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Flocculation and Filtration Dewatering of Coal Slurries Aided by a Hydrophobic Polymeric Flocculant

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Abstract

This study investigated the adsorption behavior of a totally hydrophobic polymer, FR-7A, and its role in the flocculation and filtration of fine coal slurries. Adsorption of FR-7A on coal, pyrite, and shale minerals revealed that: (a) FR-7A had a higher adsorption affinity to coal and pyrite than that on shale; (b) an acidic slurry condition favored unselective adsorption of FR-7A on coal minerals, leading to improved total flocculation and filtration of fine coal slurries, while alkaline pH and the presence of SMP favored selective adsorption and flocculation of coal from associated minerals in the slurry; and (c) FR-7A aided the flocculation of coal slurries and improved the moisture removal by filtration from 42.4 to 37%.

INTRODUCTION

The use of polymeric flocculants as aids for solid/liquid separation has been studied and applied widely in many industries (*1–4*). The adsorption of a polymeric flocculant on fine particles in suspension causes the aggregation of these particles, which significantly increases the rate of their separation from suspending liquids.

FR-7A is believed to be a totally hydrophobic polymeric flocculant. Under favorable adsorption conditions, excellent separation of coal from associated silicates can be achieved through selective flocculation by using this polymer (*5*). The high affinity of this polymer to coal is believed to be due to hydrophobic "bond" association. However, under different conditions, FR-7A adsorption becomes less selective and results in total flocculation of coal slurries. Since this polymer causes fine particles to agglomerate into flocs, the filtration rate is expected to increase. In addition, as this polymer is also hydrophobic and is dispersed as an oil-in-

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water emulsion, the final moisture content of the filter cake is expected to be lower than that achieved with hydrophilic polymeric flocculants.

The objectives of this study were: 1) to investigate the adsorption behavior of FR-7A on coal, pyrite, and shale; and 2) to determine the effects of FR-7A on flocculation and filtration dewatering of fine coal slurries.

EXPERIMENTAL TECHNIQUES

Mineral Samples

Samples for Adsorption Tests

The two raw coals used for adsorption studies were from the Upper Freeport seam, obtained from the Babcock and Wilcox Research and Development Division, Alliance, Ohio, and the Pittsburgh No. 8 seams, supplied by the R & F Coal Co., Lamira Preparation Plant, Warnok, Ohio. Each of them was precleaned by gravity separation using a heavy liquid, 1,1,1-trichloroethane, with a specific gravity of 1.33, to reject the majority of coarse size impurities (shale and pyrite). The float coal products were washed by acetone and then dried in an oven at about 105°C for 5 h to remove any residual organic liquid. The coals were dry-ground in a stainless steel attritor. The cumulative weight of the size below 25 μm was 50% for the Upper Freeport coal and 75% for the Pittsburgh No. 8 coal.

Pure pyrite (FeS_2) crystals from Hunzala, Peru, and a sample of arigillaceous shale minerals were supplied by Ward's Science Establishment, Inc., Rochester, New York. The samples of pyrite and shale were dry-attrited to about 80% by weight below 25 μm (−500 mesh).

Table 1 lists the analytical data of these samples. The surface areas of the samples were determined by an Accusorb Model 2100E produced by

TABLE 1

Analyses of the Samples Used in Polymer Adsorption Tests

Materials	Ash (%)	Total sulfur (%)	Surface area (m^2/g)	Mean diameter (μm)	pzc at pH
Upper Freeport coal (precleaned)	3.5	1.80	2.62	14.62	6.7
Pittsburgh No. 8 coal (precleaned)	4.2	1.67	3.50	9.46	7.1
Pyrite crystal	—	—	3.58	4.12	7.1
Arigillaceous shale	—	—	7.42	4.36	2.1

Micromeritics Corp., and the mean diameters of the particles were measured by using a Microtrac II Particle Size Analyzer made by Leeds & Northrup Co. The surface electrical potentials of the minerals (see Fig. 1) were measured by using a Laser Zee Model 501 apparatus, manufactured by PenKem Inc.

The measured pzc (point of zero charge) of each mineral sample was very close to that reported in the literature [e.g., pzc's of anthracite and bituminous coals were between pH 5 and 8 (*6*), pzc of pyrite was at pH 6.2–6.9 (*7*), and pzc of shale was at about pH 1.0–2.0 (*8*)].

