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Quarterly Report

**A NEW APPROACH IN ULTRAPURIFICATION OF COAL
BY SELECTIVE FLOCCULATION**

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SUMMARY

The mathematical and computational model developed for predicting optimum selectivity achievable with a polymer has been successfully verified experimentally. Adsorption density of polymer SF 362 on coal and coal pyrite was measured using the depletion method. Values of Φ_R (active sites ratio) and Θ (fractional surface coverage) were computed using the adsorption data. The mathematical model predicted a low selectivity in separating coal pyrite from coal which was also verified by selective flocculation tests. FTIR surface area and size distribution analysis indicated that mineral matter associated with both coal and coal pyrite samples can adversely affect the performance of the polymer in the selective flocculation process.

Adsorption tests are in progress to identify experimental conditions (dispersant, pH, flocculent) yielding a higher value of Φ_R so that the desired separation of coal pyrite from coal can be obtained.

INTRODUCTION

Two polymers SF 362 and SF 16 were determined to exhibit selectively in separating coal pyrite from coal. It was decided to conduct adsorption tests to generate the required parameters Θ and Φ for the mathematical model to predict the optimum selectivity achievable with SF 362.

Adsorption Tests: Adsorption tests were conducted using the depletion method. A given amount of material was contacted with a polymer solution of known concentration. After a predetermined time, the material was centrifuged out, and the concentration of residual polymer was determined using a viscosity measurement technique.

Equilibration Time: Adsorption measurements as a function of time were conducted to determine the equilibration time. It is clear from Figure 1 that equilibration was reached in 16 hours.

Saturation Adsorption (for Φ_R): Saturation adsorption tests to estimate the relative number of active sites on coal and coal pyrite were conducted. It is evident from results presented in Figure 2 that coal pyrite had a higher value of Φ as compared to coal.

Nonequilibrium Adsorption (for Θ): To determine the fractional surface coverage ' Θ ', adsorption measurements for a polymer/solid contact time of 5 minutes were conducted. It was determined that Θ was 0.23 for coal and 0.18 for coal pyrite.

MODEL PREDICTIONS

The values of Φ and Θ generated were used in the model to predict the optimum selectivity. The value of Φ_R (Φ coal pyrite/ Φ coal) is 2. It was shown for another system that the value of Θ does not affect the S.I. or recovery except for extremely small or large values of Θ . For the purpose of simulation Θ was taken as 0.5 for both coal and coal pyrite. In a model simulation carried out for various Φ_R values the trend of Φ_R equal to 2 is also presented (see Figure 3). The computational model under present conditions yielded a selectivity index of less than 10 and a recovery of about 90% (see Figures 3 and 4).

