

Dewatering of Mineral Waste Using the Flocculant Polyethylene Oxide

By B. J. Scheiner, Annie G. Smolley, and D. A. Stanley



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CONTENTS

	<i>Page</i>		<i>Page</i>
Abstract	1	Summary	11
Introduction	1	Chapter 3.—Dewatering of miscellaneous clay waste slurries	12
Chapter 1.—Dewatering of phosphate-clay waste ..	2	Ultrafine bentonitic waste	12
Laboratory and small-scale experiments	2	Potash-clay waste	12
Field testing	4	Talc slurry	13
Description of field test unit	4	Mica-clay tailings	14
Results and discussion of data	4	Uranium mill tailings	14
Determination of PEO concentration in solution	7	Summary	14
Summary	7	Chapter 4.—The mechanism of PEO-clay interaction	15
Chapter 2.—Dewatering of coal-clay waste slurries from preparation plants	8	Rheological studies	15
Small-scale dewatering	8	Effect of clay exchange ion on dewatering	15
Field testing	9	Summary	17
		References	18

ILLUSTRATIONS

1. Flocculation of phosphate-clay waste during initial stages of laboratory dewatering sequence	3
2. Formation of strong flocs	3
3. Decanting released water prior to hand squeezing	3
4. Dewatered solids after hand squeezing	3
5. Small-scale static and trommel screen dewatering of phosphate-clay waste	4
6. Field test unit (FTU) used for phosphate-clay waste dewatering	5
7. FTU flow sequence	5
8. Consolidated flocs moving down static screen	6
9. Dewatered material being discharged from trommel screen	6
10. Flow sheet for dewatering coal-clay waste	8
11. FTU for dewatering coal-clay waste	9
12. Dewatered coal-clay waste material mixed with coarse refuse from preparation plant	10
13. FTU with trommel screen at Paradise complex	11
14. Trommel screen dewatering bentonitic clay waste	12
15. Discharge of debrined potash-clay waste solids from belt-roller device	13
16. Linear relationship between relative number of plates per montmorillonite tactoid and hydrated-radius-to-charge ratio (R_H/C) for some common exchange ions	15
17. Variation of floc volume with hydrated-radius-to-charge ratio	16
18. Water structure of montmorillonite clay	17
19. Proposed mechanism for attachment of PEO to montmorillonite	17

TABLES

1. Dewatering phosphate-clay waste using hydrosieve and trommel screen combination	4
2. Summary of results from field dewatering tests using phosphate-clay wastes	7
3. Summary of results of small-scale dewatering of various coal-clay waste slurries	9
4. Screen analysis of coal-clay wastes	10
5. Results of laboratory dewatering tests on coal-clay wastes	10
6. Effect of PEO dosage on dewatering efficiency of 0.005-pct-PEO solutions for bentonitic waste	12
7. Debrining of potash-clay waste slurry	13
8. Small-scale continuous tests on potash-clay waste slurry	13
9. Laboratory tests using PEO to dewater talc slurries	13
10. Dewatering of mica-clay waste slurry	14
11. Effect of PEO concentration on PEO dosage for uranium tailings	14
12. PEO required to dewater ion-exchanged clays of montmorillonite	16

CONTENTS

UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

Å	angstrom	µm	micrometer
ft	foot	NTU	nephelometric turbidity unit
gal	gallon	pct	percent
gal/min	gallon per minute	ppm	part per million
h	hour	r/min	revolution per minute
in	inch	st	short ton
lb	pound	wt pct	weight percent
lb/st	pound per short ton	yr	year
min	minute		

DEWATERING OF MINERAL WASTE USING THE FLOCCULANT POLYETHYLENE OXIDE

By B. J. Scheiner,¹ Annie G. Smelley,² and D.A. Stanley²

ABSTRACT

This bulletin summarizes the results of Bureau of Mines investigations of a technique for dewatering clay waste that consists of flocculation with polyethylene oxide (PEO) followed by screening of the resulting flocs. Results of laboratory tests and small- and large-scale field tests are described. The PEO dewatering technique has been applied to clay wastes from phosphate, coal, bentonite, and potash operations and to tailings from uranium, talc, and mica mining operations. Fundamental studies to determine the mechanism of PEO interaction with clay particles are also described.

INTRODUCTION

In the hydrometallurgical treatment of many ores and concentrates to recover mineral values, slurries of fine particles are generated. Some of these slurries settle slowly and are difficult to dewater. These properties, which are inherent in slurries of certain fine waste materials, cause environmental and handling problems for the mining industry. For example, producers of phosphate, coal, potash, talc, mica, and bentonite are faced with these problems. These producers and others in the mining industry are presently using techniques such as impoundment, thickening, and thickening followed by filtration to dewater and dispose of waste slurries. However, in the future, new technology will be needed to handle slurries containing fine waste particles; this has become clear in light of new environmental regulations, losses of mineral values in the waste slurries, the need to recover more water for reuse in the processing facility, the lack of land suitable for impoundment, and other factors.

The Bureau of Mines has conducted considerable research to better understand the characteristics of slurries containing fine particles and to investigate methods of dewatering these slurries. During this investigation, a unique polymer, polyethylene oxide (PEO), was observed to form strong flocs in many different waste slurries containing clays consisting of layer silicates such as mont-

morillonite, kaolinite, attapulgite, and chlorite. The flocculation of these materials with PEO results in flocs that immediately release water to form a mass that can be mechanically dewatered on static and/or rotary screens (1).³ The resultant dewatered material for many of the slurries investigated has a higher solids content than that which is now being obtained where flocculation using other methods is routinely practiced.