Sample for Flocculation and Dewatering Tests

In the flocculation and filtration tests, the Pittsburgh No. 8 raw coal was used. The coal sample contained 10.8% ash and 2.65% total sulfur. The coal sample was crushed and then wet-ground down to very fine size (>90% below 25 μm) in a ball mill.

Chemicals

Flocculant

The flocculant used in the tests was a totally hydrophobic polymer provided by Calgon Corp., Pittsburgh, Pennsylvania. Its commercial name is FR-7A. However, its exact chemical structure is industrial proprietary information. The molecular weight of FR-7A is believed to be slightly less

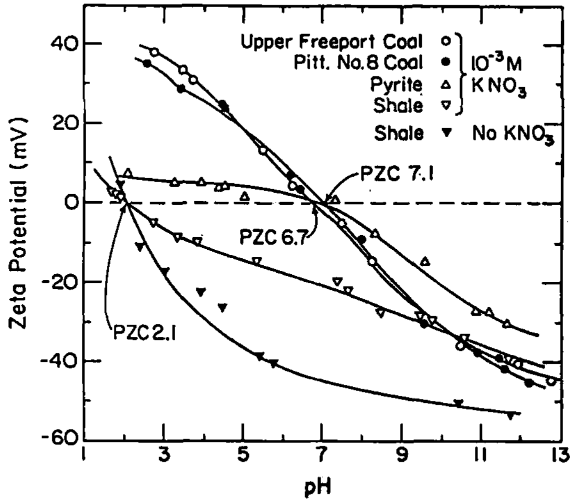

FIG. 1. Zeta potentials of coal, pyrite, and shale.

than one million (5). Since this polymer is specifically emulsified, it could well disperse in water. However, it was observed that FR-7A polymeric colloid clouds tend to slowly migrate to the anode in an electrophoresis cell. It seems that FR-7A is weakly negatively charged.

Dispersant

Sodium metaphosphate (SMP) was used as a general dispersant, particularly in the selective flocculation separation of coal from shale. This chemical was obtained from Fisher Scientific Co.

Procedures

Nephelometry for Determination of Polymer Concentration

A nephelometer, Model 21, manufactured by Monitek Inc., Hayward, California, was used for determining the concentration of FR-7A in solution. Since FR-7A is a naturally turbid emulsion, its turbidity can be measured directly. The calibration curve, established by using known concentrations of polymer in solutions, showed a linear relationship between turbidity and FR-7A concentration; this relationship was not influenced by the pH of the solution (9). By using this calibration curve, the polymer concentration of an unknown solution could be determined from the measured turbidity.

Polymer Adsorption

The adsorption studies included the adsorption kinetics and isotherms of FR-7A on coal, pyrite, and shale, and the effects of pH and SMP on the adsorption of FR-7A. The aqueous suspensions at 2% solids by weight were mixed with the predetermined concentration of FR-7A for 5 min with a magnetic stirrer, and then allowed to stand for another period of time, which was varied from 0.5 through 24 h for the kinetics study, and for 12 h for establishing adsorption isotherms. After the adsorption, the solids were separated from the suspensions by a membrane (0.2 μm) filter under air pressure. Then the concentration of FR-7A in the solid-free solution was determined by the nephelometry from the calibration curve of FR-7A concentration versus turbidity. From the difference in FR-7A concentration between the initial and residual solutions after adsorption of FR-7A, the quantity of adsorbed FR-7A on the minerals could be derived. Finally, the adsorption densities were calculated by dividing the amount of adsorbed FR-7A by the surface area of the sample.

Flocculation

The coal slurry was diluted to about 5 wt% solids content and was adjusted to desired conditions (various pH levels and SMP concentrations). The flocculant, FR-7A, was added to the slurry under mechanical agitation. The agitation was continued for 2 min at high speed and for another 1 min at low speed. The slurry was then allowed to settle for 10 min, and the flocculated fraction was separated from the slurry by decantation. After drying, the weight and ash content of each of the floc and supernatant fractions were measured.

Filtration

A 50-mL flocculated coal slurry with about 5% solids was transferred into a membrane pressure filter apparatus connected to a compressed air cylinder. Ordinary filter papers were used instead of membranes. The air pressure was adjusted to the required value, and filtration was continued for a predetermined time. The filter cake was weighed before and after drying in an oven at 105°C to determine its moisture content.

