotation pH: 3.9							
0	0	0	0	100.0	100.0	0	0
200	11.4	4.69	0.93	95.6	96.2	12.4	8.6
300	42.6	4.49	0.93	84.1	86.0	46.3	32.3
500	62.3	4.84	0.96	75.0	78.8	67.4	46.2
750	74.0	5.15	1.07	68.4	71.9	79.8	51.7
1000	84.4	5.19	1.14	63.7	64.9	91.0	56.9
Illinois HVCb: Flotation pH: 6.7							
0	0	0	0	100.0	100.0	0	0
500	8.4	7.31	0.74	96.2	97.6	9.3	6.9
750	16.2	7.08	0.71	92.9	95.5	18.0	13.5
1000	22.5	7.76	0.67	89.2	94.1	24.8	18.9
1500	42.5	8.65	0.84	77.3	86.1	46.3	32.4

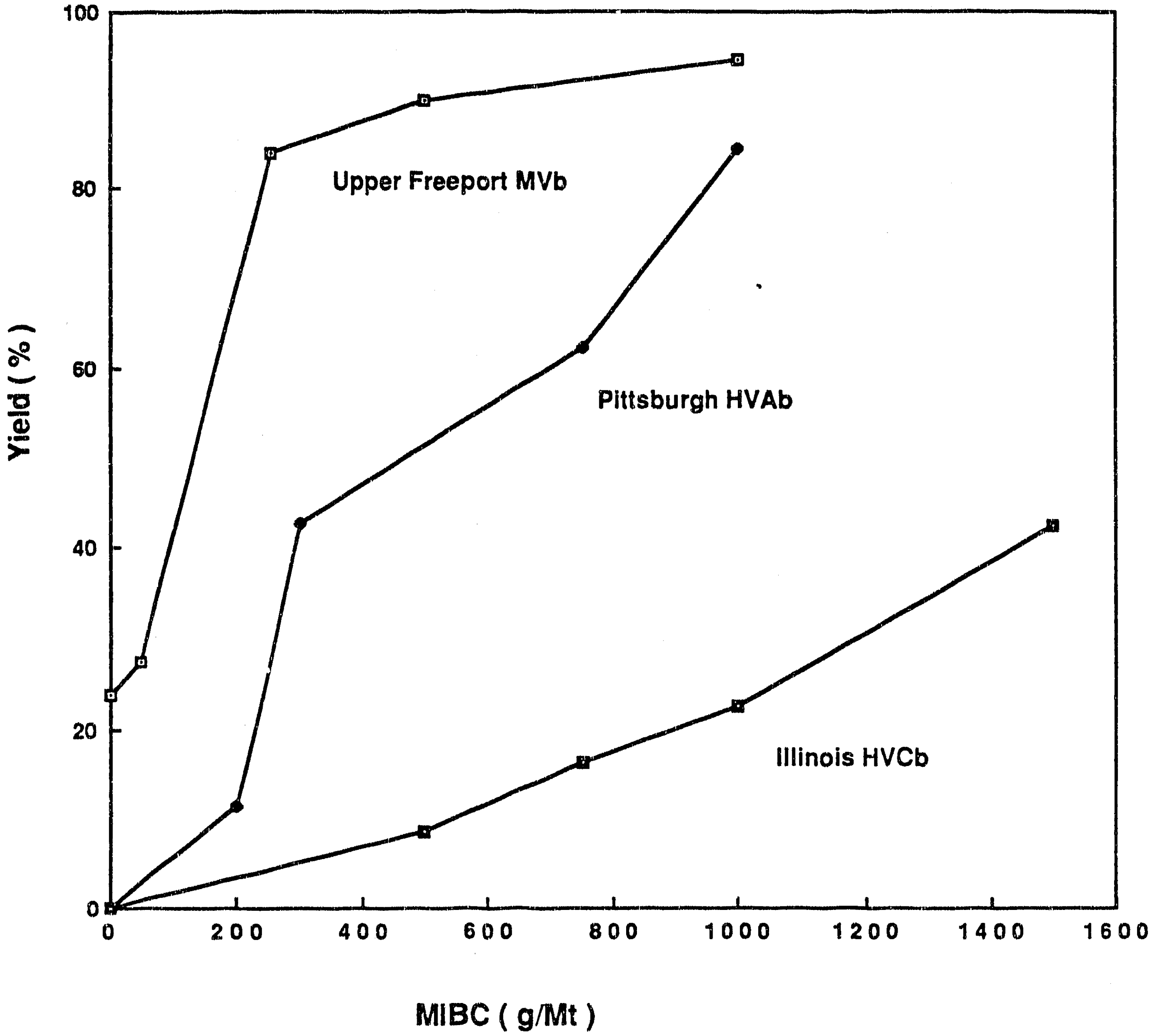

Fig. 1-1 Natural Flotability of Coal Samples and

Effect of MIBC on Flotation

(Size: -200mesh)