ADSORPTION OF SF 362

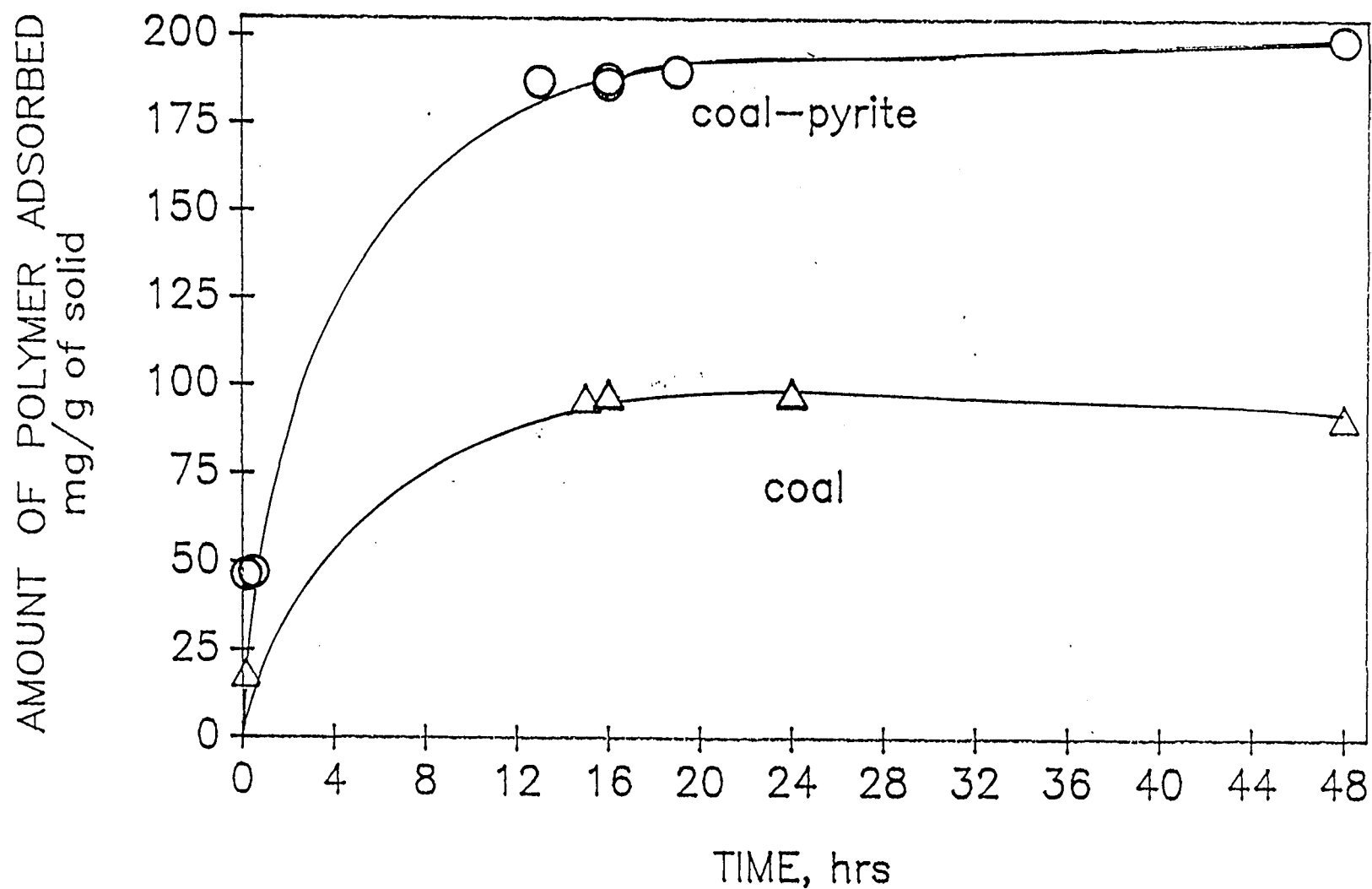


Figure 1. Amount of Polymer (SF 362) Adsorbed on Coal and Coal Pyrite as a Function of Equilibration Time