RESULTS AND DISCUSSION

Adsorption of FR-7A

Adsorption Kinetics

The kinetic study (Fig. 2) shows that equilibrium was attained in about 4 to 7 h, with a steady adsorption rate until the adsorption plateau was

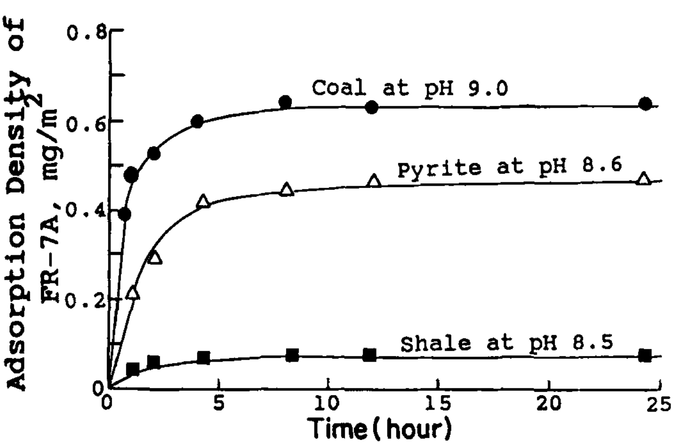

FIG. 2. Adsorption kinetics of FR-7A on coal, pyrite, and shale at pH 8.5–9.0.

reached. The figure also indicates that polymer adsorption in the early adsorption period was faster on coal than on pyrite, and was much faster than on shale. For subsequent experiments, 12 h was taken as the time to achieve adsorption equilibrium.

Adsorption Isotherms

Figure 3 illustrates the adsorption isotherms at various pH levels. The adsorption density increased with increasing equilibrium concentration of the polymer in the early stages and then remained constant, indicating a monolayer coverage (*10*). This type of adsorption follows the Langmuir adsorption model (see Fig. 4 for the Langmuir plots).

The figure also shows that equilibrium adsorption density of FR-7A on coal was about 6 times higher than that on shale; i.e., the FR-7A had a higher adsorption affinity to coal than to shale minerals. The higher adsorption affinity of FR-7A to coal can be attributed to "hydrophobic bond" formation (*11*, *12*) between the hydrophobic polymer molecules and the naturally hydrophobic surface of coal. For shale, adsorption was depressed due both to the natural hydrophilicity and to the relatively high negative zeta-potential of the surface. Referring to the results of adsorption kinetics which show the adsorption rate was much higher for coal than for shale in the early adsorption period, it is believed that the ratio of the polymer adsorption density on coal to that on shale minerals would be much greater if the polymer adsorption and floc formation were conducted for only 5 to 10 min, which is usually the time for a selective flocculation process.

However, the equilibrium adsorption density of FR-7A on pyrite was also high. Pyrite, like coal, also has a hydrophobic surface in nature, and

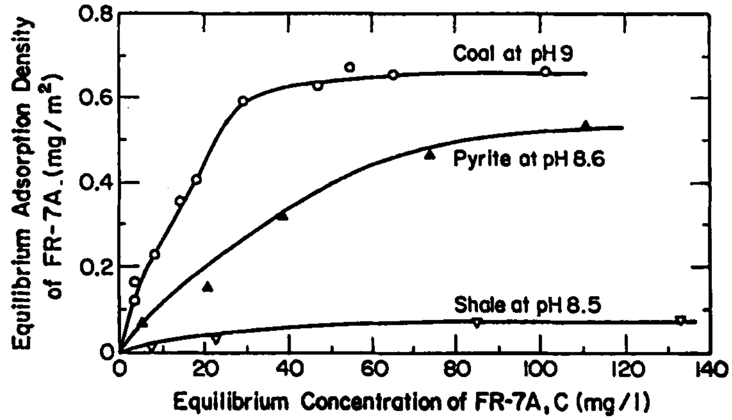

FIG. 3. Adsorption isotherms of FR-7A on coal, pyrite, and shale at pH 8.5–9.0.