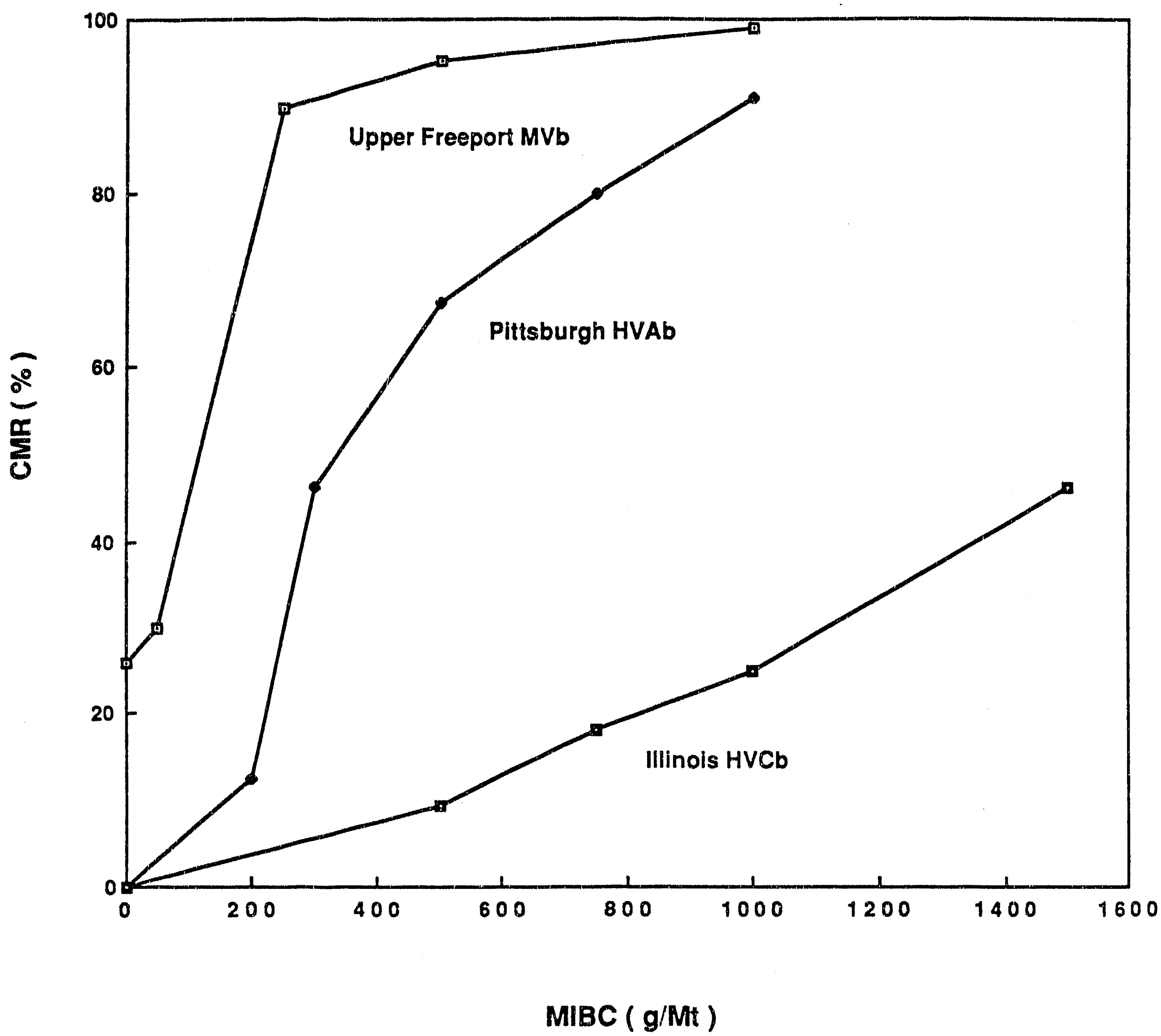

Fig. 1-2 Relation Between the Dosage of MIBC and the Combustible

Material Recovery (CMR)

(size: -200 mesh)

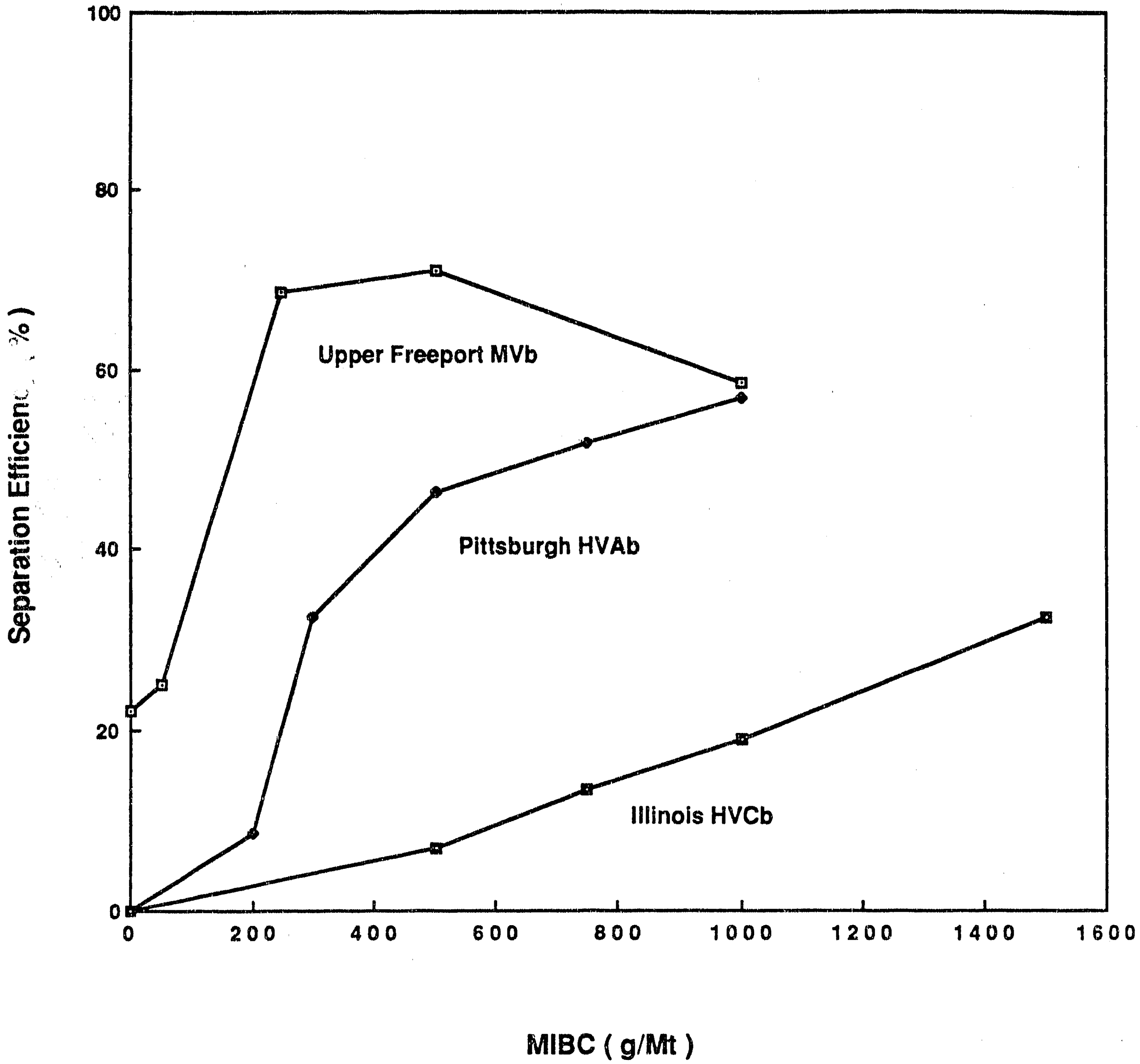

Fig. 1-3 Relation Between Separation Efficiency of Pyritic Sulfur and Dosage of MIBC

(Size: -200 mesh)

Table 4

Flotation Kinetics of Coal Samples Tested by Using MIBC

Flotation Condition: Wet Ground, -200 mesh,

MIBC: 100 g/Mt
Collector: 0

Flotation Time (second)	Yield (%)	Ash (%)	PSC (%)	AR (%)	PSR (%)	CMR (%)	EI (%)
Upper Freeport MVb							
0	0	0	0	100.0	100.0	0	0
15	20.3	5.78	0.42	90.3	94.7	21.8	16.5
30	47.0	5.96	0.42	76.9	87.8	50.3	38.1
60	68.1	6.25	0.45	64.9	81.1	72.7	53.8
90	83.8	6.95	0.52	52.0	73.1	88.8	61.9
150	91.0	7.55	0.60	43.4	66.3	95.8	62.1
300	94.7	8.16	0.69	36.3	59.7	99.0	58.7
Pittsburgh HVAb							
0	0	0	0	100.0	100.0	0	0
15	7.2	4.35	0.91	97.4	97.7	7.8	5.5
30	14.2	4.45	0.91	94.8	95.4	15.4	10.8
60	27.0	4.42	0.92	90.1	91.2	29.3	20.5
90	39.5	4.42	0.94	85.5	86.8	42.9	29.7
150	61.5	4.69	0.98	76.1	78.6	66.7	45.3
300	84.4	5.19	1.14	63.7	65.9	91.0	56.9
Illinois HVCb							
0	0	0	0	100.0	100.0	0	0
15	1.9	5.94	0.59	99.3	99.6	2.1	1.7
30	4.3	6.57	0.56	98.3	99.1	4.8	3.9
60	7.6	7.06	0.59	96.7	98.2	8.4	6.6
90	11.1	7.32	0.62	95.0	97.3	12.3	9.6
150	16.0	7.54	0.65	92.5	95.9	17.7	13.6
300	22.5	7.76	0.67	89.2	94.1	24.8	18.9