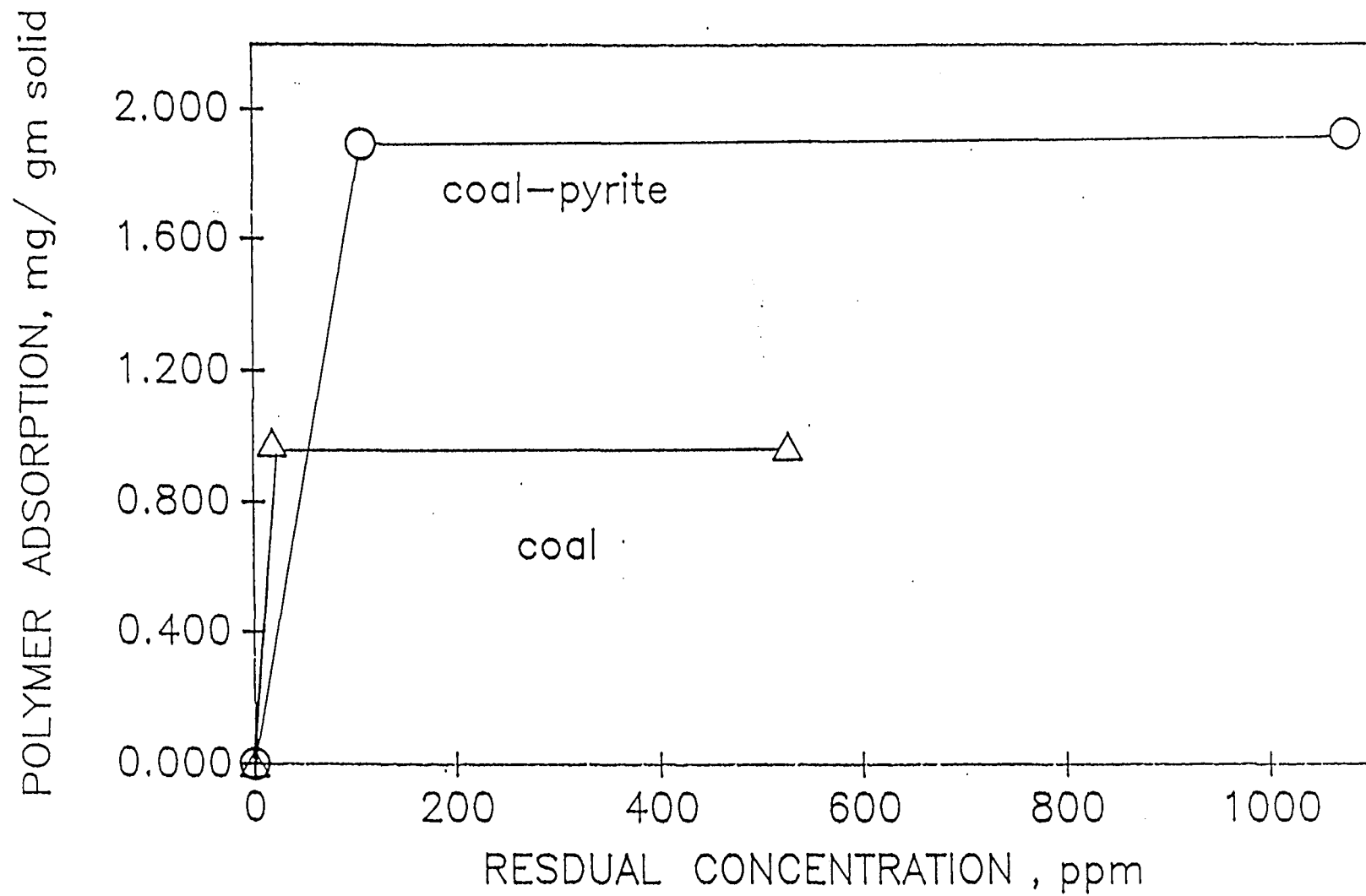


Figure 2. Equilibrium Adsorption of Polymer SF 362 on Coal & Coal Pyrite

COAL - COAL PYRITE SYSTEM
TOTAL NUMBER OF PARTICLES 10,000,000

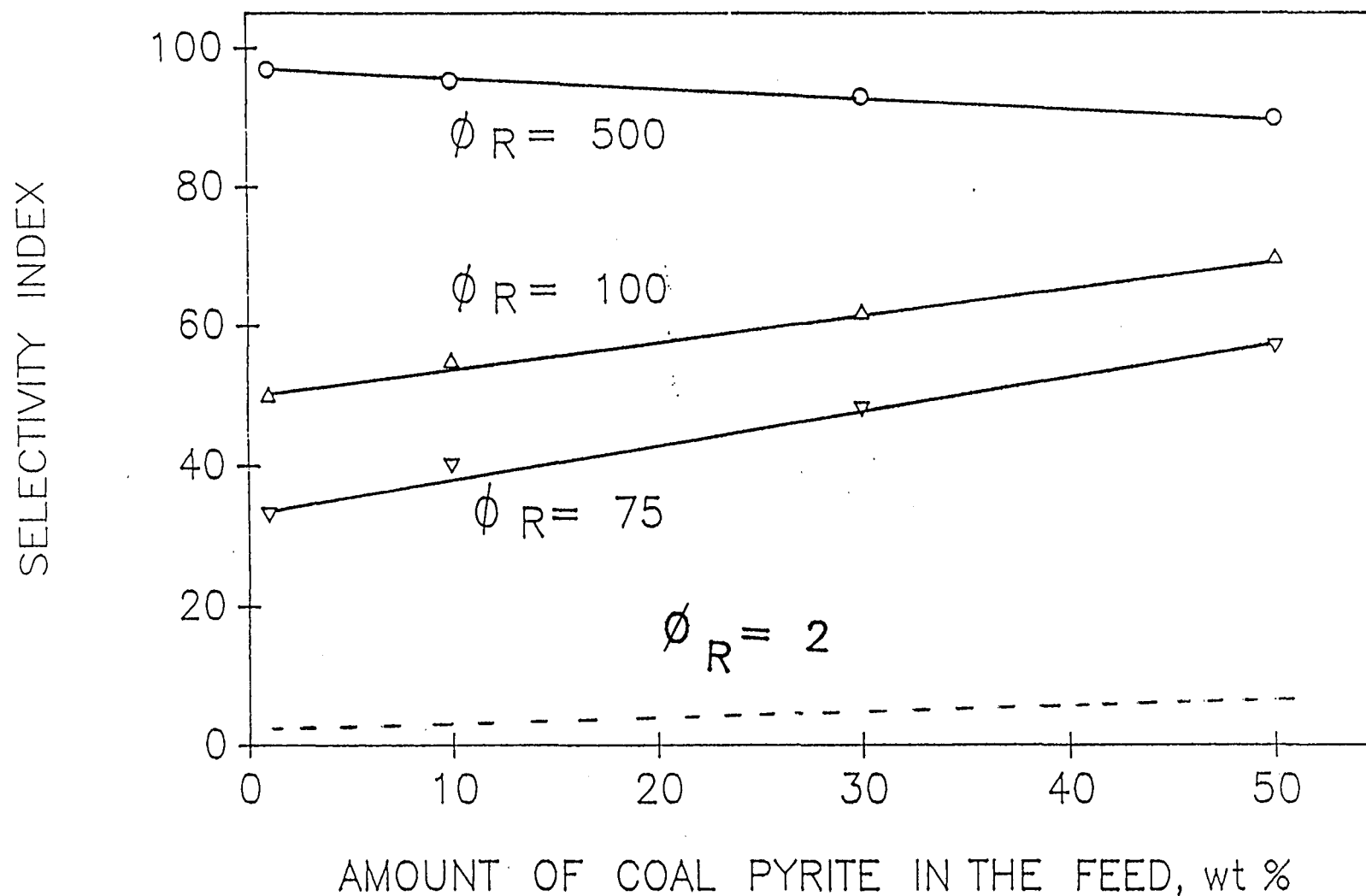


Figure 3. Effect of Amount of Coal Pyrite in Feed on Selectivity Index

COAL - COAL PYRITE SYSTEM
TOTAL NUMBER OF PARTICLES 10,000,000

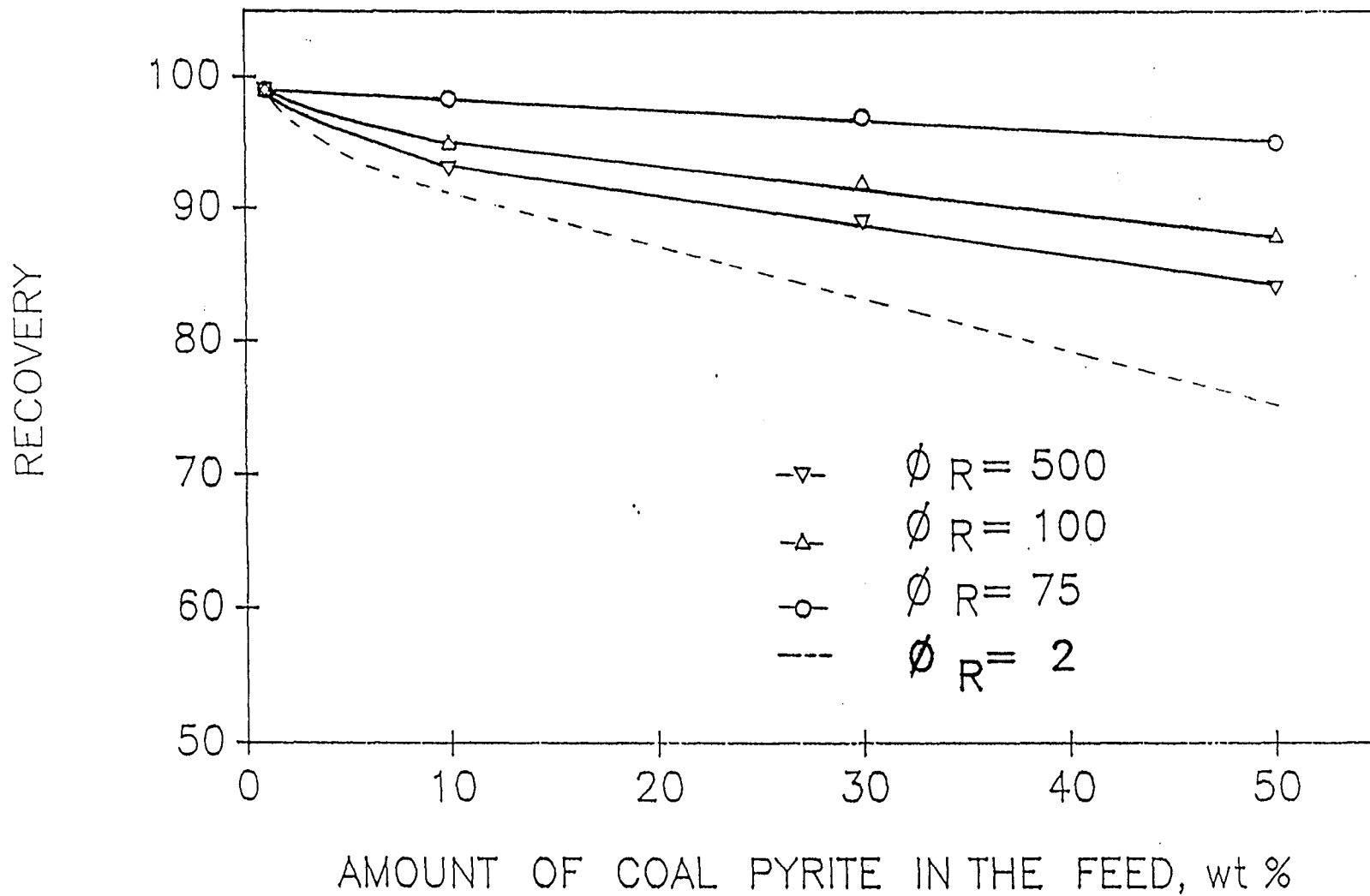


Figure 4. Effect of Amount of Coal Pyrite in Feed on Recovery

SELECTIVE FLOCCULATION TESTS

Selective flocculation tests were conducted with 90:10 and 95:5 coal:coal pyrite mixtures using SF 362 as the flocculent. Results presented in Table 1 agree with the model predictions. The less than expected selectivity achieved was attributed to the surprisingly low value of Φ_R . It was decided to examine the surface area and the surface components of the coal, coal pyrite samples to determine the reasons for the low Φ_R values.

Surface Area Measurements: Results of surface area measurements by BET are presented in Table 2. The specific surface area of coal and coal pyrite are determined to be ~ 1.55 and $\sim 7 \text{ m}^2/\text{g}$ indicating that either there is a difference in their particle sizes or the pyrite sample is highly porous. The d_{50} of coal and coal pyrite, as determined by HORIBA particle size analyzer, was found to be $30\mu\text{m}$ and $5\mu\text{m}$ respectively indicating higher surface area of coal pyrite as determined experimentally.