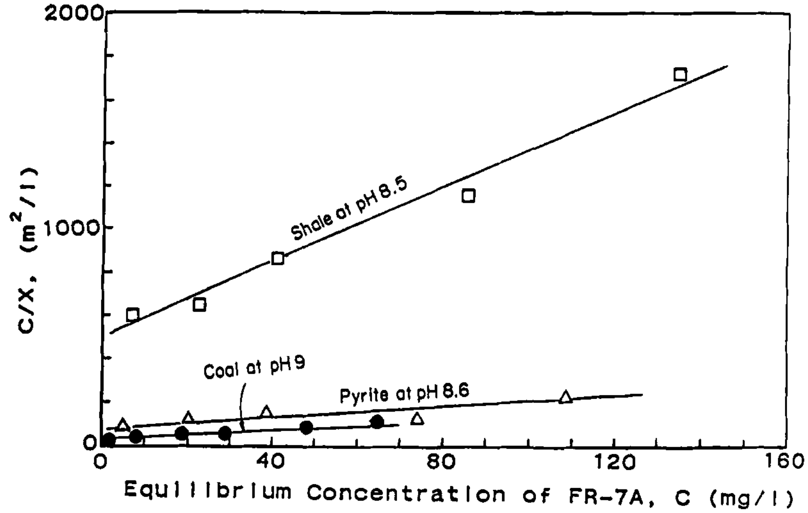

FIG. 4. Langmuir plots for the adsorption of FR-7A on coal, pyrite, and shale at pH 8.5–9.0.

a pzc very close to coal's. These properties imply a difficulty for separating pyrite from coal in a selective flocculation process using FR-7A as the selective flocculant. To remove pyrite, a selective dispersant should be used to modify the pyrite surface and render it hydrophilic to depress its adsorption of FR-7A. Studies with such a polyxanthate reagent have been reported by Attia and coworkers (*13*).

Effect of pH on Adsorption

The effect of pH on the adsorption of FR-7A on the minerals was studied at various initial FR-7A concentrations. Figure 5 illustrates that at increasing pH, the equilibrium adsorption density of FR-7A molecules on all the minerals generally decreased. This is understandable as both FR-7A and the minerals became negatively charged at alkaline pH, which resulted in a depression of adsorption of the weakly negatively charged FR-7A molecules because electrostatic repulsion became operative. Coal and pyrite became positively charged at acidic pH while shale minerals became less negatively charged. As a consequence, polymer adsorption was enhanced due to electrical attraction on all the minerals. However, at neutral pH, adsorption of FR-7A on coal was high mainly due to hydrophobic association, while that on shale was low because the electrical repulsion was operative. This means that selective flocculation using FR-7A will likely

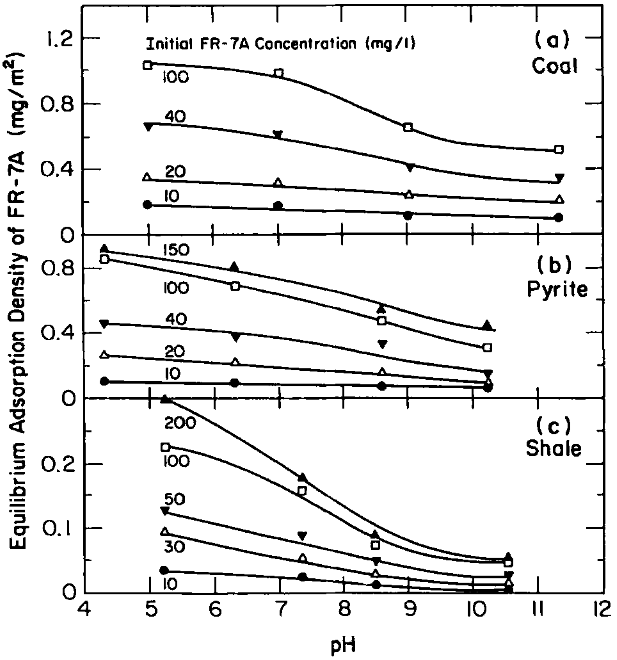

FIG. 5. Effect of pH on the adsorption density of FR-7A on coal, pyrite, and shale.

occur in a neutral pH range. This has been demonstrated by Attia and coworkers (*5*).

Effect of SMP on Polymer Adsorption

SMP dispersant dissolves in water to form an anionic phosphate group and Na^+. The adsorption of the SMP anionic group on mineral surfaces causes the mineral surface to be more electrically negative, as shown in Fig. 6. However, when SMP concentration was above 400 mg/L, no further increase in the zeta potentials of the minerals was observed. This implied that there was no more adsorption of metaphosphate anions on the mineral surface because the active sites were probably fully occupied.

