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## Separation Science and Technology

Publication details, including instructions for authors and subscription information:

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**To cite this Article** Cheng, Y. S. , Fang, S. R. , Tierney, J. W. and Chiang, S. H.(1988) 'Application of Enhanced Vacuum Filtration to Dewatering of Fine Coal Refuse', *Separation Science and Technology*, 23: 12, 2113 – 2130

**To link to this Article:** DOI: 10.1080/01496398808075686

URL: <http://dx.doi.org/10.1080/01496398808075686>

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## APPLICATION OF ENHANCED VACUUM FILTRATION TO DEWATERING OF FINE COAL REFUSE

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### ABSTRACT

It is difficult to reduce the moisture content of fine coal refuse to a satisfactory level because of the high mineral content and the large capillary forces associated with small particle sizes. An experimental investigation of important operating variables on dewatering of fine coal refuse is reported. The cake permeability, cake formation time and final moisture content are used to measure the efficiency of moisture removal. Factors that were studied are the addition of coarse particles, level of vacuum, pH and the use of coagulants, flocculants and surfactants as additives. Addition of a flocculant was the most effective single means of improving dewatering and the permeability could be increased by more than an order of magnitude and the moisture content lowered by as much as 0.05 kg water/kg dry cake. It was found that the ionic nature and molecular weight of the flocculant, the flocculant dosage, the mixing time and the mixing intensity must be carefully studied to obtain optimal performance.

### INTRODUCTION

Modern mining and wet coal cleaning operations produce large quantities of fine refuse (-28 mesh, <638  $\mu\text{m}$ ) in the form of a water

slurry. It has been common practice to dispose of this material by pumping behind a permanent impoundment or into settling ponds (1,2). However, because of growing concern over water contamination and sludge pond dam collapse (3), there is renewed interest in dewatering of this material so that it can be disposed of in suitable landfills. Mechanical dewatering using vacuum filtration is widely used in coal preparation plants for dewatering of fine coals, but is less successful with refuse because of the small size and high clay content. In order to obtain structurally stable filter cakes economically, enhanced dewatering is necessary.

Two types of enhanced dewatering techniques are practiced--slurry pre-treatment and cake post-treatment (4,5). In the former, the filtration characteristics of the slurry are modified by adding materials such as inorganic coagulants or polymeric flocculants. In the latter, the filter cake is treated to lower the capillary pressure and permit further reduction in moisture. Both methods have been successfully used for dewatering of fine coals (6,7), but little has been reported on enhanced mechanical dewatering of fine refuse. In this paper, a comprehensive study of dewatering of a fine refuse is reported. The factors investigated include pH control, vacuum level, particle size distribution and the use of coagulants, flocculants and surfactants. The objective is to identify the most promising types of enhancements and to quantitatively determine their efficacy. The results, while specific to the refuse studied, are indicative of the improvements which can be obtained by enhanced dewatering and can serve as a guide when studying a specific refuse.

## EXPERIMENTAL

The experimental measurements were made in a specially designed vacuum filtration cell shown schematically in Figure 1. Filter cakes are formed in a Plexiglas cylinder with a 0.05 m i.d. equipped with a flexible rubber sleeve inside the cylinder to ensure that there is no seepage of filtrate from the periphery of the cake (8). The filtrate collected is continuously weighed and recorded using a load cell. The resultant record of filtrate weight as a function of time is used to determine the filtration and dewatering rate and the permeability of the cake being studied.

The refuse used in this study is typical of that obtained when cleaning a Pittsburgh seam coal. Two samples were collected at different times from an operating coal preparation plant in southwestern Pennsylvania. Each was separated by wet screening into three size ranges. The physical and chemical properties of the individual and composite samples were measured and are shown in Table 1 as Batch 1 and Batch 2. It can be seen that there is a larger fraction of clay and mineral material in the small size ranges, and there will, therefore, be particles of micron size in the -200 mesh portion. There are only minor differences in the properties of the two batches. A particle size analysis was made for Batch 1 using a computer aided Leitz TAS image analyzer and is shown in Figure 2. Batch 1 was used for determining the effect of vacuum, particle size and pH, while Batch 2 was used for all other measurements.