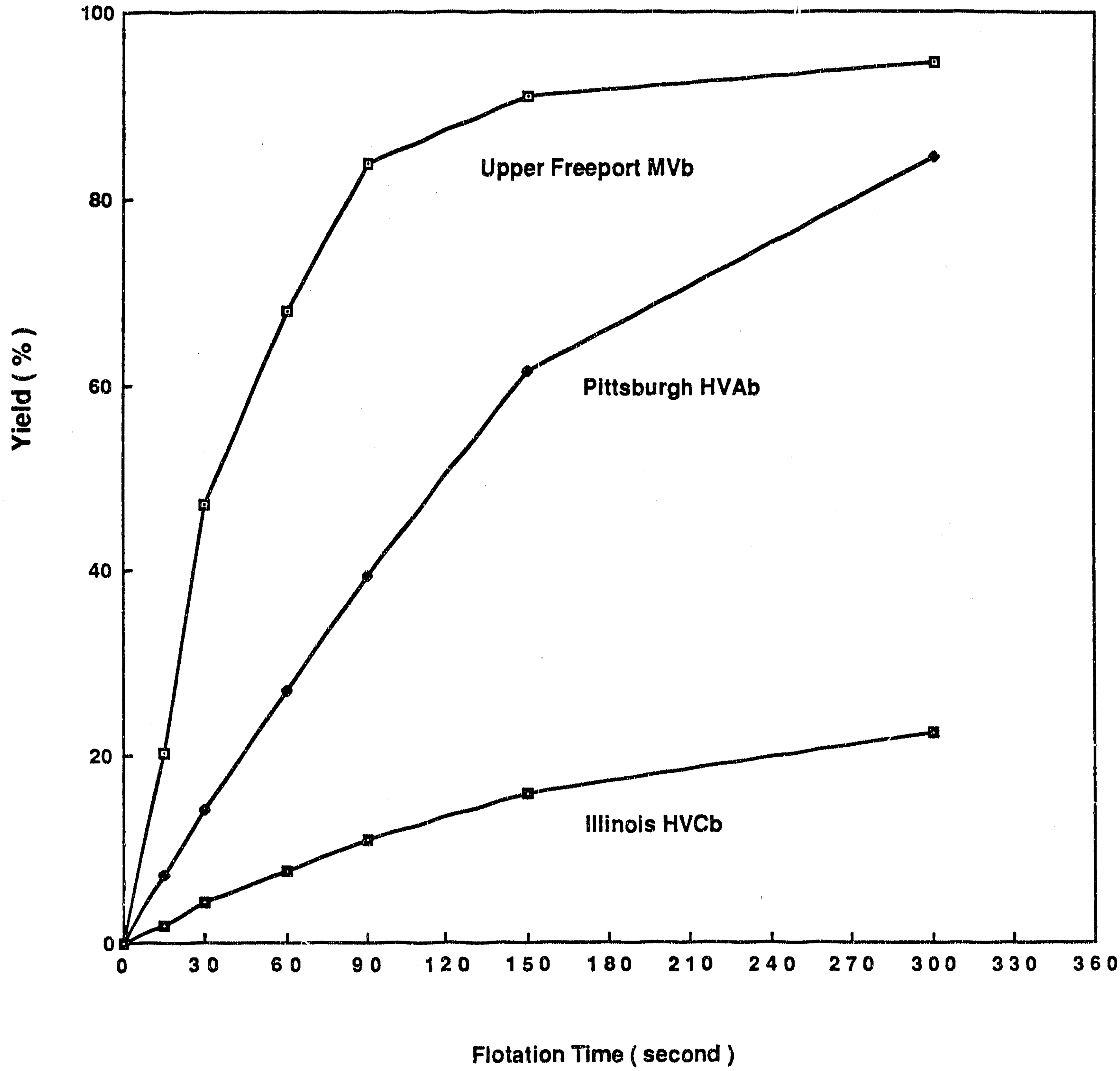

Fig. 2-1 Flotation Kinetics Using MIBC Only

(Size: -200 mesh, MIBC: 1000 g/Mt)