Even though SMP causes the mineral surfaces to be more electrically negative, the adsorption of FR-7A on the minerals was not significantly influenced, as shown in Fig. 7. Evidently the larger polymer molecules were able to displace the smaller metaphosphate anions from the mineral surface when enough time was allowed to achieve equilibrium adsorption. With shorter periods for adsorption, the smaller metaphosphate anion would be kinetically favored due to its faster diffusion rate.

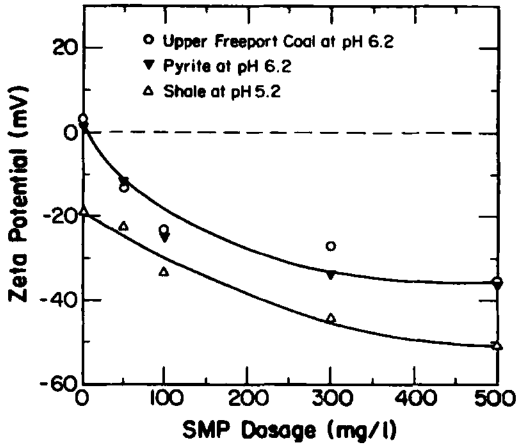

FIG. 6. Effect of SMP dosage on the zeta potential of coal, pyrite, and shale.

Adsorption Thermodynamics

Since the adsorption was found to follow the Langmuir adsorption model, the standard free energy change for adsorption of FR-7A was calculated for each mineral at various pH levels (*14*) by estimating the adsorption equilibrium constant, *K*, from the Langmuir plot by using the

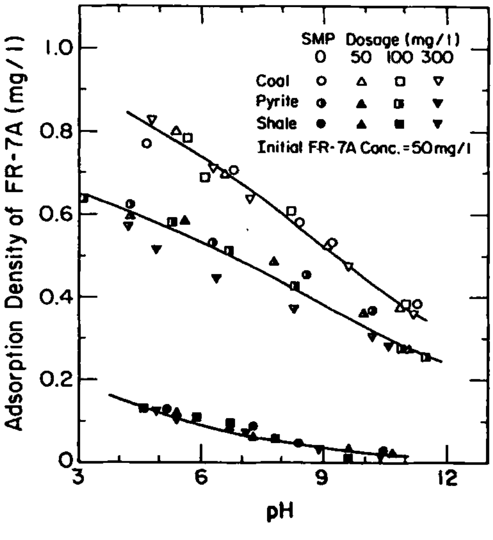

FIG. 7. Effect of SMP on the equilibrium adsorption density of FR-7A on coal, pyrite, and shale at various levels of pH.

equation: $\Delta G^\circ_{\mathrm{ads}} = -RT\ln K$. Following the Stern model (*15*), the standard energy of adsorption, $\Delta G^\circ_{\mathrm{ads}}$, can be split to two parts: $\Delta G^\circ_{\mathrm{el}}$, the standard free energy change due to electrical double-layer adsorption forces, and $\Delta G^\circ_{\mathrm{sp}}$, the standard free energy change due to specific adsorption forces. $\Delta G^\circ_{\mathrm{el}} = zF\zeta$, where ζ is the solid's zeta potential, z is the valency of the adsorbing molecule, and F is the Faraday constant. The value of $\Delta G^\circ_{\mathrm{sp}}$ can be estimated from $\Delta G^\circ_{\mathrm{ads}}$ and $\Delta G^\circ_{\mathrm{el}}$. At pH higher than $\mathrm{pH_{pzc}}$, the zeta potential of the mineral was negative, and $\Delta G^\circ_{\mathrm{el}}$ became positive, which meant that adsorption was depressed by the electrostatic repulsion. When pH was below $\mathrm{pH_{pzc}}$, the zeta potential was positive, $\Delta G^\circ_{\mathrm{el}}$ was negative, and adsorption was favored.

Based on the calculated cross-sectional areas of the FR-7A functional groups and the water molecule from the bond lengths and stereo angles (*8, 16*), calculations showed that one functional group of FR-7A molecule, which was random in solution, could replace 5.16 of water molecules, which were arranged in order on a coal surface. The entropy change due to adsorption of 1 mol monomer FR-7A, $\Delta S^\circ_{\mathrm{ads}}$, was calculated at 60.9 J/mol·K. And the contribution due to the configurational rearrangement to the Gibbs free energy of adsorption at room temperature, 25°C (298 K), was $-T\Delta S^\circ_{\mathrm{ads}} = -18.2$ kJ/mol (*14*). These data indicate that configurational rearrangement made the major contribution to the adsorption driving force, $\Delta G^\circ_{\mathrm{ads}}$ (*11, 12*).