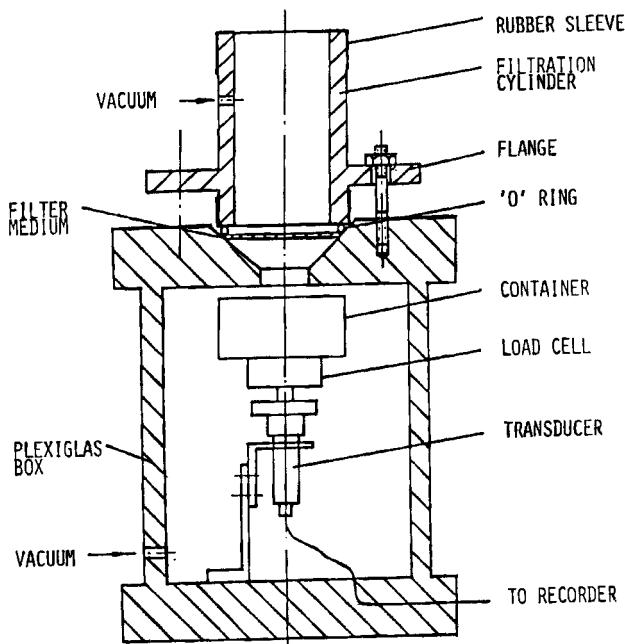
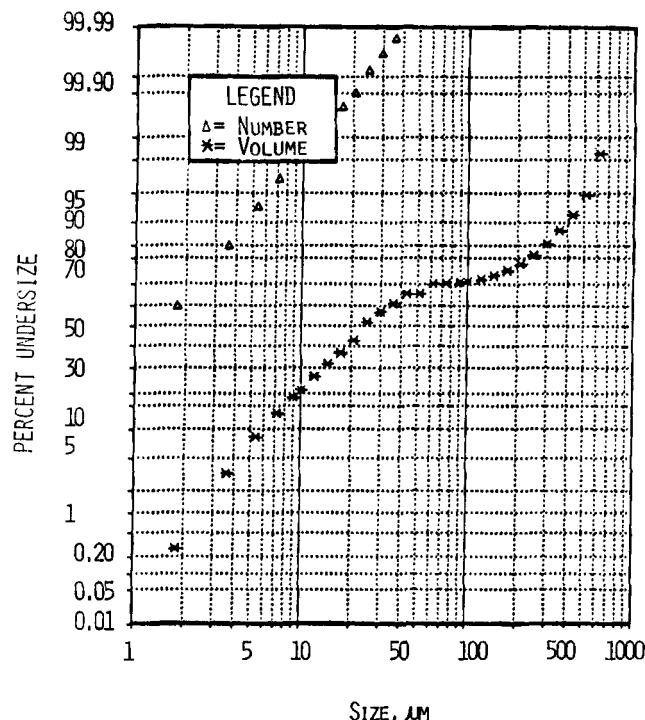


Figure 1. Schematic diagram of filtration cell.

Table 1. Characteristics of Coal Refuse Samples Used  
Weight Percent and Percent Ash Are on a Dry Basis

Size (mesh)	Batch	Wt. %	Ash %	Bulk Density
+28 (> 638 $\mu\text{m}$ )	1	3.71	6.48	1.310
	2	2.94	5.49	1.338
-28+200 (> 74 $\mu\text{m}$ , < 638 $\mu\text{m}$ )	1	28.11	10.40	1.363
	2	30.31	8.16	1.354
-200 (< 74 $\mu\text{m}$ )	1	68.18	47.83	1.846
	2	66.75	42.16	1.789
Composite	1	100.00	35.96	1.640
	2	100.00	31.10	1.647



**Figure 2. Particle size distribution of Batch 1 coal refuse sample.**

The properties of the additives used are listed in Table 2. The aluminum sulfate was obtained from Fisher Scientific, the polymeric flocculants from American Cyanamid, and the surfactants from Rohm and Haas. All additives, whether originally solid or liquid, were dissolved in water to make water solution with concentrations of 1000 ppm.

The experimental procedure was as follows:

1. Water was added to the lumpy refuse sample and gently stirred to produce a uniform paste. Representative samples of this paste were used in the experiments.
2. A sample of the paste containing 25 gm of dry refuse was diluted with distilled water and surfactant solution if used. The amount of water added was such that the final concentration of solid in the slurry was 25 wt. % for all runs including those in which flocculant was used. The prepared slurry was then stirred for 30 minutes at a stirrer speed of 380 rpm.