The PEO dewatering technique has been applied to waste slurry streams from the phosphate, coal, potash, talc, mica, and bentonite industries. Research has included both laboratory and field testing. Cooperative field tests by the Bureau and the minerals industry have been conducted at four phosphate mines in Florida and two coal mines in Alabama and Kentucky.

Inasmuch as the Bureau has completed its field testing of the PEO dewatering technique, this bulletin is intended to consolidate the data reported in previous publications and present data on recent research not reported elsewhere. Chapter 1 describes the research pertaining to phosphatic clay waste dewatering. Chapter 2 covers dewatering of coal-clay waste. Chapter 3 describes research on potash-, talc-, and bentonite-clay wastes and other slurries. Chapter 4 presents basic data obtained in laboratory experiments to determine how PEO interacts with clay particles, and a mechanism for this interaction is postulated.

¹Supervisory metallurgist.

²Supervisory research chemist.

Tuscaloosa Research Center, Bureau of Mines, Tuscaloosa, AL.

³Italic numbers in parentheses refer to items in the list of references at the end of this report.

CHAPTER 1.—DEWATERING OF PHOSPHATE-CLAY WASTE

In Florida, the process of mining and beneficiating phosphate rock to produce concentrates for subsequent conversion to agricultural fertilizer generates two waste products. One, a plus 150-mesh flotation tailings sand, is easily disposed of. However, the other is a slurry containing minus 150-mesh phosphatic clay waste, with approximately 75 pct of the material consisting of less than 10- μ m particles (1). Disposal of this colloidal waste is more difficult, especially in light of new State disposal regulations restricting the method currently used, which is impoundment behind earthen dams (2-3).

The overall natural settling rate of phosphatic clay waste is extremely slow. Generally, the clay waste slurries thicken from a nominal 2 to 6 pct solids initially to 10 pct solids in 1 week and to 15 pct solids in about 3 months. Densification to about 18 pct solids requires about 1 year, and to reach a solids contents of 20 pct or higher requires several years.

With respect to water recovery, studies have shown that approximately 90 pct of the water pumped to the settling pond is recovered; the remaining 10 pct is associated with the clay waste in the pond. Estimates indicate that for each short ton of phosphate concentrate produced, as much as 5 st of water is stored with the waste (4).

Typically, for a single mining operation, the rate of flow of phosphatic clay waste to settling areas ranges between 20,000 and 80,000 gal/min of 2- to 6-pct-solids material. To accommodate the waste, impoundments covering 400 to 600 acres, with dam heights ranging from 20 to 60 ft, are required (4-5). At the present time, there are approximately 80,000 acres of active and inactive clay settling areas in Florida.

To eliminate aboveground disposal, which appears to be the ultimate goal of Florida's regulatory agencies, new technology is required for processing the phosphate mine wastes. Using only mined-out areas for disposal of the clay wastes requires that the solids content of the material be consolidated from an initial 2 to 6 pct to a final 30 to 38 pct. Generally, at this degree of consolidation, the clay waste, mixed with the sand from the phosphate beneficiation sequence, would fit into the mine cut and eliminate the need for aboveground storage (6).

LABORATORY AND SMALL-SCALE EXPERIMENTS

In bench-scale experiments it was observed that PEO, a nonionic polymer consisting of repeating $\text{CH}_2\text{-CH}_2\text{-O}$ groups, formed strong flocs when added to phosphatic clay waste, which then readily dewatered (7-9). The bench-scale tests were conducted by adding a solution of PEO from a buret to a beaker containing the clay suspension. The slurry was stirred during the entire flocculation-dewatering procedure. Upon addition of a reagent, there was immediate visual evidence of flocculation, but polymer addition was continued until the flocs began to stick together in a coherent mass and clear water began to separate. This degree of solids cohesion must be achieved before dewatering can be continued. At this point, reagent addition was stopped and the separated water was removed by decantation. As the remaining mass was manipulated by stirring, more water separated and was removed. This was continued until no more water could be decanted. The mass was then

removed from the beaker and further dewatered by hand squeezing. The dewatering sequence is shown in figures 1 through 4.

It was observed that if the entire amount of PEO required for flocculation was mixed with the waste in a single step, flocs formed but then disintegrated upon standing, thus indicating that the manipulation process described above is extremely important. The disintegration of the flocs also indicated that PEO cannot be used in a thickener as the flocculant for phosphatic clay waste (7,9).

The effect of the molecular weight of the polymer was also investigated. Polymers having molecular weights of 100,000 to 8 million were investigated. The results indicated that a molecular weight of at least 4 million was required to obtain strong flocs. Maximum results were obtained in the 5- to 8-million molecular weight range (7).

Since the bench-scale studies showed that strong flocs could be formed with PEO if the water was rapidly removed and the flocs manipulated, a variety of mechanical devices such as a horizontal vibrating screen, a static wedge-wire screen, and a rotating trommel screen were investigated to determine if the laboratory results could be scaled up. Based upon this investigation, a trommel screen was chosen for further study. Researchers then investigated parameters affecting the dewatering of phosphatic clay waste such as mixer design, trommel rotation rate, clay waste feed rate, and the effect of PEO concentration on dosage (7,10).