FTIR Analysis: FTIR analysis was performed on dried coal and coal pyrite samples using a diffuse reflection stage. From the results presented in Figures 5 and 6 it is clear that both coal and coal pyrite have mineral matter, especially kaolinite⁽⁹⁾, associated with them.

To characterize the adsorption of the polymer SF 362 on coal and coal pyrite, a number of spectra's were obtained in a liquid cell with a ZnSe crystal. Figure 7 shows spectra's of polymer, coal and coal with polymer (SF 362) while Figure 8 illustrates the spectra's of SF 362, coal pyrite and coal pyrite with SF 362. In Figures 7 and 8 the kaolinite peak is very dominant unlike the coal spectra for diffuse reflectance, indicating coating of the ZnSe crystal by kaolinite.

Another important observation in Figure 7 is the "blunting" of the peak around the kaolinite peak for the spectra of coal plus polymer SF 362 (wave number, 1035). This could imply either adsorption of the polymer on kaolinite or it could be an artifact introduced during data analysis (subtraction of spectra). However, a strong influence of kaolinite on SF 362 adsorption in both the coal and coal pyrite cannot be ruled out. Moreover, due to the inherent complexities of the coal and coal pyrite spectras it is

TABLE 1

Separation of Coal and Coal Pyrite Using SF 362 as the Flocculent

Feed Coal/Coal Pyrite	Polymer Dosage kg/ton	pH	Settling Time Min	Coal Grade % Coal	Coal Yield, %		Selectivity Index	
					Experimental	Expected	Experimental	Expected
90/10	0.01	5.7	4	90.4, 90.27	96.84	92	4., 2.7	3
90/10	0.01	3.0	4	90.00	93.79	-	0	-
5/95	0.1	5.9	4	8.15	74.66	-	3.31	-

TABLE 2

Physical Characteristics of Coal and Coal Pyrite

Sample	Specific Surface Area m ² /gm		Pore Size, A°		Pore Volume, cm ³ /g
	Single Point	Multiple Point	Average	Maximum	
Coal	1.5411	1.6348	65	1150	5.27 X 10 ⁻³
Coal Pyrite	6.705	7.201	64.5	1276.4	2.34 X 10 ⁻²