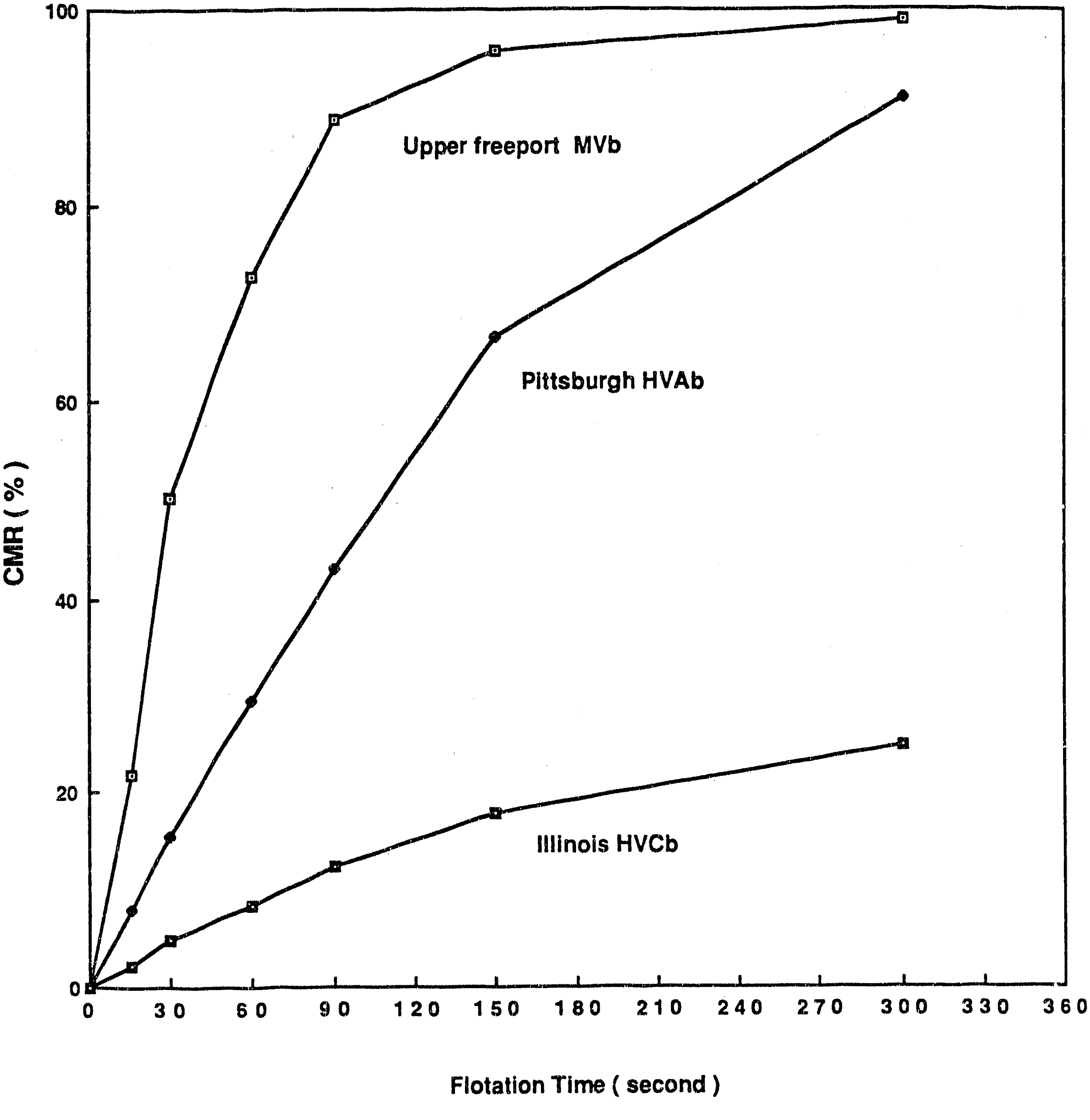

Fig. 2-2 Relation Between Combustible Material Recovery and Flotation Time

(Size: -200 mesh, MIBC: 1000 g/Mt)

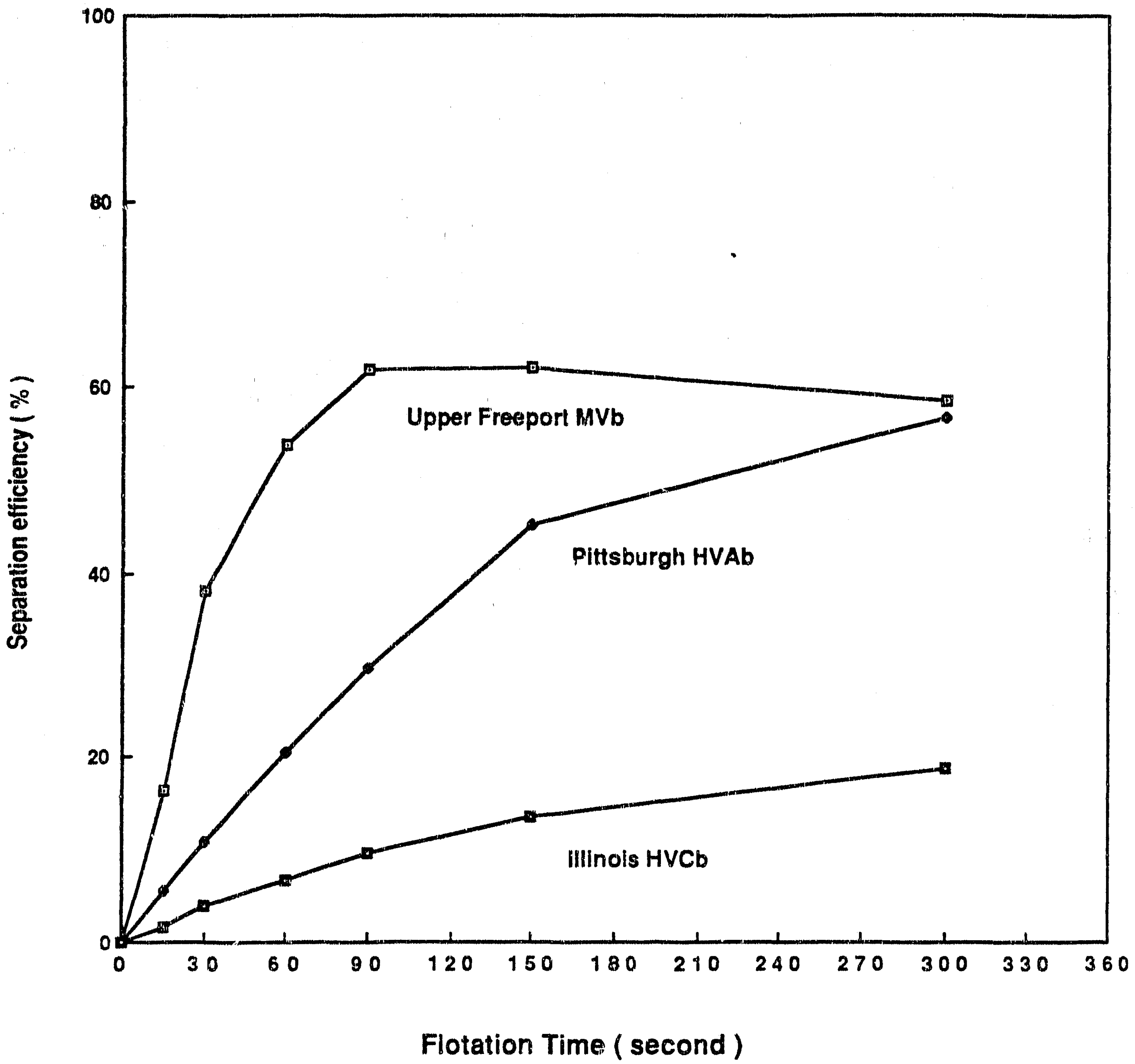

Fig. 2-3 Relation Between Separation efficiency of Pyritic Sulfur and the Flotation Times

(Size: -200 mesh, MIBC: 1000 g/Mt)

Table 5

Effect of Salt (NaCl) on Floatability and Selectivity

Flotation Condition: Wet-Ground, -200 mesh
Collector: 0
MIBC: 0

NaCl wt. %	Result & Product Analysis						
	Yield (%)	Ash (%)	PSC (%)	AR (%)	PSR (%)	CMR (%)	EI (%)
Upper Freeport MVb							
0	23.8	4.01	0.27	92.1	96.0	26.0	22.0
0.5	56.4	5.90	0.54	72.6	81.2	60.4	41.6
1	74.3	5.50	0.45	66.3	79.4	79.9	59.3
3	95.8	8.44	0.91	33.4	46.2	99.8	46.0
6	96.6	9.22	0.90	26.6	46.3	99.8	46.1
Pittsburgh HVAb							
0	0	0	0	100.7	100.0	0	0
0.5	8.0	7.96	1.81	94.7	94.9	8.4	3.3
1	17.3	5.35	1.15	92.3	92.9	18.4	13.2
3	77.5	5.40	1.37	65.3	62.3	83.4	45.7
6	84.7	5.08	1.26	64.3	62.2	91.4	53.6
Illinois HVCb							
0	0	0	0	100.0	100.0	0	0
1	7.0	9.98	0.82	95.7	97.8	7.5	5.3
2	17.0	9.48	0.79	90.0	94.8	18.4	13.2
3	33.6	10.25	0.93	78.7	87.8	36.0	23.8
4	41.0	10.01	0.83	74.7	86.7	44.0	31.7
6	51.0	9.62	0.82	69.7	83.7	55.0	38.7