Since $\Delta H^\circ_{\mathrm{ads}} = \Delta G^\circ_{\mathrm{ads}} + T\Delta S^\circ_{\mathrm{ads}}$, the adsorption heat, or enthalpy change due to polymer adsorption, can be derived from the data listed in Table 2. The data indicate that stronger interactions occur at acidic pH conditions mainly due to electrostatic attraction between the polymer molecules and coal. Also, the interaction between the polymer and naturally hydrophobic coal surface is greater than that between the polymer and pyrite, and much greater than that between the polymer and shale.

Flocculation of Coal Slurries Using FR-7A

Effect of pH and SMP on Flocculation

Figure 8 shows the effect of pH on flocculation when using FR-7A. It is indicated that at pH 4.8, almost total flocculation (over 98 wt%) occurred, and the solids content in the supernatant was reduced from 5% to less than 0.2%. This could be attributed to the relatively high and unselective adsorption of FR-7A molecules on the minerals, while at alkaline pH, due to the relatively highly negative surface zeta potential of the minerals, adsorption of FR-7A molecules and flocculation were reduced.

Figure 8 also shows the effect of SMP concentration on flocculation of coal slurries. Since the adsorption of metaphosphate anions rendered the

TABLE 2
Calculation of Thermodynamic Quantities for the Adsorption of FR-7A on Coal, Pyrite, and Shale at Various Levels of pH

pH	X_0 (mg/m^2) Calculated	X_0 (mg/m^2) Measured	K × 10^4	ΔG°_{ads} (kJ/mol)	ζ (mV)	ΔG°_{el} (kJ/mol)	ΔG°_{sp} (kJ/mol)	ΔH°_{ads} (kJ/mol)
			Upper Freeport Coal					
5.0	1.14	1.14	4.49	−26.5	14.4	−1.4	−25.1	−8.3
7.0	1.09	1.00	2.42	−25.0	−3	−0.3	−24.7	−6.8
9.0	0.69	0.68	1.79	−24.3	−26	2.5	−26.8	−6.1
11.3	0.57	0.51	1.19	−23.3	−37	3.6	−26.9	−5.1
			Pittsburgh No. 8 Coal					
6.5	0.93	0.89	2.79	−25.4	5	−0.5	−24.9	−7.2
8.9	0.76	0.60	1.88	−24.4	−22	+2.1	−26.5	−6.2
11.0	0.64	0.35	1.10	−23.1	−38.2	+3.8	−26.9	−4.9
			Pyrite					
4.3	0.69	0.67	2.10	−24.7	6.0	−0.6	−24.1	−6.5
6.3	0.54	0.51	1.60	−24.0	1.5	−0.1	−23.9	−5.8
8.6	0.50	0.33	1.14	−23.1	−8	0.8	−24.1	−4.9
10.2	0.28	0.27	0.85	−22.4	−20	1.9	−24.3	−4.2
			Shale					
5.3	0.36	0.30	0.94	−22.7	−14	1.4	−24.1	−4.5
7.3	0.23	0.20	0.63	−21.7	−22	2.1	−23.8	−3.5
8.4	0.11	0.16	0.38	−20.4	−26	2.5	−22.9	−2.2
10.5	0.09	0.12	0.28	−19.7	−35	3.4	−23.1	−1.5

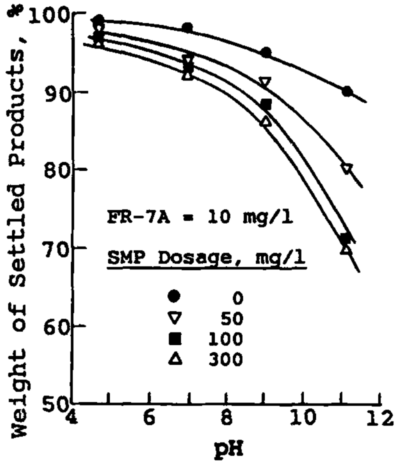

FIG. 8. Effects of pH and SMP dosage on flocculation of coal slurry using FR-7A.

minerals' (especially the shale group) surfaces more negative, and thus more dispersible, increasing the SMP concentration caused a decrease in the weight percent flocculated. As mentioned earlier, SMP is able to compete with FR-7A at the shorter mixing time periods used for flocculation tests. It was noticed that the dispersed products due to SMP consisted mainly of shale minerals. This confirms that the existence of SMP enhances the removal of shale from coal slurry through a selective flocculation process.