The trommel-screen tests indicated that the lowest PEO dosages were obtained with dilute solutions of PEO. However, using dilute solutions of PEO decreased the capacity of the trommel because of the addition of the dilution water. For example, if 100 gal/min of 3 pct clay waste is treated with 1.3 lb/st PEO⁴ as a 0.025-pct solution, the total volume of solution entering the trommel is 108 gal/min; whereas, if 0.6 lb/st PEO as a 0.0025-pct solution is used, the flow to the trommel is 137 gal/min. One answer to the problem of decreased trommel capacity is to use a hydrosieve screen between the mixer system and the trommel. In previous experiments, material flocculated at 3 pct solids and then passed over a hydrosieve screen dewatered to 12 pct solids (7). Such partial dewatering of the clay waste by a hydrosieve removes most of the water and allows for use of dilute PEO solutions. Thus, the efficiency of the trommel would be increased, since 12 pct material would be treated, rather than 3 pct material.

A series of experiments was conducted using the small-scale hydrosieve and trommel dewatering device shown in figure 5. Three dilute solutions of PEO were tested, and the results are summarized in table 1. The data indicate that a PEO dosage as low as 0.53 lb/st was obtained when a 0.0025-pct-PEO solution was used (10).

Other experiments showed that the PEO dosage could be reduced further by increasing the solids content of the clay waste feed from 3 pct solids to 11.8 pct solids (11). Using 0.0025-pct-PEO solutions, a PEO dosage of 0.38 lb/st was required, as compared to the previous requirement of 0.53 lb/st, to obtain a dewatered product containing 18.2 pct solids. This investigation also indicated that the speed of the mixer used to flocculate the clay waste with PEO is critical when dilute PEO is used.

It was also observed in diluting the 0.25-pct-PEO solution, the concentration of PEO commonly made by dissolv-

⁴All PEO dosages are based on dry solids content of slurry.

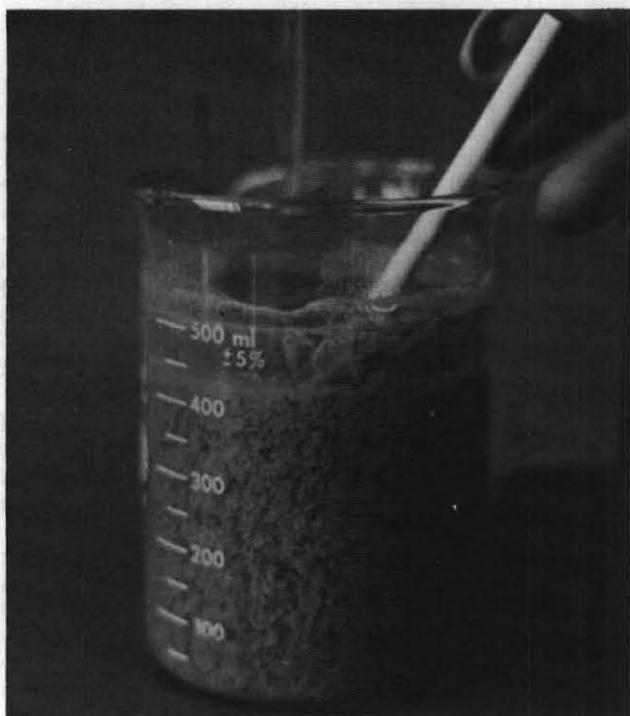


FIGURE 1.—Flocculation of phosphate-clay waste during initial stages of laboratory dewatering sequence.

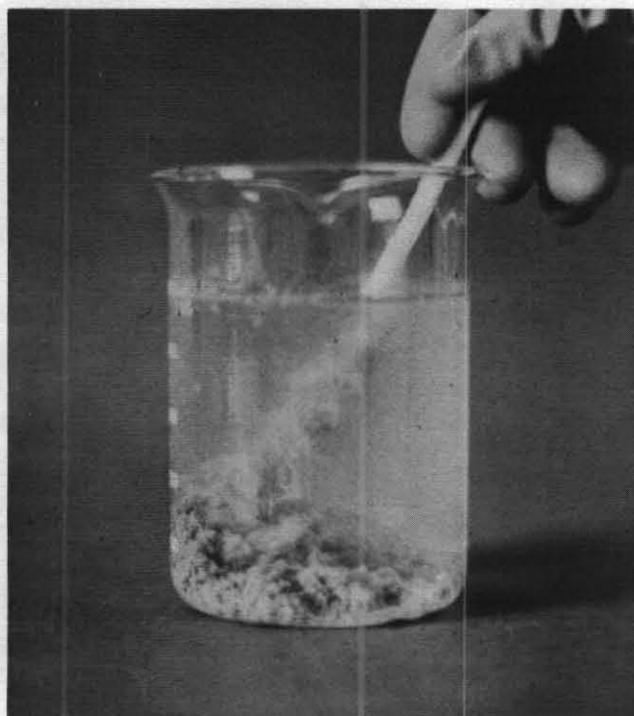


FIGURE 2.—Formation of strong flocs.