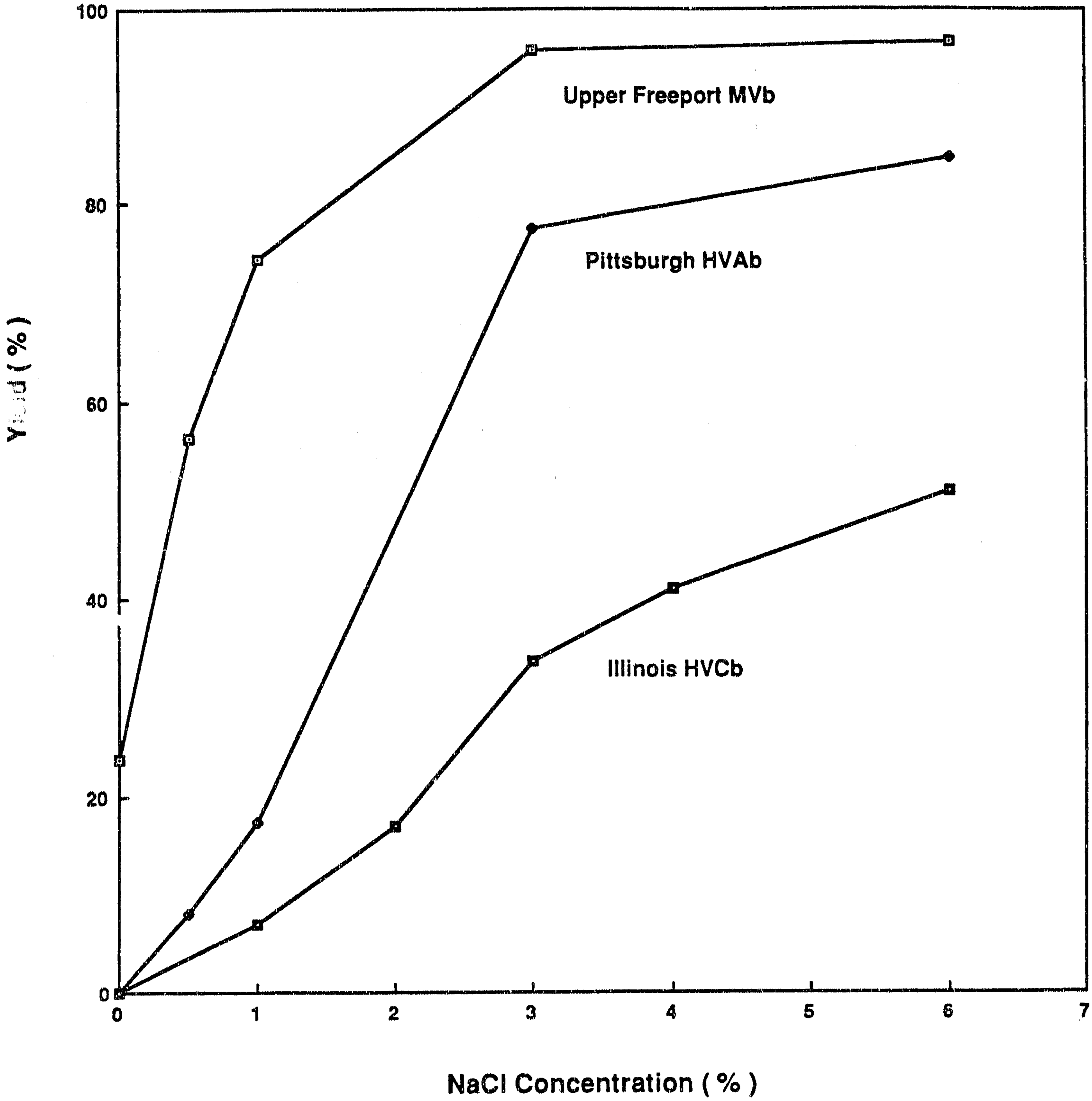

Fig. 3-1 The Effect of Salt (NaCl) for Flotability of Coal

(No Frother & Collector)

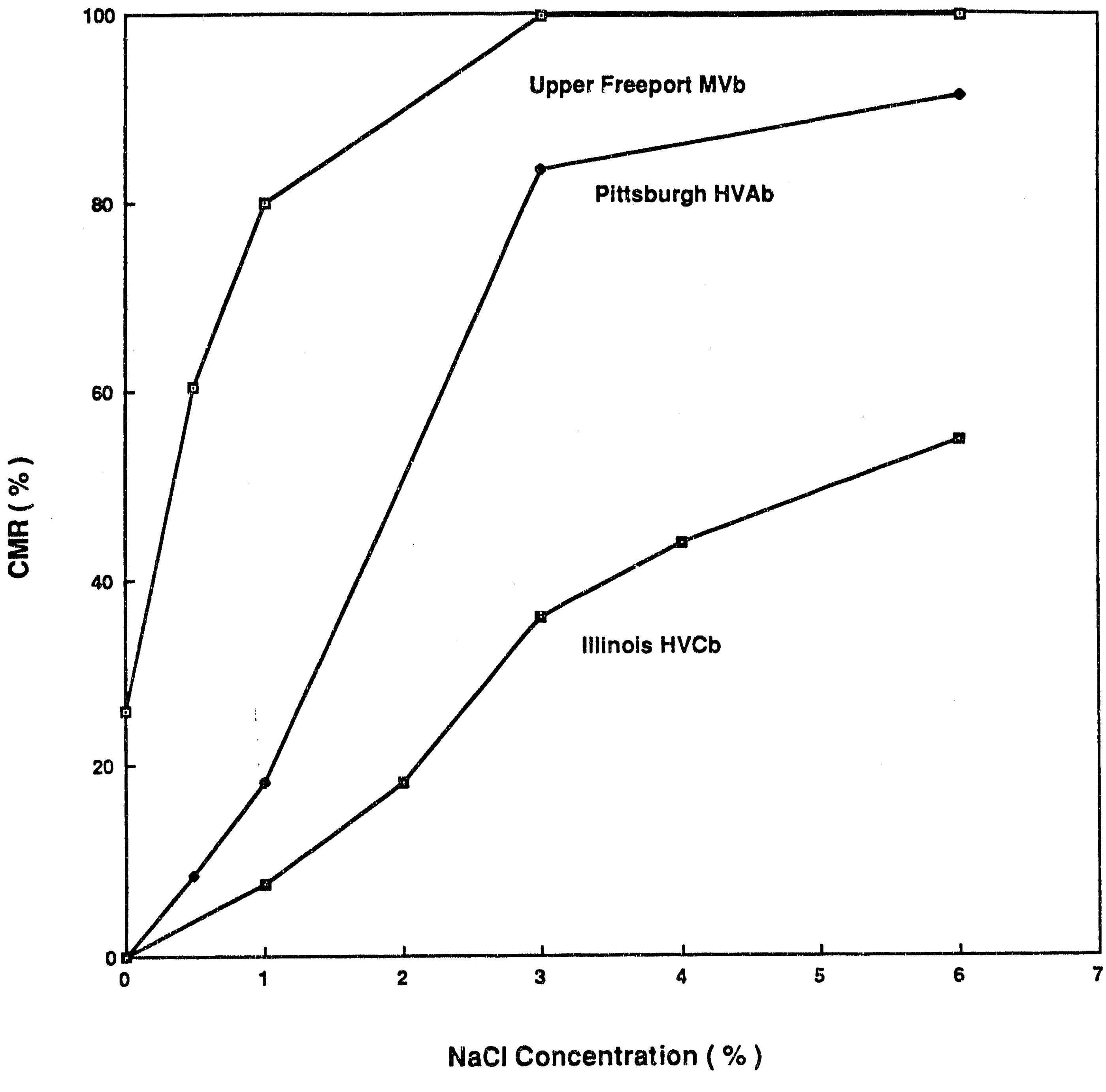

Fig. 3-2 Effect of Salt Concentration on the Combustible Material Recovery

(No Frother & Collector)