**Table 2. List of Chemical Additives Used.**

Name	Type	Mol. Wt.
Aluminum Sulfate	Inorganic Coagulant	342.1
Accoal-Floc 550	Flocculant, Anionic	250,000
Accoal-Floc 204	Flocculant, Anionic	$4-6 \times 10^6$
Accoal-Floc 218	Flocculant, Anionic	$18-20 \times 10^6$
Accoal-Floc 16	Flocculant, Nonionic	$4-6 \times 10^6$
Accoal-Floc 330	Flocculant, Cationic	50,000
Accoal-Floc 355	Flocculant, Cationic	250,000
Magnifloc 494C	Flocculant, Cationic	$2-4 \times 10^6$
Triton-X114	Surfactant, Nonionic	536
Aerosol-OT	Surfactant, Anionic	444.5

3. If used, a flocculant was added into the slurry and mixed at the desired stirrer speed for a predetermined period of time.
4. The slurry was poured into the filtration cell and a constant vacuum (usually 50 cm Hg) was applied and remained as a constant in both filtration and dewatering period. Whatman #42 paper was used as the filter medium. The weight of filtrate was recorded as a function of time.
5. The time at which the last drop of liquid disappeared from the surface of the cake was recorded as the cake formation time, and marked the beginning of dewatering. When 5 minutes passed with no filtrate collected, the experiment was terminated.
6. The filter cake was weighed, dried at 50°C for at least 48 hours and weighed again to determine the final moisture content.
7. Mass balances for the solid and liquid were made, and the permeability of the cake calculated by using the Ruth equation and assuming constant medium resistance (4,7).

## RESULTS AND DISCUSSION

Two characteristics of the filtration and dewatering are of most importance--the ease of filtration and the moisture content attainable. The former determines the throughput rate in a drum filter and is measured by the permeability of the cake or the cake formation time. The latter determines the suitability of the cake for disposal and is measured by the final moisture content. In the discussion which follows, these properties are used to evaluate the effectiveness of dewatering enhancements.

Mass balance for each run showed maximum relative errors of 3% and 4% for liquid and solid, respectively. Duplicate runs yielded a maximum

**TABLE 3. Effect of Applied Vacuum on Filtration Characteristics of Filter Cakes**

Vacuum (cm Hg)	Porosity (%)	Permeability (mD)	Final Moisture (kg/kg dry)
30	43.2	2.4	0.396
40	42.7	2.1	0.385
50	42.7	2.0	0.366
60	43.4	1.8	0.365

deviation of 10% in the measured permeability and 5% in the measured final moisture content.

#### Effect of Applied Vacuum

The permeability and porosity of filter cakes were measured for vacuum between 30 cm Hg and 60 cm Hg. Results are summarized in Table 3. The permeability decreases slightly with increasing pressure drop while porosity remains constant. The cake is essentially incompressible for this range of pressure drop. The slight decrease in permeability may be due to migration of small particles during the filtration. The final moisture content decreases with increased vacuum up to 50 cm Hg, but decreases only slightly with higher vacuum. A similar result has been reported for fine coal dewatering (9).

#### Effect of Particle Size

It has been suggested in the literature (10) that adding coarse particles to fine refuse can improve filterability of the slurry. Since segregation became serious as large particles are added, the Ruth equation is no longer valid. The cake formation time was used to measure the filterabilities of the samples. The cake formation time, defined as the time when the last drop of fluid disappears from the surface of the cake, is different from that defined by Purchas (11). Several samples of differening particle size distribution were prepared by mixing portions of the -200 mesh refuse with either the +28 mesh or -28+200 mesh refuse fractions shown in Table 1. In each case the total weight of solids was maintained at 25 gm so the amount of -200 mesh decreases as the percent of coarse material increases. Some decrease in both cake formation time and final moisture content is to be expected. Results are shown in Figure 3. There is a decrease in cake formation time with addition of coarse particles, and a distinct minimum can be seen. The decrease in final moisture content is not as sharp. It was noted that the cake settling time (the time for the supernatant liquid over the filter cake to become clear) was noticeably less when larger particles were present. The settling characteristics of the slurry are affected by the addition of coarse particles, and there is an improvement in cake formation time. However,

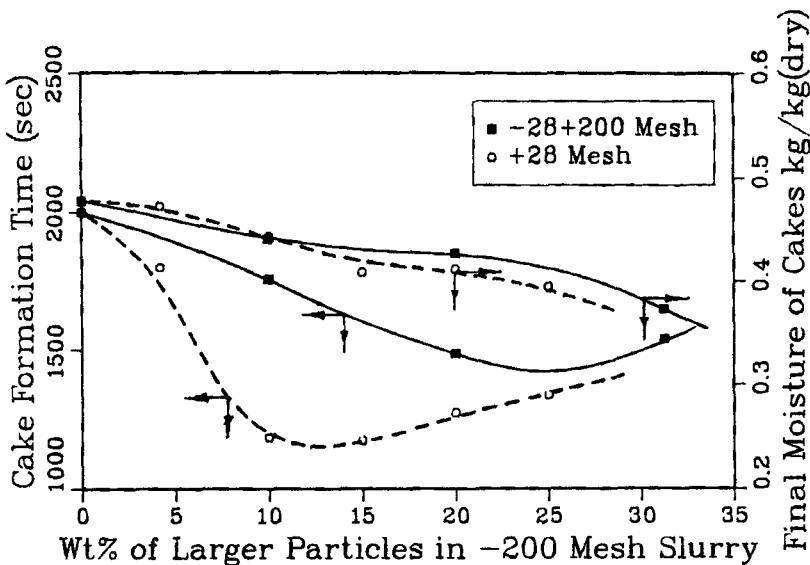


Figure 3. Effect of addition of coarse refuse to -200 mesh refuse.