The influence of SMP on flocculation was more effective at alkaline condition, and increasing the SMP concentration above about 100 mg/L produced only a slight additional decrease in flocculation.

Filtration of Coal Slurries with the Aid of FR-7A

In this set of tests, the variables investigated were filtration pressure, time, slurry pH, and SMP and FR-7A concentrations.

Effect of Pressure on Filtration Rate and Cake Moisture

The purpose of these tests was to determine the workable pressure and time for filtration tests. Two parameters were used for the evaluation: percent water removal and percent cake moisture. Figure 9 shows the effect of filtration time and pressure on percent water removal. The flocculation

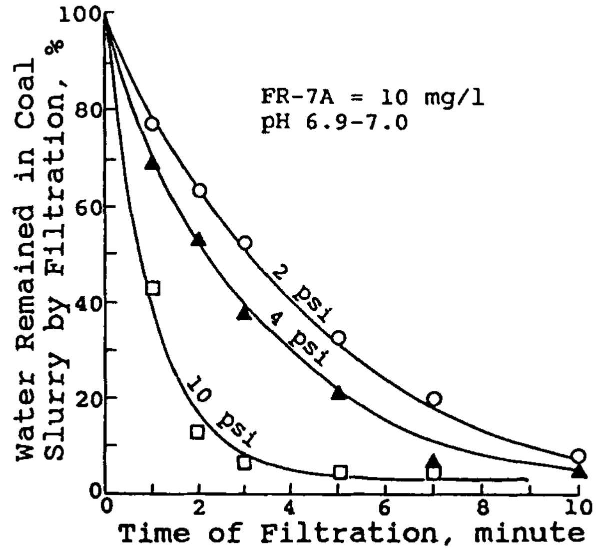

FIG. 9. Dewatering of coal slurries by press filtration (1 psig = 6.9 kPa).

tests were conducted with 10 mg/L of FR-7A at pH 7, using the same procedure described earlier, and the filtration time for these tests was arbitrarily set at 10 min. The graph clearly indicates that the dewatering rate is much faster under higher pressure. However, because of the weakness of filter paper, the tests could not be conducted at pressures higher than 82.8–96.6 kPa (12–14 psig) before the formation of cake.

After formation of a filter cake, the pressure could be adjusted up to 276 kPa (40 psig). In testing the effect of pressure on cake moisture, the conditions were set at pH 6.9–7.0, a dosage of 10 mg/L FR-7A, and 2-min duration of filtration. From Fig. 10 we can see that moisture content in the floc product decreased as the pressure was increased to about 138 kPa (20 psig). At pressures higher than 138 kPa, little additional moisture could be removed.

It seemed that the residual moisture content of the filter cake was still quite high, but it was in reasonable agreement with results in the literature for feed slurries with sizes below −25 μm (*17*). Since the fine particles have a very large surface area, it would be expected that the moisture content would be higher than that for coarser size particles.

From the results presented in Figs. 9 and 10, the effective pressure after formation of cake was selected at about 138 kPa (20 psig), and the filtration time was set at 2 min.

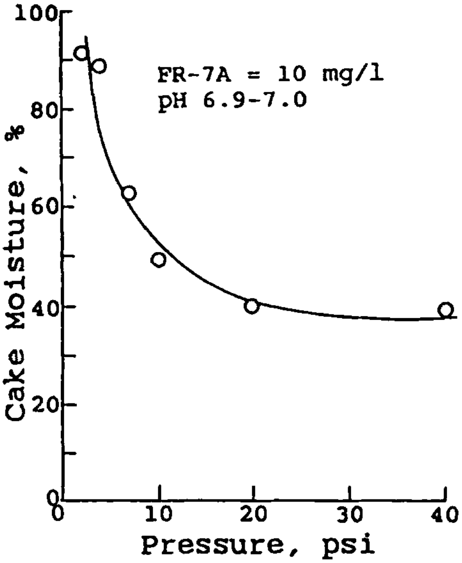

FIG. 10. Effect of pressure on moisture content of the filter cakes (1 psig = 6.9 kPa).