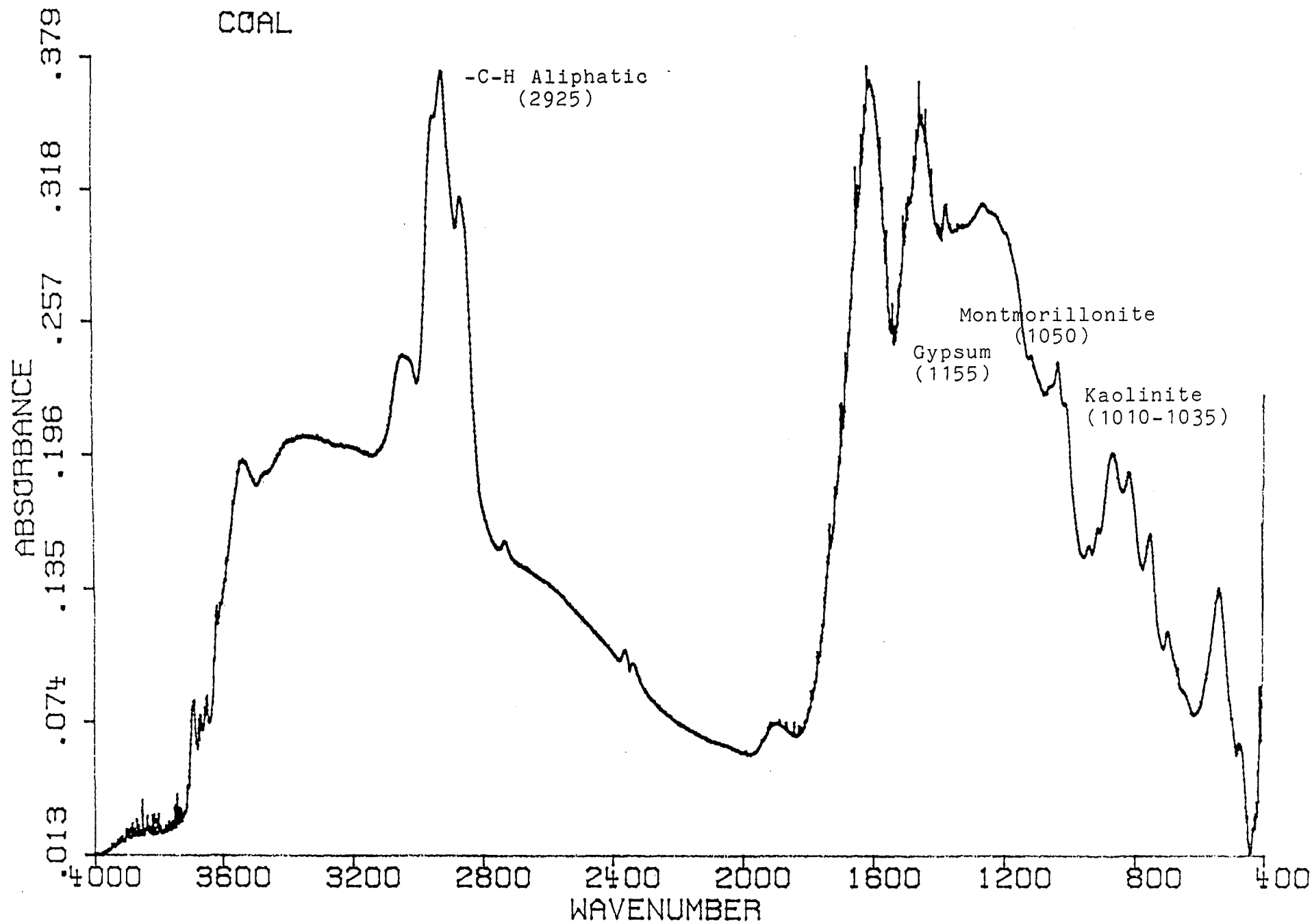


Figure 5. Diffuse Reflectance Spectra of Coal

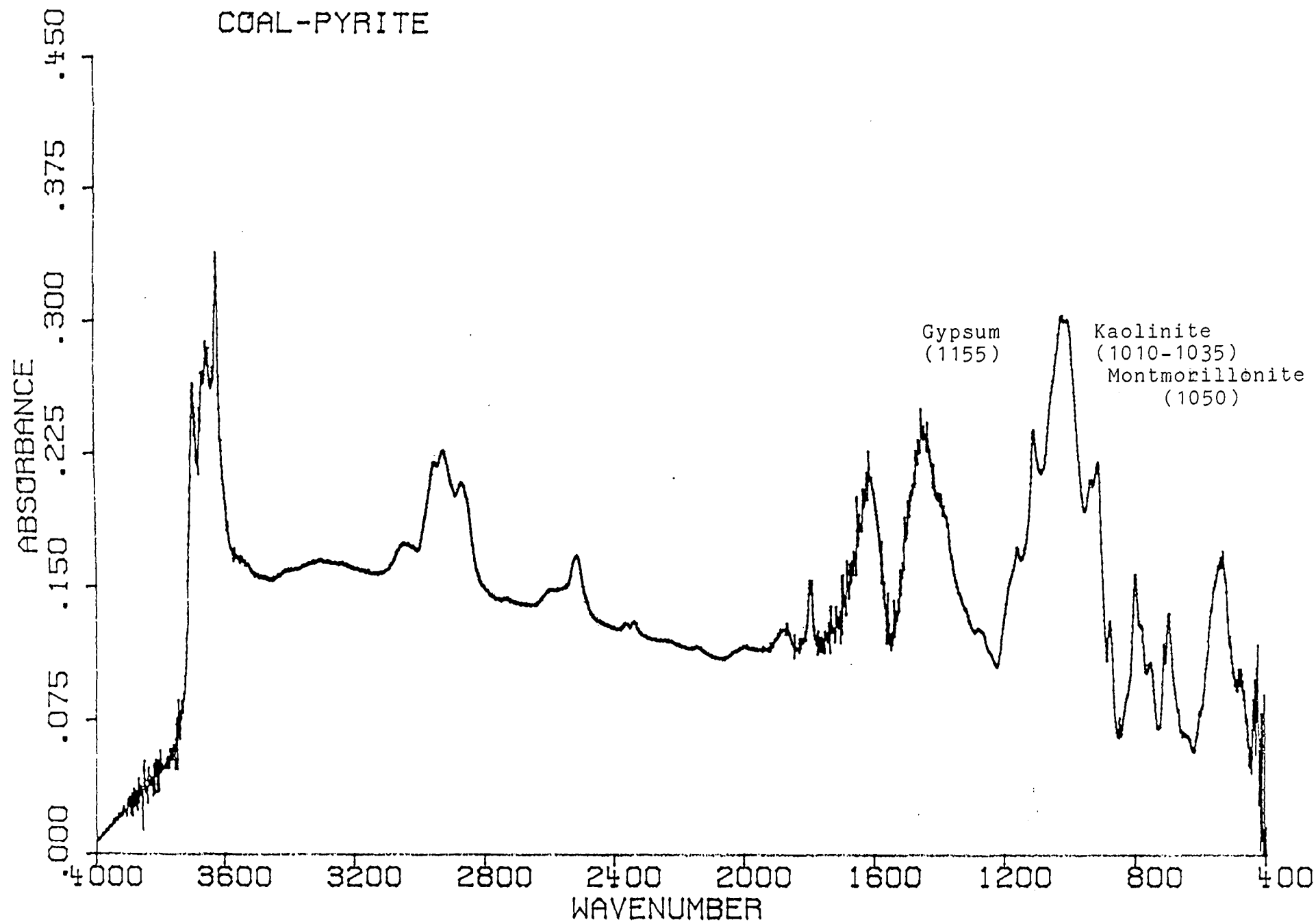