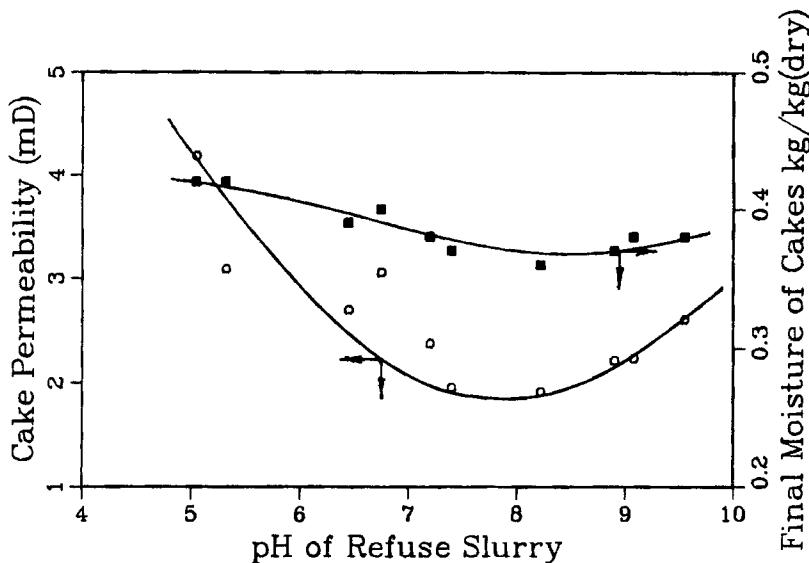
this improvement is not enough to make vacuum filtration of these fine refuse streams practical.

#### Effect of pH

The slurry of refuse material (as received) showed a neutral pH value of 7.4. In order to investigate the effect of pH on filterability, the slurry was treated with an acid or a base prior to filtration. When a strong acid (sulfuric) was added, there was evolution of  $H_2S$  and some flocculation of fine particles. The acid was obviously reacting with pyrites and other sulfur containing compounds. Large amounts of acid were required to lower the pH. Addition of small amounts of a strong base ( $NaOH$ ) rapidly raised the pH. Figure 4 shows the effect of changing pH on permeability and moisture content. There is a slight improvement of permeability with changes in pH, but the improvement is marginal.

#### Effect of Inorganic Coagulants

Coagulation is the adhesion of particles because of inter-molecular and atomic forces between the particles (12). The presence or absence of coagulation depends on the balance between attractive (Van der Waals) forces and dispersive forces resulting from the electrical double layer of charges at the particle surface. In aqueous solutions containing finely divided clay, a double layer is formed with a negative zeta potential



**Figure 4. Effect of pH on permeability and final moisture content of filter cakes.**

(13,14). An inorganic electrolyte can serve as a coagulant by providing cations which reduce the zeta potential. An effective coagulant should provide large cations with high valence according to the Schultz-Hardy rule (15). Even though other coagulants such as aluminum chloride should be more effective, we used aluminum sulfate for testing because it is a commonly used and less costly coagulant. Before filtration, an aluminum sulfate solution was added to the slurry and mixed at a stirrer speed of 380 rpm for 30 minutes. The results are plotted in Figure 5 and show a desirable increase in permeability and an undesirable increase in moisture content. While the slurry is more rapidly dewatered, the final product contains more water. Furthermore, the amount of coagulant required to effect a significant change is too large to be practically useful.

#### Effect of Polymeric Flocculants

It has been reported that polymeric flocculants may be more effective when coarse particles are present (13). These flocculants can be classified by ionic nature into three groups—anionic, cationic, and nonionic—and come in a wide range of molecular weights and charge densities in each category (10). The effectiveness of a flocculant depends on the interaction between the polymer and the particles, which in turn is governed by the charge characteristics, the concentration, and the