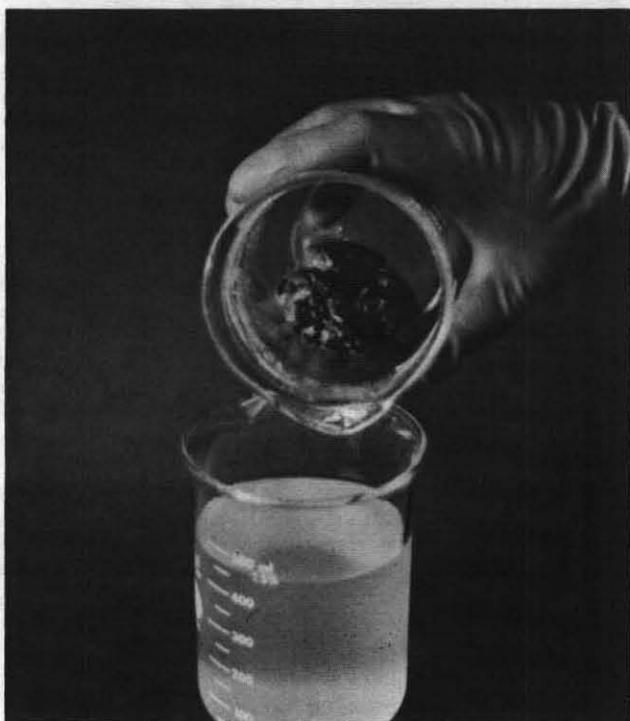


FIGURE 3.—Decanting released water prior to hand squeezing.

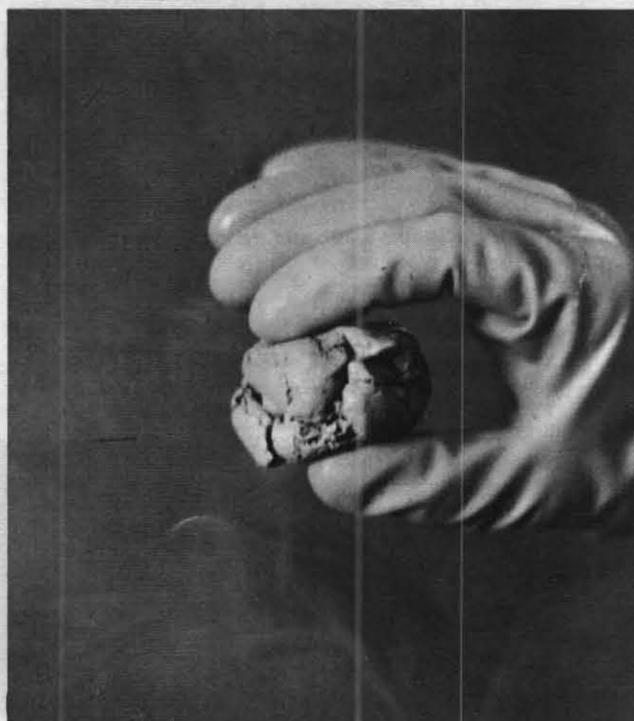


FIGURE 4.—Dewatered solids after hand squeezing.



FIGURE 5.—Small-scale static and trommel screen dewatering of phosphate clay waste.

Table 1. — Dewatering phosphate-clay waste using hydrosieve and trommel screen combination

PEO		Discharge, pct solids	
Conc., pct	Dosage, lb/st	Hydrosieve	Trommel
0.01	1.0	10.0	16.3
.005	.55	12.1	15.5
.0025	.53	8.6	15.6

ing PEO solids in water, that time was required to allow the polymer to uncoil in solution (relaxation time). To obtain optimum results, 15 min was required for a 0.05-pct solution and 45 min for a 0.01-pct solution.

The results from the small-scale experiments indicate that successful dewatering of phosphatic clay waste with PEO requires consideration of the following factors: (1) proper mixing of the clay waste feed and PEO, (2) use of dilute PEO for lower PEO requirements, (3) rapid removal of water as it is released, and (4) physical manipulation of the flocculated material on static and trommel screens, which promotes cohesion of the solids.

FIELD TESTING

Description of Field Test Unit

Based on the small-scale tests, a field test unit (FTU), shown in figure 6, was built to evaluate the PEO dewatering technique on a variety of phosphatic clay wastes. The flow sequence for the trailer-mounted FTU is shown in figure 7. In addition to the dewatering screens, the unit includes a system for making PEO solutions; holding tanks for PEO solutions, slimes, and makeup water; a stirred vessel for mixing PEO solutions with the clay waste; and various pumps to transport solutions, slurries, and consolidated material. The hydrosieve screen is 8 ft wide and 4 ft high. The screen openings are 0.03 in wide.

The trommel screen is 3 ft in diameter by 12 ft long and was constructed of 10-mesh stainless steel. The first 4 ft from the feed end was lined with a second screen of 48-mesh stainless steel to prevent loss of solids as the flocculated material flows onto the screen.

Clay waste was pumped to a 1,200-gal-capacity holding tank on the FTU. From there, the slime was pumped to the mixer at rates ranging from 60 to 265 gal/min. The PEO solution was mixed with the slime, and the resulting flocs were discharged into a trough that overflowed onto the hydrosieve screen. As the flocculated material moved down the hydrosieve by gravity flow, water released by the consolidation of the flocs passed through the screen, and the consolidated material moved down the screen, as shown in figure 8. The water removed by screening contained some flocculated solids which settled rapidly. The partially dewatered material flowed from the static screen into the trommel screen, where a roll was formed, and more dewatering occurred. The dewatered material was discharged from the trommel (fig. 9) into an auger-feed positive-displacement pump, where it was pumped to a pit to allow further dewatering to occur.

Results and Discussion of Data

The FTU was operated at Estech, Inc.'s Silver City Mine near Bartow, FL; Occidental Chemical Co.'s Suwannee River Mine near White Springs, FL; and Mobil Chemical Co.'s Fort Meade Mine in Fort Meade, FL. Initial test results indicated that dewatered material containing greater than 20 pct solids could be obtained.