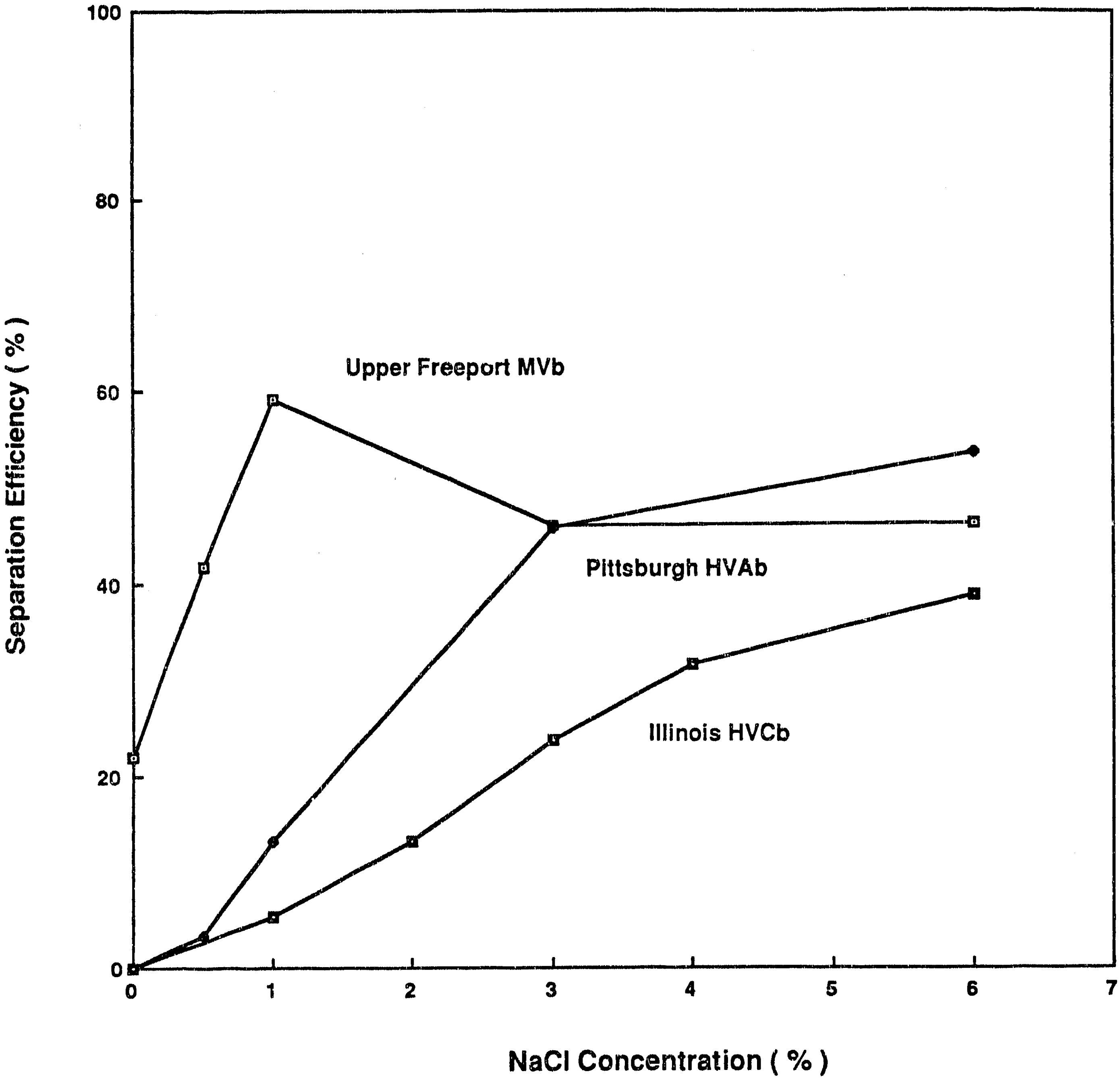

Fig. 3-3 Effect of NaCl Concentration for Separation Efficiency of Pyritic Sulfur

(No Frother & Collector)

Table 6

Kinetics of Salt Flotation

Flotation Condition: Wet-Ground, -200 mesh,

NaCl Concentration: 3%
Collector: 0
MIBC: 0

Flotation Time (second)	Results & Product Analy. Yield (%)	Ash (%)	PSC (%)	AR (%)	PSR (%)	CMR (%)	EI (%)
Upper Freeport MVb							
0	0	0	0	100.0	100.0	0	0
15	22.1	5.29	0.39	90.4	94.7	23.8	18.5
30	33.1	5.25	0.40	85.7	91.8	35.7	27.5
60	57.3	5.42	0.41	74.4	85.5	61.7	47.2
90	70.5	5.75	0.43	66.6	81.3	75.6	56.9
150	89.7	7.28	0.66	46.2	63.5	94.7	58.2
300	95.8	8.44	0.91	33.4	46.2	99.8	46.0
Pittsburgh HVAb							
0	0	0	0	100.0	100.0	0	0
15	4.6	4.37	1.00	98.3	98.4	5.0	3.4
30	10.3	4.31	0.95	96.3	96.5	11.2	7.7
60	20.8	4.41	0.97	92.4	92.8	22.6	15.4
90	30.9	4.45	0.99	88.6	89.2	33.6	22.8
150	50.4	4.67	1.07	80.5	80.9	54.6	35.5
300	77.5	5.40	1.37	65.3	62.3	83.4	45.7
Illinois HVCb							
0	0	0	0	100.0	100.0	0	0
15	2.4	9.56	0.86	98.6	99.2	2.6	1.8
30	4.9	9.76	0.90	97.0	98.3	5.3	3.6
60	9.7	9.98	0.91	94.0	96.6	10.4	7.0
90	15.2	10.16	0.92	90.5	94.5	16.3	10.8
150	20.9	10.21	0.93	86.8	92.4	22.4	14.8
300	33.6	10.25	0.93	78.7	87.8	36.0	23.8