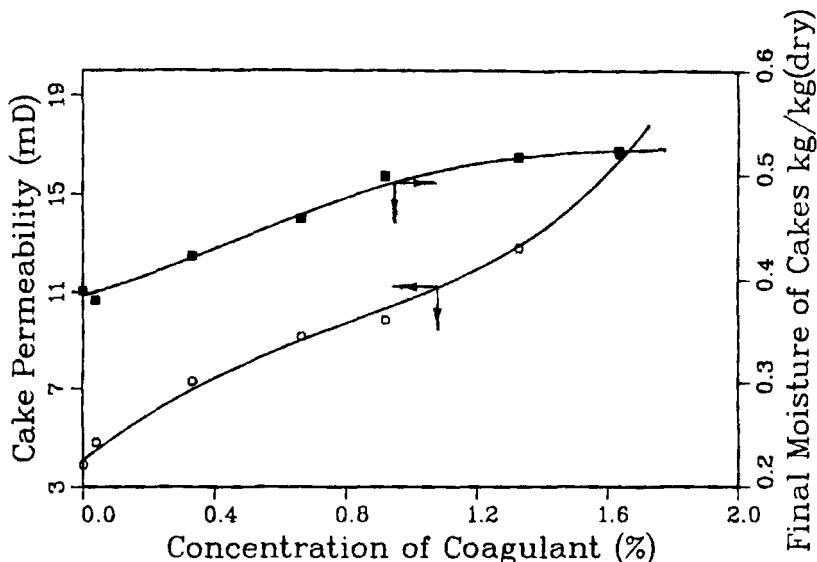


Figure 5. Effect of inorganic coagulant (aluminum sulfate) on permeability and final moisture content of filter cakes.

molecular weight of the polymer. Although it is widely accepted that flocculants are effective for filtration and dewatering of fine coal and refuse, there have been few reports in the open literature (16,17,18). Furthermore, most of the data available in the literature are based on settling tests which are used for design of clarifiers. These results are not necessarily appropriate for filtration because, for example, while large flocs settle rapidly, small tight flocs may be better for filtration. More meaningful measures for filtration are the permeability and moisture content as reported here. The ionic nature of the flocculant, its molecular weight and the method used to add the flocculant to the slurry are important. These factors were investigated.

Ionic nature of flocculant. An anionic, a nonionic and a cationic flocculant were tested. These flocculants have similar molecular weights (from 2 to 6 million), and the same preparation procedure was used--mixing for 60 seconds at 380 rpm before filtration. The variation of final moisture content with concentration is shown in Figure 6. None of the three showed any significant improvement in moisture content at any concentration. At low concentrations there is a small decrease, and at higher concentrations the moisture content increases significantly for the anionic flocculant. As shown in Figure 7, the nonionic and anionic flocculants can increase the permeability by more than an order of

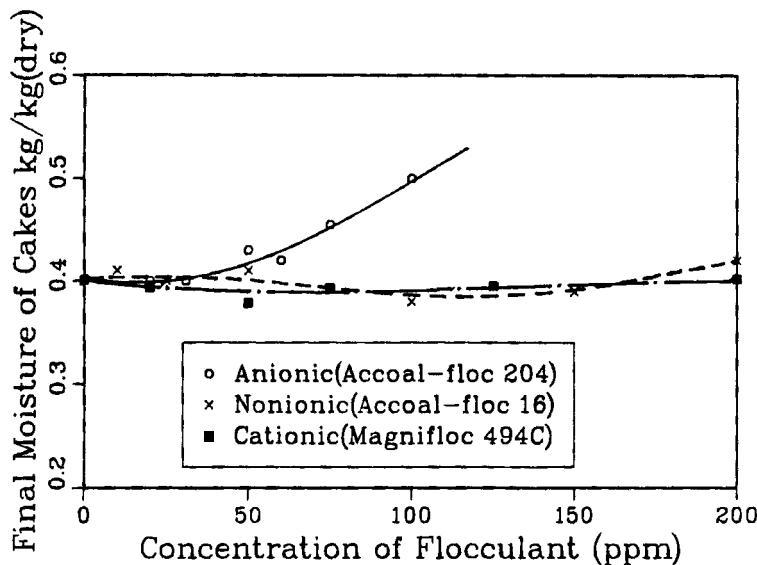


Figure 6. Effect of ionic nature of flocculant on final moisture content of filter cakes.