Figure 6. Diffuse Reflectance Spectra of Coal Pyrite

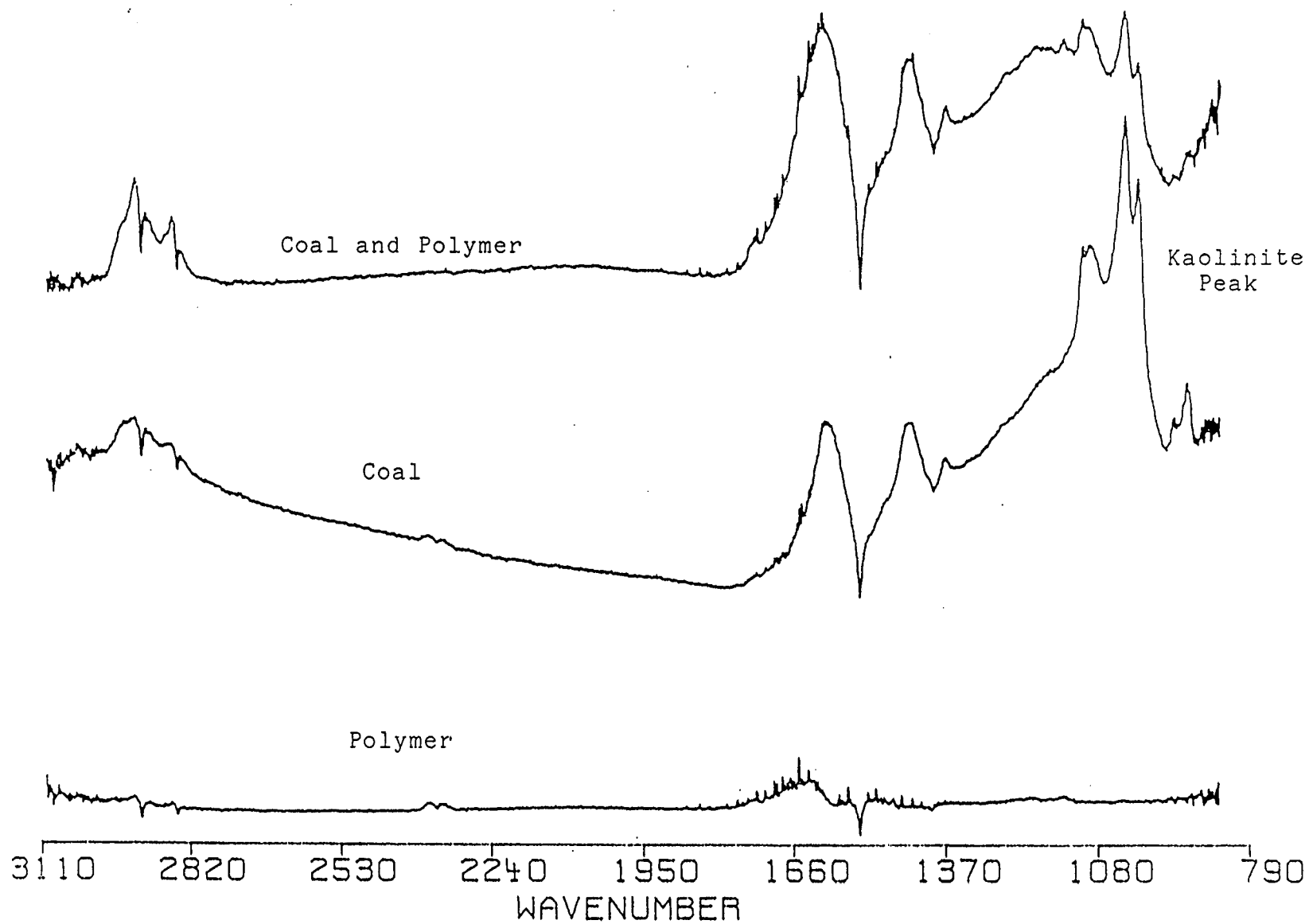


Figure 7. Transmission Spectra of Coal With and Without Polymer (SF 362)