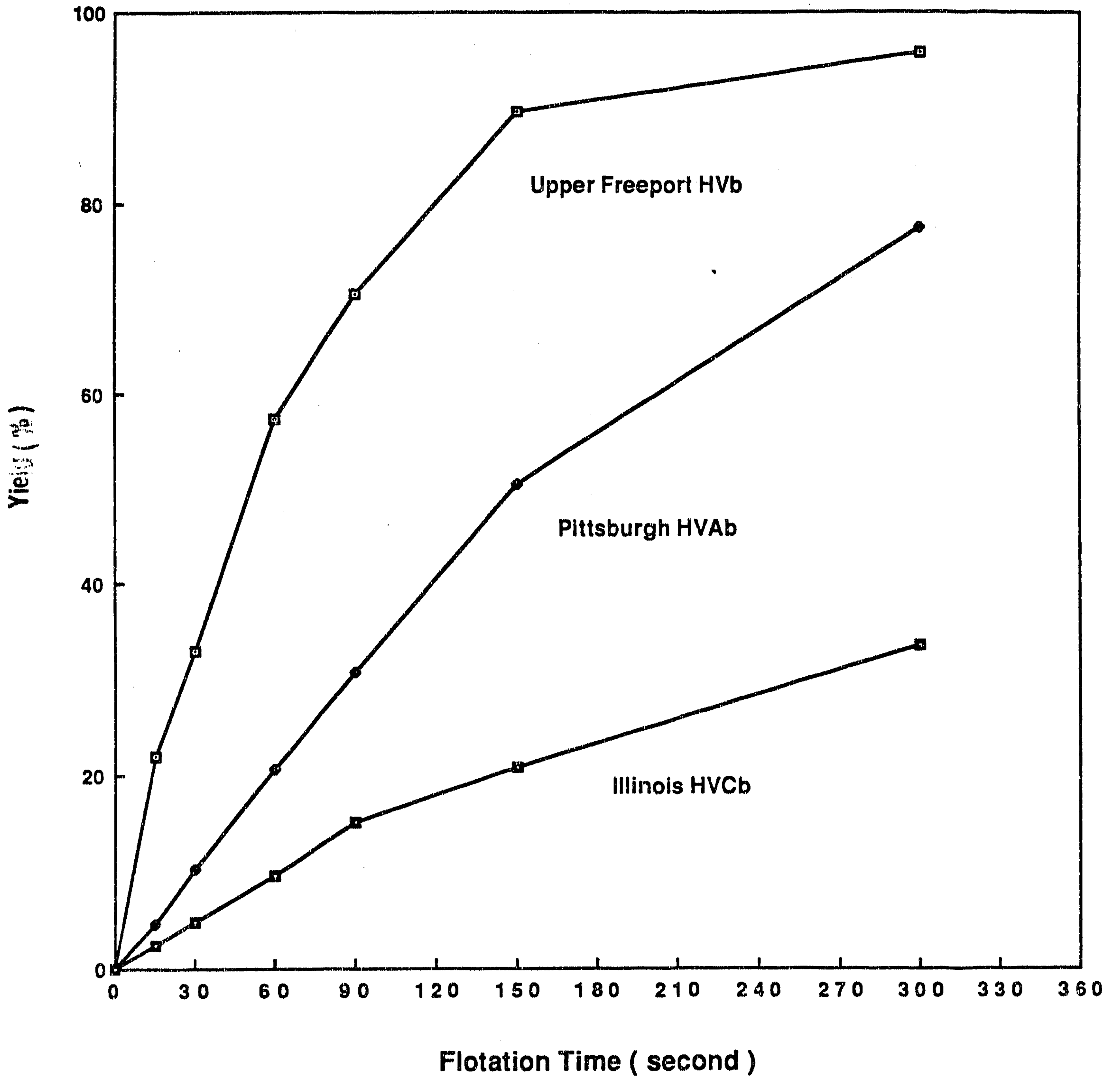

Fig. 4-1 Relation Between the Yield of Salt Flotation and the Flotation Times

(Salt Concentration: 3 %, NO Frother & Collector)

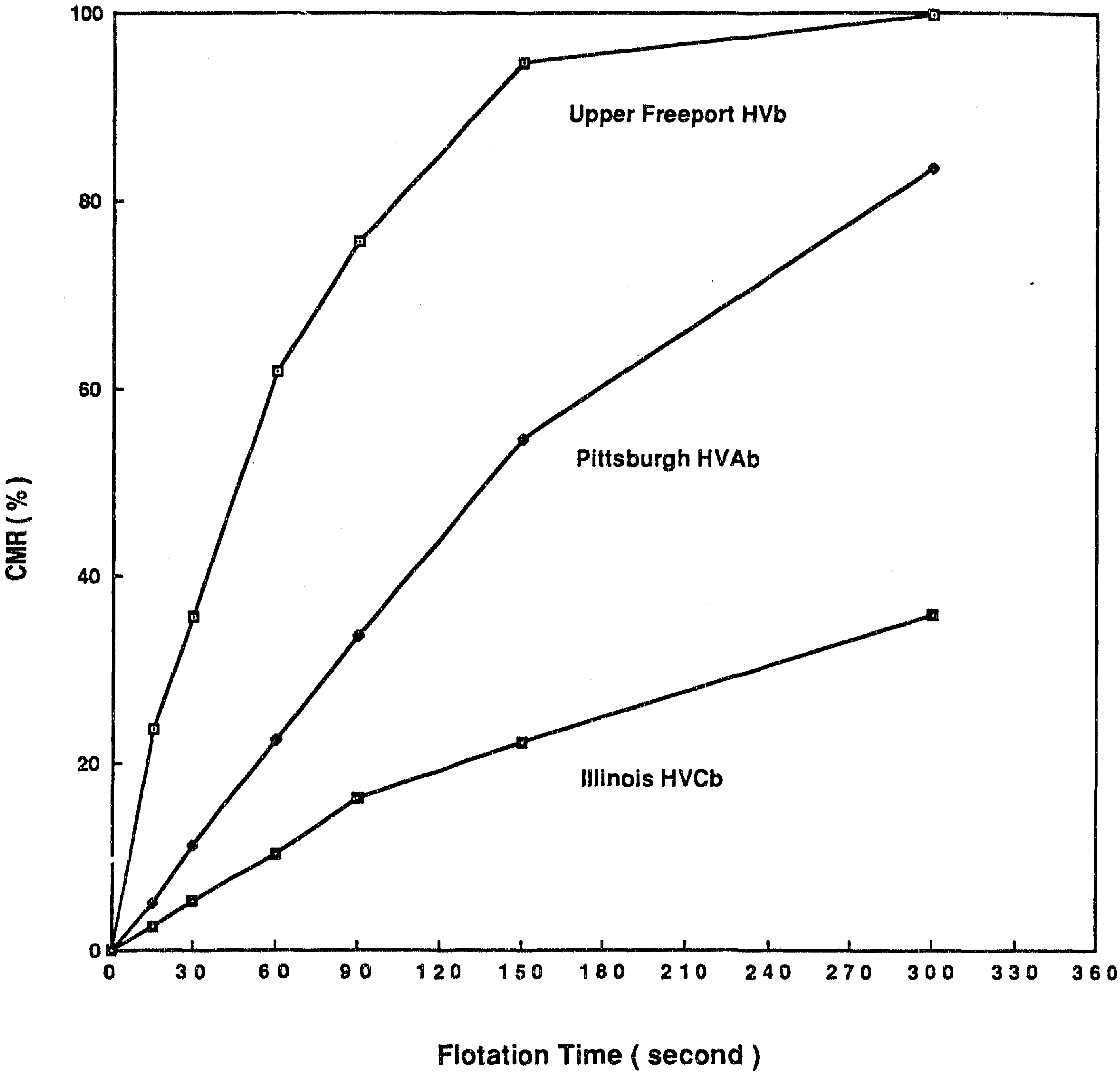

Fig. 4-2 Relation Between the Combustible Material Recovery and the Flotation Time