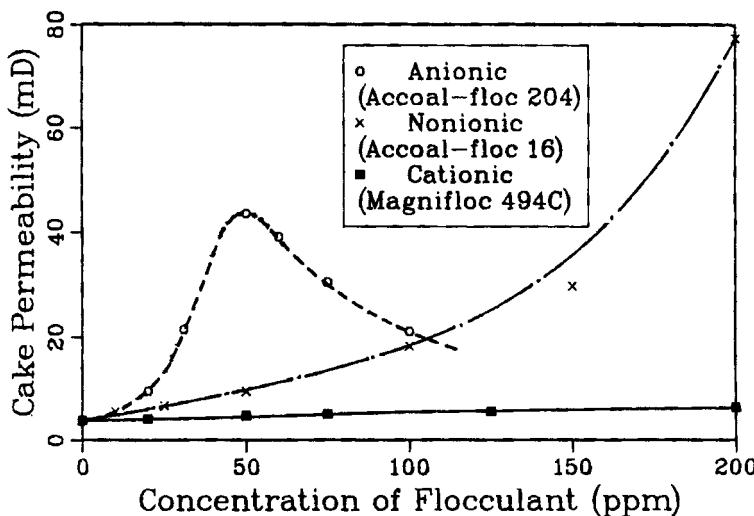


Figure 7. Effect of ionic nature of flocculants on permeability of filter cakes.

**Table 4. Calcium and Magnesium Concentrations in Filtrates  
Obtained from Treated and Untreated Filter Cakes**

Sample	Concentration (ppm)	
	<u>Ca</u> <sup>2+</sup>	<u>Mg</u> <sup>2+</sup>
Blank Filtrate	351	27.9
Accoal-Floc 204, 50 ppm, Anionic	142	14.6
Accoal-Floc 16, 50 ppm, Nonionic	205	19.5
Magnifloc 494C 50 ppm, Cationic	275	22.5

magnitude, while the cationic flocculant has little effect. The increase in permeability is due to a reduction in number of small particles by the formation of large flocs. The failure to reduce moisture content indicates that the flocs have a high water content. At low concentrations the anionic flocculant is most effective. This is surprising because the refuse particles have a negative charge at neutral pH, and it seems that a cationic or nonionic type would be preferred. We believe the explanation for this is the presence of relatively high concentrations of calcium and magnesium ions in the recycle water used in coal preparation plants. The calcium and magnesium ion concentrations are shown in Table 4 for the filtrate with and without additives. The addition of anionic flocculant significantly lowered these concentrations. Lewellyn and Wang (19) postulated that the improved performance of anionic flocculants with clays was due to polyvalent cation bridging. Our results are consistent with this.

Molecular weight of flocculant. The molecular weight of a flocculant determines the shape of the dissolved molecules in aqueous solution and thus the flocculation characteristics (16). Three anionic and three cationic flocculants with varying molecular weights were used. Results are shown in Figures 8 through 11. Permeability increases by as much as a factor of 11 were found at low concentrations for the anionic flocculant (Figure 8). The increase for a molecular weight of 4-6 million is higher than for either higher or lower molecular weights. The final moisture content for each of these is shown in Figure 9 and is about the same for each flocculant for dosages less than 60 ppm. The finding that there is an optimum molecular weight is believed to be due to changes in shape of the dissolved polymers. In dilute aqueous solutions the shape can change from extended long chains to strongly swollen and inflated balls as the

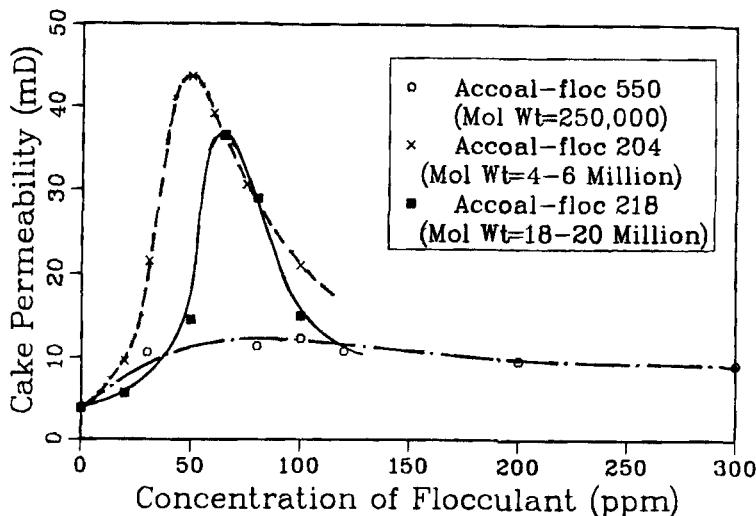


Figure 8. Effect of molecular weight of anionic flocculants on permeability of filter cakes.