However, during these field tests, the character of the phosphatic clay waste changed dramatically, with significant effects on the test results. The PEO technique as practiced did not work. Discussions with the mine operators indicated that the active mine area contained considerable quantities of a bluish-green clay commonly referred to as "blue gumbo." X-ray diffraction analysis showed that the bluish-green clay material is typical in phosphatic clay waste from central Florida (1).

Phosphatic clay wastes are generally fully oxidized and are in the calcium-exchanged ion form. Experimentation with the blue gumbo clay indicated that it contained small amounts of sulfides and produced H_2S gas when treated with acid (12). Further experiments indicated that H_2S rapidly attacks PEO, scissoring the polymer chain, thus reducing the molecular weight. Also it was observed that the blue gumbo was not fully calcium-exchanged. To overcome the blue gumbo problem, it was determined that lime should be added, followed by (1) oxidation of the sulfides by agitation or (2) the addition of chemical oxidants such as H_2O_2 and $KMnO_4$, which rendered the clay waste treatable with

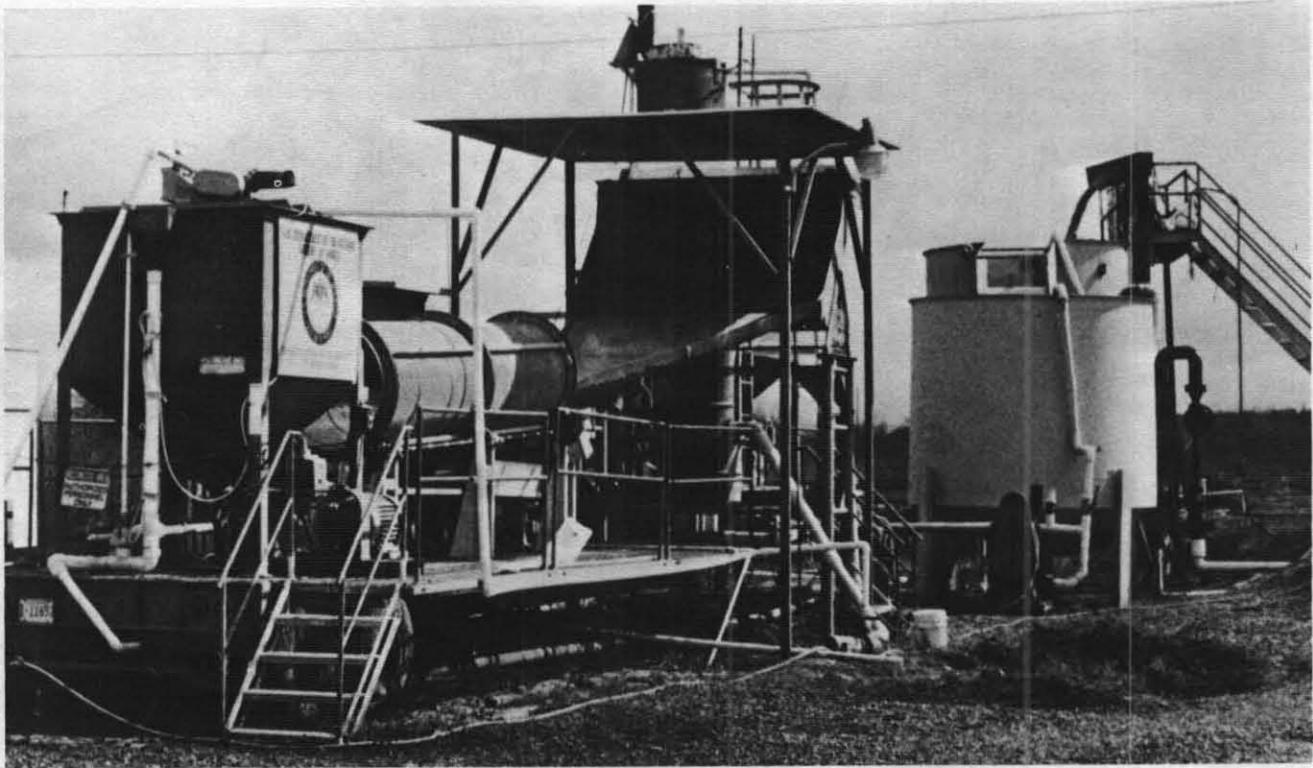


FIGURE 6.—Field test unit (FTU) used for phosphate-clay waste dewatering.