(Salt Concentraion: 3%, No Frother & Collector)

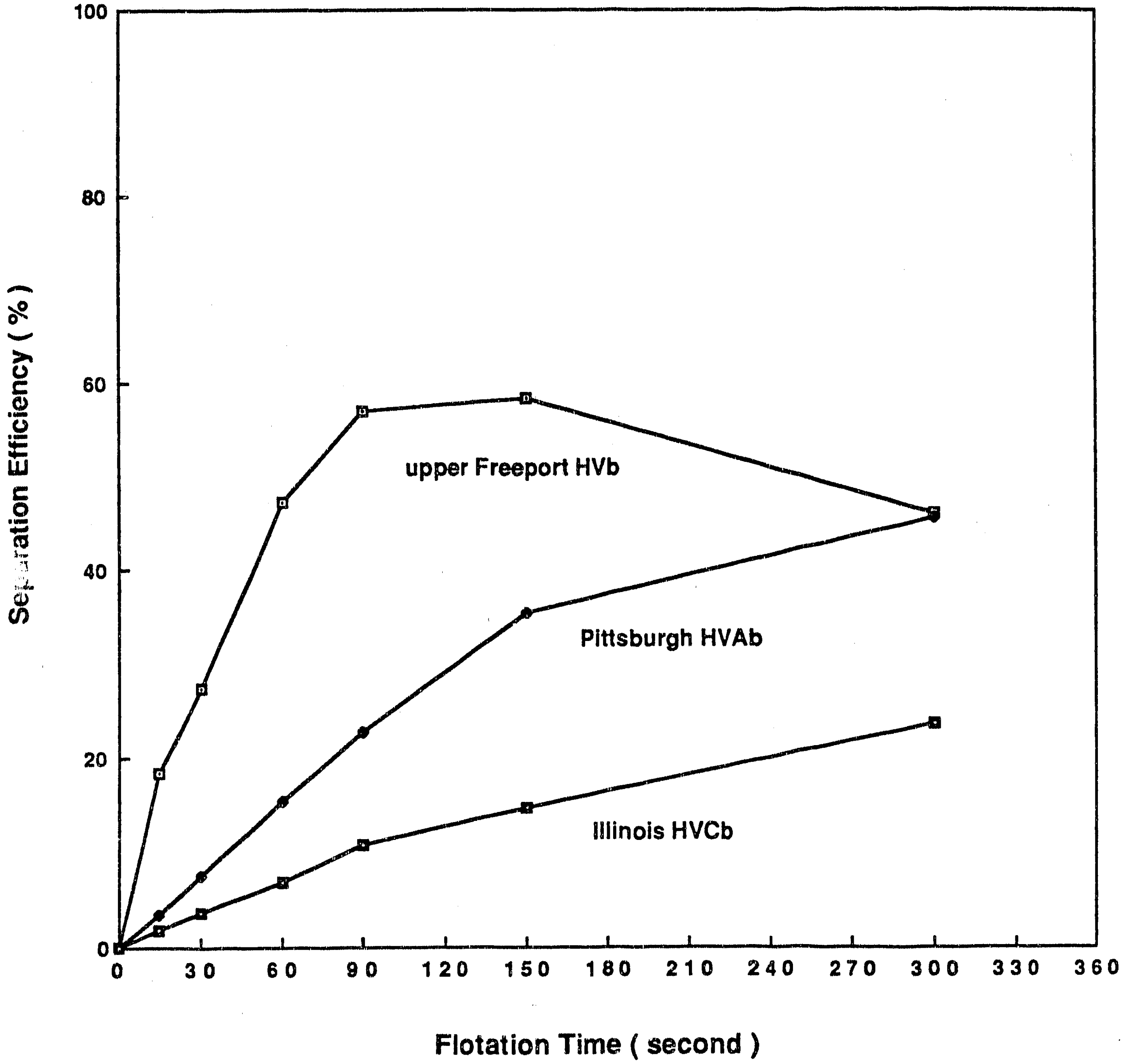

Fig. 4-3 Relation Between the Separation Efficiency of Pyritic Sulfur and the Flotation Time

(Salt Concentration: 3%, No Frother & Collector)

CONCLUSIONS

Test results show that Upper Freeport MVb coal has a definite degree of natural floatability in the absence of collector and frother. The flotation yield obtained was 23.8%. The other two coal samples tested in this study (Pittsburgh HVAb and Illinois HVCb) displayed no natural floatability in the absence flotation reagents.

The flotation yield and the combustible material recovery (CMR) for all three samples increased with increasing additions of frother (MIBC). For an MIBC addition of 1000g/Mt, the Yield and CMR for the three coals were respectively: 94.7% and 98.9% for Upper Freeport MVb; 84.4% and 91.0% for Pittsburgh HVAb; and, 22.5% and 24.8% for Illinois HVCb. Accordingly, the floatability order of the three coal sample was found to be:

Upper Freeport MVb > Pittsburgh HVAb > Illinois HVCb

The separation Efficiency Index for pyritic sulfur (EI) in the presence of MIBC displayed different value for the three coal samples. The EI values of Upper Freeport MVb coal were the highest and the Illinois HVCb was the lowest of the three coal samples. EI values of the Upper Freeport coal increased with MIBC dosage for additions lower than 500 g/Mt of MIBC dosage. An upper MIBC dosage of 500 g/Mt was suitable for Upper Freeport coal. However, the EI values of the other two coal samples continued to increase for MIBC dosages greater than 500 g/Mt.

Flotation kinetics test results indicated that 150 seconds flotation time for Upper Freeport coal sample was sufficient. Extended flotation times adversely affected the separation efficiency.

The experimental results of salt (NACl) flotation showed that under the salt flotation conditions of this study (6% NaCl) flotation Yield and CMR values were respectively: 96.6% and 99.8% for Upper Freeport MVb; 84.7% and 91.4% for Pittsburgh HVAb; and, 51.0% and 55.0% for Illinois HVCb in the absence of any flotation reagents. Their floatability rank was the same as that observed for flotation results using MIBC; namely,

Upper freeport MVb > Pittsburgh > Illinois HVCb

Pyritic sulfur separation efficiency EI of Upper Freeport MVb coal sample was highest at a NaCl concentration of 1%. Above and below this value it was found to decrease. In the case of the other two coal samples, EI values were found to increase with increasing concentrations of NaCl.

Salt flotation kinetics test results show that 150 seconds of flotation time for the Upper Freeport sample was adequate. As in the case of flotation in the presence of MIBC, extending flotation time lead to a decrease in separation efficiency since the time dependent flotation of ash and pyritic sulfur increased for Upper Freeport. In the case of the other two coals studied, the separation efficiency continued to increase with extended flotation time.

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