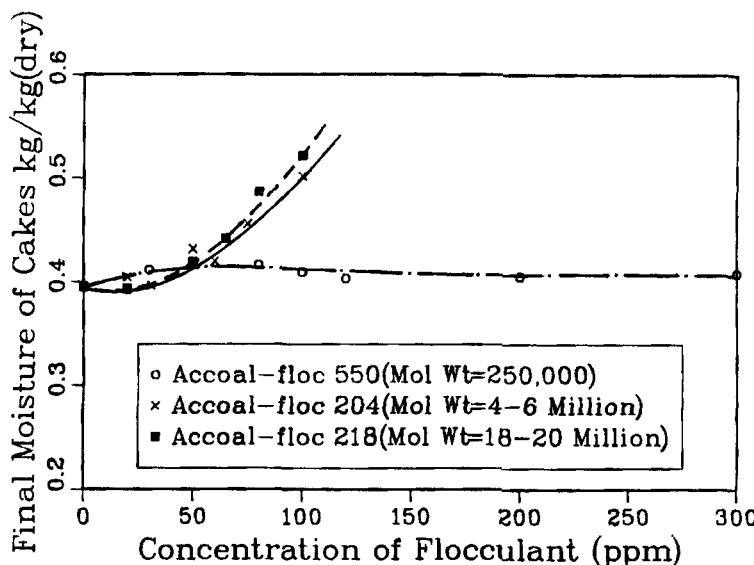


Figure 9. Effect of molecular weight of anionic flocculants on final moisture content of filter cakes.

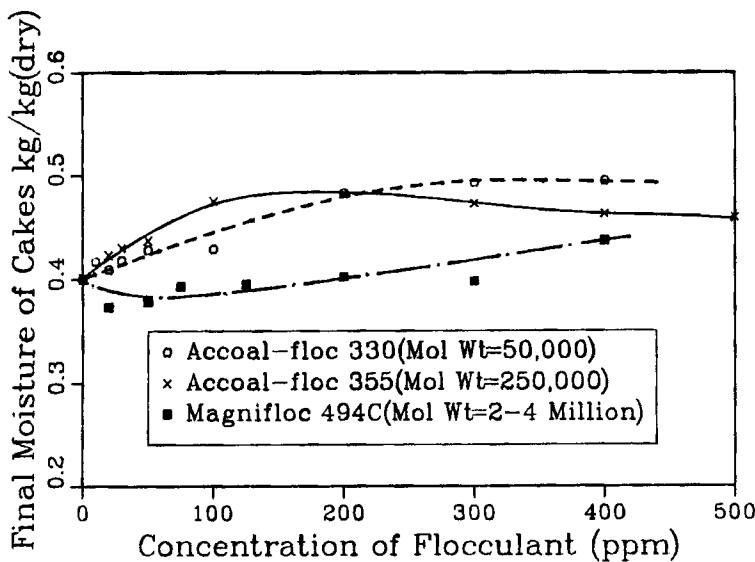


Figure 10. Effect of molecular weight of cationic flocculants on final moisture content of filter cakes.

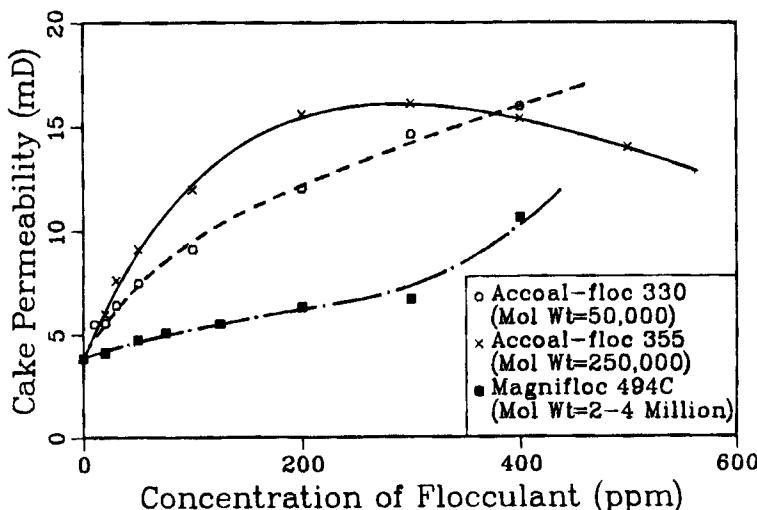


Figure 11. Effect of molecular weight of cationic flocculants on permeability of filter cakes.

**Table 5. Effect of Mixing Time and Mixing Intensity on Filtration Characteristics of Filter Cakes**

Mixing Time (sec)	Stirring Time (rpm)	Permeability (mD)	Final Moisture (kg/kg dry)
60	100	37.8	0.424
60	180	37.0	0.422
60	380	44.0	0.391
60	550	37.3	0.367
60	730	29.5	0.382
15	380	40.6	0.432
45	380	43.0	0.401
60	380	44.0	0.391
90	380	43.1	0.368
180	380	39.3	0.357
300	380	30.3	0.383

molecular weight increases (16). The swollen and inflated molecules knit suspended particles to form flocs. With higher molecular weights the flocs are looser, and conditions are less favorable for filtration and dewatering. The optimum molecular weight should be a function of the particle sizes and types.