Effect of FR-7A Concentration on Filter Cake Dewatering

Filtration rate greatly depends upon the permeability of the filter cake, which is a function of the particle size distribution (2). In the absence of flocculant, since the particles in the slurry were very fine, the permeability of the filter cake and the filtration rate became very low. With the addition of a flocculant, the filtration rate became significantly faster. Also, the increased surface hydrophobicity of the coal solids, due to adsorption of the hydrophobic polymer, enhanced filtration dewatering. Figure 11 shows the effect of FR-7A polymer dosage on the moisture content of the filter cake. From this figure, as FR-7A concentration was increased, the floc sizes increased, and therefore the cake moisture content was reduced to about 37%. But as the FR-7A concentration was raised higher than 50 mg/L, the moisture content of the cake was no longer affected by the dosage of FR-7A.

Effects of pH and SMP on Filter Cake Dewatering

The tests examining the cake moisture content versus pH in the absence and presence of SMP were set at 10 mg/L FR-7A, 138 kPa (20 psig) filtration pressure, and 2 min filtration time. The results are shown in Fig. 12. The relatively low moisture content in cake at acidic pH could be attributed to the relatively high adsorption of FR-7A, the formation of large flocs, and increased surface hydrophobicity. While the adsorption of FR-7A was reduced at high pH, the flocs formed were very small in size and the particle surface became more hydrophilic and highly charged, which resulted in higher moisture content in the filter cake.

The addition of SMP to the slurry caused the particles to be more dispersible, thus affecting the formation of flocs and cake permeability. How-

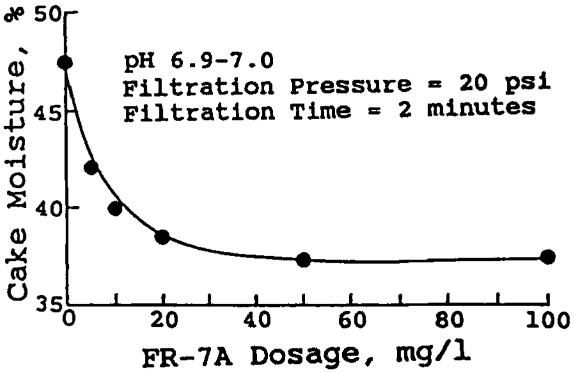

FIG. 11. Effect of FR-7A dosage on moisture of filter cake.

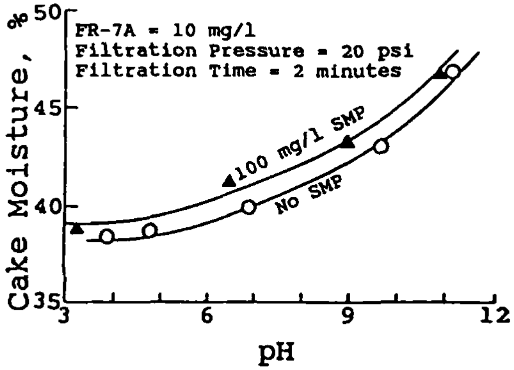

Fig. 12. Effects of pH and SMP on moisture content of the filter cake.

ever, the presence of SMP did not appear to affect the final moisture content significantly.

CONCLUSIONS

From the above results and discussion, the following conclusions can be drawn:

1. The adsorption of the hydrophobic polymer on selected minerals follows the Langmuir adsorption model at relatively dilute concentration of the polymer (below about 200 mg/L).
2. The adsorption isotherms indicate that this polymer has a higher affinity to coal than to shale minerals. However, its affinity to naturally hydrophobic pyrite is also quite high.
3. The adsorption of FR-7A on the minerals was very sensitive to suspension pH. At acidic condition, the equilibrium adsorption densities of FR-7A on the minerals are relatively high, and correspondingly, total flocculation can occur. Filtration is more efficient under these conditions due to increased cake permeability and increased surface hydrophobicity. At alkaline conditions, the adsorption of FR-7A on the minerals is greatly depressed by electrostatic repulsion, and total flocculation and filtration of coal slurry become less effective.
4. The rate of filtration dewatering of coal cake increases with increasing polymer concentration.
5. The presence of SMP in the coal slurry causes the minerals' surfaces to be more electrically negative, but it does not significantly affect the equilibrium adsorption of FR-7A on the minerals. However, at nonequilibrium shorter adsorption periods, SMP can affect the floc-

culation ability of FR-7A polymer, which decreases the efficiency of filtration of the coal slurries.

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