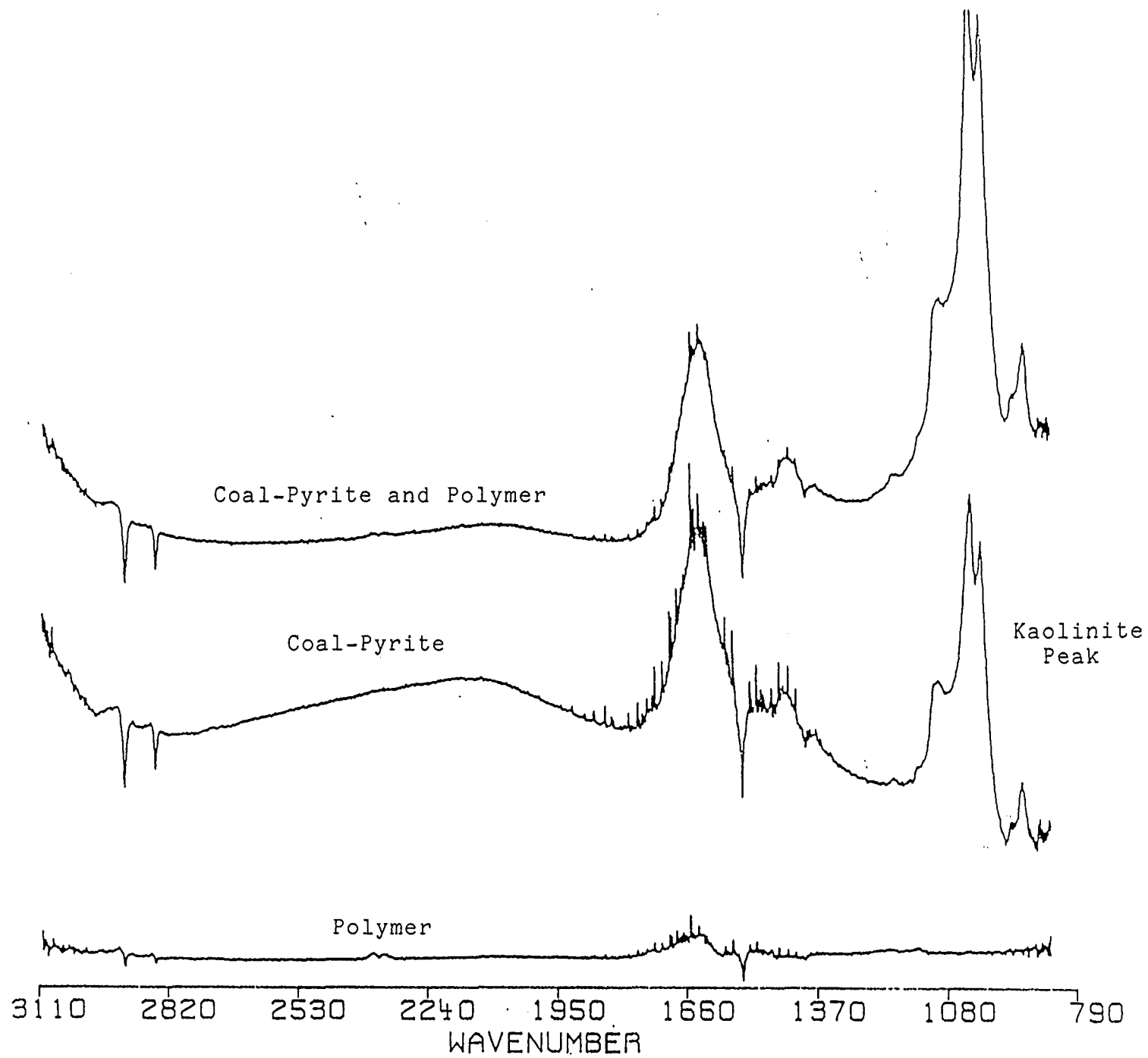


Figure 8. Transmission Spectra of Coal Pyrite With and Without Polymer (SF 362)

difficult to say how much of the polymer adsorbed on coal or coal pyrite and how much on kaolinite. Therefore, it is possible that the presence of kaolinite and other matter associated with coal and coal pyrite governs the overall adsorption behavior of SF 362 and is responsible for relatively low Φ_R values.

It was decided to further examine the nonionic polyacrylamide (polymer SF 16) which had indicated selectivity during flocculation tests. A summary of the selective flocculation test results presented in Table 3 again show a potential of SF 16 for achieving the desired separation.

FUTURE WORK

Adsorption and flocculation tests with SF 16 are in progress. Other polymers such as the hydrophobic polymer from Calgon Corp. WCL 762 (FR-7A) are also being reexamined since they have been reported to selectively flocculate coal in a coal-coal pyrite mixture⁽²⁾. Experimental conditions yielding a higher Φ_R (active sites ratio) will be identified and model predictions of optimum selectivity will be verified by conducting selective flocculation tests under given experimental conditions.

TABLE 3

Separation of Coal and Coal Pyrite Using SF 16 as the Flocculent

Feed Coal/Coal Pyrite	Polymer Dosage kg/ton	pH	Settling Time min	Coal		Selectivity Index
				Yield, %	Grade, %	
90/10	1.0	5.7	10	8.09	75.25	-
90/10	1.0	5.7	4	86.52	90.32	3.2
90/10	1.0	3.25	4	94.8	92.05	20.5
95/5	1.0	5.7	10	64.75	93.24	-
95/5	1.0	9.25	4	88.98	95.2	4.0
95/5	1.0	2.9	4	98.05	95.34	6.8
5/95	0.1	5.9	4	48.53	13.06	8.45

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- (2) Attia, Y., Flocculation in Biotechnology and Separation Systems, Editor Elsevier Science Publishers, Amsterdam 1982