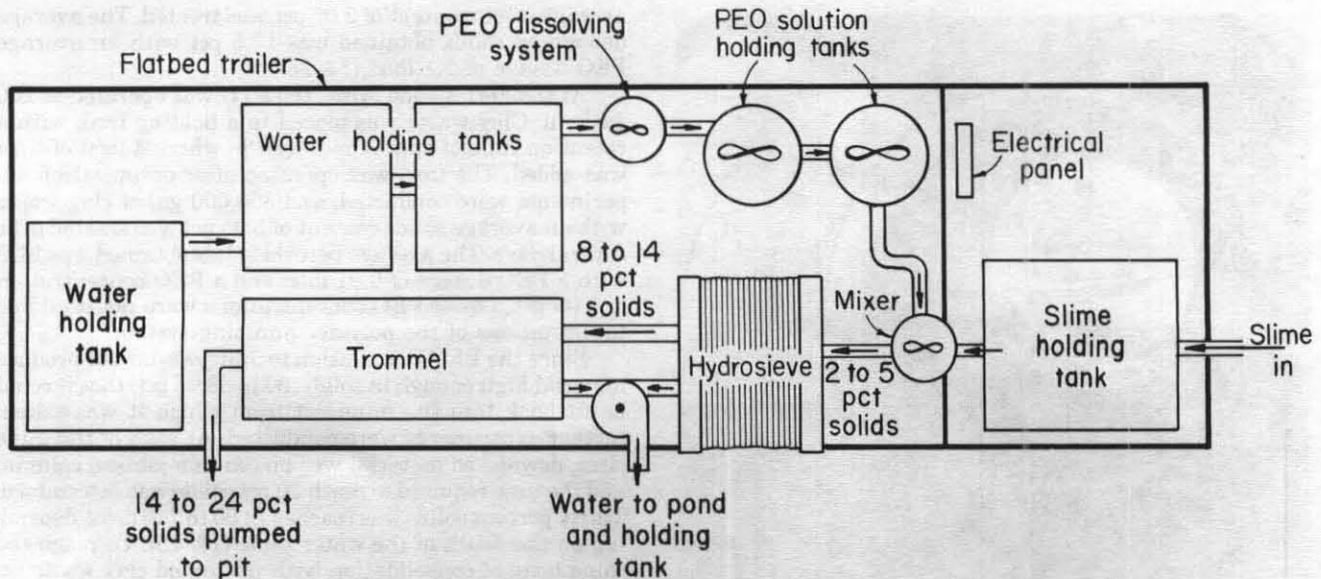


FIGURE 7.—FTU flow sequence.

PEO. Based upon these findings, the clay waste at the Silver City Mine was treated with lime and agitated prior to PEO addition.

The FTU was operated at the rate of 100 gal/min using 0.01- and 0.005-pct-PEO solutions. The best results were obtained when clay waste was treated with 1 to 2 lb/st lime and stirred prior to dewatering. Using 0.01-pct-PEO solutions, 20.4 pct solids was obtained with a dosage of 1.0 lb/st PEO. When using 0.005-pct-PEO, the percent solids produced was 20.6 with a PEO dosage of 0.69 lb/st (13).

The test unit was operated at the Suwannee River Mine at a rate of 165 to 200 gal/min using a 0.25-pct-PEO solution. Using lower concentrations of PEO had little effect on the dosage of PEO required. To obtain solids in the 20 pct range, 5 lb/st lime was required, along with an aging time of up to 16 h prior to PEO flocculation. Operating without aging the clay waste after the lime addition resulted in solids as low as 11 pct. The unit was operated over a 14-day period for 141 h, and a storage pit was filled. During the test, 1.4 million gal of clay waste with an



FIGURE 8.—Consolidated flocs moving down static screen.

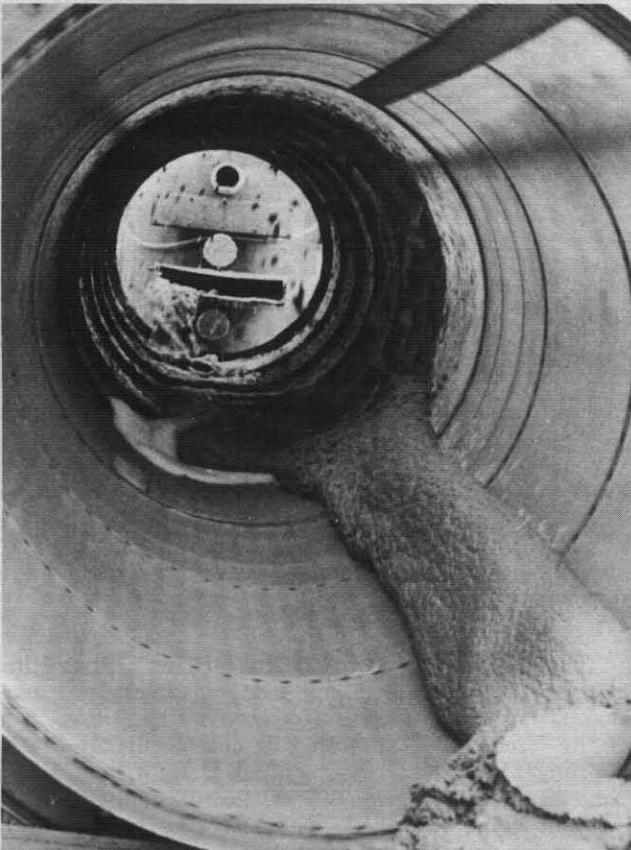


FIGURE 9.—Dewatered material being discharged from trommel screen.

average solids content of 2.05 pct was treated. The average dewatered solids obtained was 18.5 pct with an average PEO dosage of 1.1 lb/st (14–15).

At the Fort Meade Mine, the FTU was operated at 200 gal/min. Clay waste was placed in a holding tank with a retention time of approximately 1/h, where 4 lb/st of lime was added. The unit was operated after optimization experiments were conducted, and 350,000 gal of clay waste with an average solids content of 3.95 pct was treated over several days. The average percent solids obtained was 21.9 with a PEO dosage of 0.91 lb/st and a PEO concentration of 0.05 pct. Lower PEO concentrations were not used due to limitations of the polymer pumping system.