The final moisture contents for three cationic flocculants are shown in Figure 10. For reduction of moisture content, the highest molecular weight (2 - 4 million) flocculant is the most effective. The reduction is not as great as that for anionic flocculant, however. The permeabilities are shown in Figure 11. The most effective cationic flocculant is much less effective than the most effective anionic flocculant in increasing the permeability.

Mixing of flocculant with slurry. For a given flocculant and particulate sample the intensity of mixing and the time of mixing determine the size and shape of flocs and thus the filtration characteristics (20). The stirring speed was varied from 100 to 720 rpm for a constant mixing time of 60 seconds at constant flocculant dosage of 50 ppm of Accoal-floc 204. Moisture contents and permeabilities are shown in Table 5. The maximum permeability occurs at 380 rpm and the lowest moisture content at 550 rpm. Then, using the same flocculant and dosage and a mixing speed of 380 rpm, the mixing time was varied from 15 to 300 seconds with the results shown in Table 5. The permeability is a maximum at 60 seconds and the lowest moisture content is at 180 seconds. The best filtration rate is found at a relatively short mixing time and slow mixing speed, while lower moisture contents are found for longer times and higher speeds. It is clear that mixing intensity and time

**Table 6. Effect of Two Types of Surfactant Treatment (Premix or Wash) on Final Moisture Content of Filter Cakes**

		Premixing	
Aerosol-OT		Triton-X114	
Cone. ppm	Final Moisture kg/kg dry	Cone. ppm	Final Moisture kg/kg dry
0	0.404	0	0.404
25	0.385	50	0.368
50	0.373	100	0.369
100	0.370	150	0.368
150	0.365	200	0.372

#### Washing

Flocculant: Accoal-Floc 204, 50 ppm

Aerosol-OT, 100 ppm		Triton-X114, 150 ppm	
Vol. cc	Final Moisture kg/kg dry	Vol. cc	Final Moisture kg/kg dry
0	0.404	0	0.404
30	0.360	30	0.390
50	0.350	50	0.370
75	0.360	75	0.340
		100	0.380

are important in determining the filtration characteristics, and that tradeoffs between high filterability and low cake moisture content must be made.

#### Effect of Surfactant

Venkatadri et al. (7) found sharp reductions in cake moisture content when surfactants were used in conjunction with vacuum filtration of fine coals. The role of the surfactant is primarily to lower the liquid-air interfacial surface tension and reduce the capillary forces holding water in the fine pores. To determine whether they could be used effectively in dewatering of refuse, an anionic (Aerosol-OT) and a nonionic (Triton-X114) surfactant were tested. The surfactants were applied in two ways--by premixing with the slurry before filtration without flocculant and as a post filtration wash in conjunction with an anionic flocculant. The results

are given in Table 6, and indicate a reduction in cake moisture as a function of surfactant concentrations. The combined surfactant-flocculant effect on moisture reduction is comparable to the results obtained when flocculant is applied under optimal mixing conditions (Table 5).

### CONCLUSIONS

Special treatment is necessary if fine coal refuse such as that studied here (with two thirds of the particles less than 200 mesh) is to be dewatered in a vacuum filter. Proper selection of filtration conditions can result in a reduction in the moisture content and significantly improved filterability. The effect of treatment methods on moisture content and filterability of the sample tested are summarized below. They are indicative of the benefits to be expected and can be used as a guide for dewatering of similar materials.

1. The most effective treatment was the use of a polymeric flocculants. For the samples tested the best results were obtained for an anionic flocculant with a molecular weight of 4-6 million and a dosage level of 50 ppm. In the laboratory experiments, the optimum mixing time was 60 seconds with a stirrer speed of 380 rpm.
2. Adding coarse particles improved filterability as measured by reduced cake formation time and reduced moisture content. Optimum addition exists for a certain size range of large particles. In the present case, the addition of 10 wt % of +28 mesh particles seems to be best.
3. Increased pressure drop had little effect on permeability and gave a slight decrease in final moisture content.
4. Changing pH by addition of acid or base had a small effect and is not recommended.
5. Inorganic coagulants improved filterability but also increased final moisture content and are not recommended.
6. Addition of surfactants either as premixes or as a wash liquor were relatively ineffective and are not recommended.

### ACKNOWLEDGMENT

Financial support for this work by the United States Department of Energy under Contract No. DE-AC22-85PC81582 is gratefully acknowledged. Analytical assistance was provided by Dr. S. O'Donnell, Department of Chemistry, University of Pittsburgh.

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