Since the PEO flocculation techniques did not produce material high enough in solids (30 to 38 wt pct) that it could be fit back into the mine cut from which it was taken, further experiments were conducted. At each of the mine sites, dewatered material was placed in a pit or a column, and the time required to reach 30 pct solids was determined. Thirty percent solids was reached in 30 to 140 days, depending on the depth of the water table (10, 15). To reach the same level of consolidation with untreated clay waste requires years to decades (2).

Another important criterion in evaluating a method for clay waste disposal is the terminal density reached by the clay waste after it is placed in the mine cut. Since settling occurs over several years, small-scale laboratory procedures were devised to determine the terminal density (16–18). Tests conducted by a commercial test laboratory indicated that flocculated material treated using the Bureau's PEO technique produced solids of higher density than solids obtained from unflocculated material (19).

Based upon results obtained from the FTU tests, a prototype unit was constructed. The prototype unit was similar

in concept to the FTU, except that it contained six static screens 8 ft wide and 4 ft long and a trommel screen 5 ft in diameter and 18 ft long. The prototype was designed for the Hopewell Mine, located south of Plant City, FL. Small-scale continuous tests had shown that more than 20 pct solids could be obtained with 0.4 lb/st PEO. However, due to delays in construction of the plant, at the Hopewell Mine, the unit was tested at Agric Chemical Co.'s Ft. Green Mine, located on Highway 37 in central Florida. Laboratory tests conducted by the Bureau on the Ft. Green waste indicated that a pretreatment of 1 to 4 h and the addition of several pounds of lime prior to PEO flocculation would be required. Based upon these results, the unit was installed at Ft. Green and operated at the rate of 500 to 1,500 gal/min. Dewatered material containing 20- to 25-pct-solids was obtained using 1 lb/st PEO.

A major problem was encountered during operation of the unit at Ft. Green. It was determined that with a pretreatment of 1 to 2 h, 20 to 100 lb/st lime was required to obtain good flocculation. However, the clay was quite variable in how it flocculated. A variety of laboratory tests and analyses were conducted to determine the reason for this unexpected behavior. Chemical and X-ray diffraction analyses indicated that the clay waste was typical of clay waste indigenous to central Florida. Further analysis of the Ft. Green phosphatic clay waste slurries indicated that small amounts of CO₂ were given off periodically. Also, it was determined that the solution was highly buffered, and there large amounts of lime were required to raise the pH to 9. Based upon these results, it appears that in the initial stages a majority of the lime added to the clay waste at Ft. Green was rapidly precipitated as a carbonate and had little effect on the ion-exchange character of the clay.

A summary of the results from the field testing program is shown in table 2. The data show that for phosphatic clay waste from Florida, each waste is different in its response to the PEO dewatering technique. In other words, the technique is site specific.

Determination of PEO Concentration in Solution

Residual PEO left in solution after flocculation can have an effect on flotation of phosphate values with fatty acids.

Table 2. — Summary of results from field dewatering tests using phosphate-clay wastes

Mine (test location)	PEO dosage, lb/st	Pretreatment time, h	Lime required, lb/st
Suwannee River	1.1	16	5
Ft. Green	1.0	1	20-100
Ft. Meade93	1	4
Silver City69	1	1- 5
Hopewell ¹4	0	0

¹Small-scale tests conducted at Tuscaloosa Research Center. Prototype unit to be operated at Hopewell Mine during the latter part of 1986.

It was determined by Lawver that a concentration of 1 ppm PEO has little or no effect on flotation, whereas a concentration of 10 ppm has a significant negative effect on flotation (6). PEO concentrations of waters recovered from PEO dewatering tests were measured using a technique developed by the Bureau, and the PEO concentration was found to be less than 1 ppm (20-21). The technique is based upon the concept that polymer concentration is related to a measured pressure drop observed during turbulent flow. This drag-reduction technique is capable of measuring polymer concentrations of 10 ppm or less.

SUMMARY

PEO was shown to be an effective flocculant for phosphatic clay waste. In addition, a dewatering technique has been developed for phosphatic clay waste. After flocculation, the clay waste is dewatered on a static screen followed by further dewatering in a trommel screen. In field tests, several different clay wastes were dewatered from a nominal 3 pct solids to greater than 20 pct solids using 0.4 to 1.1 lb/st PEO. It has been shown that for waste slurries containing blue gumbo clays, a pretreatment with lime and oxidation is required to obtain strong flocs that can be dewatered on static and trommel screens. A solids content of 30 pct solids was obtained in 30 to 140 days when the dewatered material was placed in a pit. Also, a method was developed for determining PEO concentration in solutions based on the principles of drag reduction.

CHAPTER 2.—DEWATERING OF COAL-CLAY WASTE SLURRIES FROM PREPARATION PLANTS

A slurry waste product containing fine coal and clay is generated when coal is processed into a low-ash material. This waste product is difficult to handle because of the slow settling rate of the fine-size particles—which are generally less than 28 mesh in size, with the majority less than 325 mesh (22). It has been estimated that approximately 16 million st of waste contained in slurries is generated annually.

The coal-clay waste slurry is usually treated with a flocculant and sent to a thickener to produce a slurry containing 20 to 30 pct solids. The thickened material is disposed of by impoundment behind earthen dams or is further dewatered by filtration, then mixed with coarse mine waste and placed in available mine cuts or stacked on the terrain. The mixed material often reslurries when contacted with rainwater. Disposal by either method is a potential safety and environmental hazard because of the possibility of dam failure or reslurrying that could lead to a mud-flow situation (23-25).

SMALL-SCALE DEWATERING

Laboratory experiments were conducted to determine

if the PEO-dewatering technique could be used for coal-clay waste. Initial tests were conducted on three waste slurries, one from a mine located in West Virginia and two from mines located in Alabama. These coal-clay wastes contained 70 to 80 wt pct of minus 325-mesh material. Initial solids content ranged from 7.3 to 23.9 pct. In each case, consolidated material containing more than 50 pct solids was obtained using 0.3 to 0.5 lb PEO per short ton of dry solids. The addition of from 1 to 3 lb lime per short ton of dry solids was found to be necessary to assure flocculation.

Based upon results of laboratory-scale tests, small-scale continuous tests were conducted on the three coal-clay waste slurries. A variety of dewatering devices such as static and rotary screens was tested. It was determined that a static hydrosieve-type screen was sufficient to dewater the material to 50 pct solids. A schematic flowsheet of the procedure developed is shown in figure 10. Typical data obtained from the small-scale continuous tests are summarized in table 3. The results presented in table 3 are typical results. The dosage of PEO and the percent solids varied as the coal-clay waste varied in clay content. During testing, it was observed that the initial solids content had little or no effect on PEO dosage or the solids content of discharged materials (26-27).

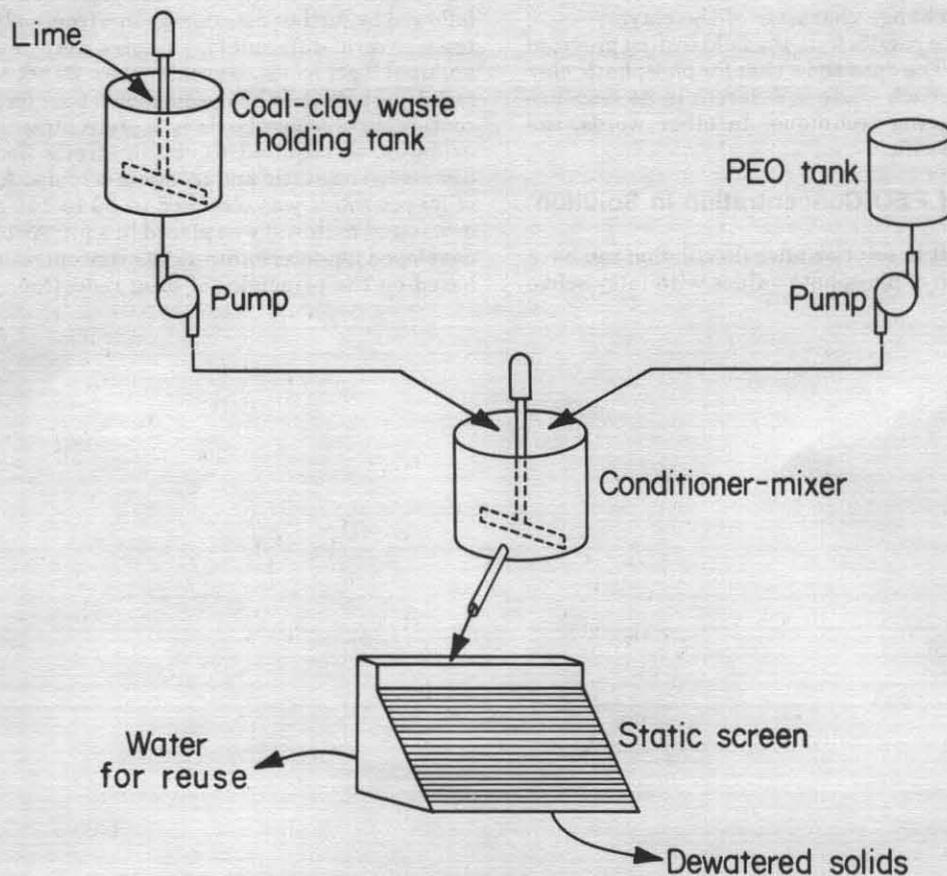


FIGURE 10.—Flow sheet for dewatering coal clay waste.

Table 3. — Summary of results of small-scale dewatering of various coal-clay waste slurries

Slurry sample	PEO dosage, lb/st	Screen discharge, pct solids
West Virginia	0.5	47.1
Alabama A5	51.3
Alabama B3	53.6

FIELD TESTING

Based upon the encouraging results of the small-scale continuous tests, a larger scale FTU was installed at Jim Walter Resources Inc.'s No. 7 Mine at Brookwood, AL. Coal-clay slurry was supplied to the FTU from either the flotation circuit of the preparation plant or from the thickener underflow. The slurry flowed to a holding tank where its pH was adjusted with lime or caustic soda from initial

values of pH 7.5 to 8.5 to a desired value. It was then pumped to a mixing tank, where PEO was added, and the resulting flocculated material was passed over a set of static screens (hydrosieve) for water removal. The hydrosieve was 8 ft wide and 8 ft long and was built in two sections of stainless steel wedge-wire screen. The design allowed the two sections to be inclined at independent angles. The upper section of the screen (8 ft wide, 4 ft long) had slot openings 2.75 in long and 0.02 in wide. The lower section of screen had slot openings 2.75 in long and 0.01 in wide. The FTU is shown in figure 11. Dewatered material from the hydrosieve was conveyed to a pit, where further dewatering occurred. The material reached 65 to 70 pct solids in 3 days.

Initial tests were conducted using the underflow from the flotation circuit. The operating parameters were optimized, including the configuration of the tank where the coal-clay waste and PEO were mixed, the speed of the stirrer

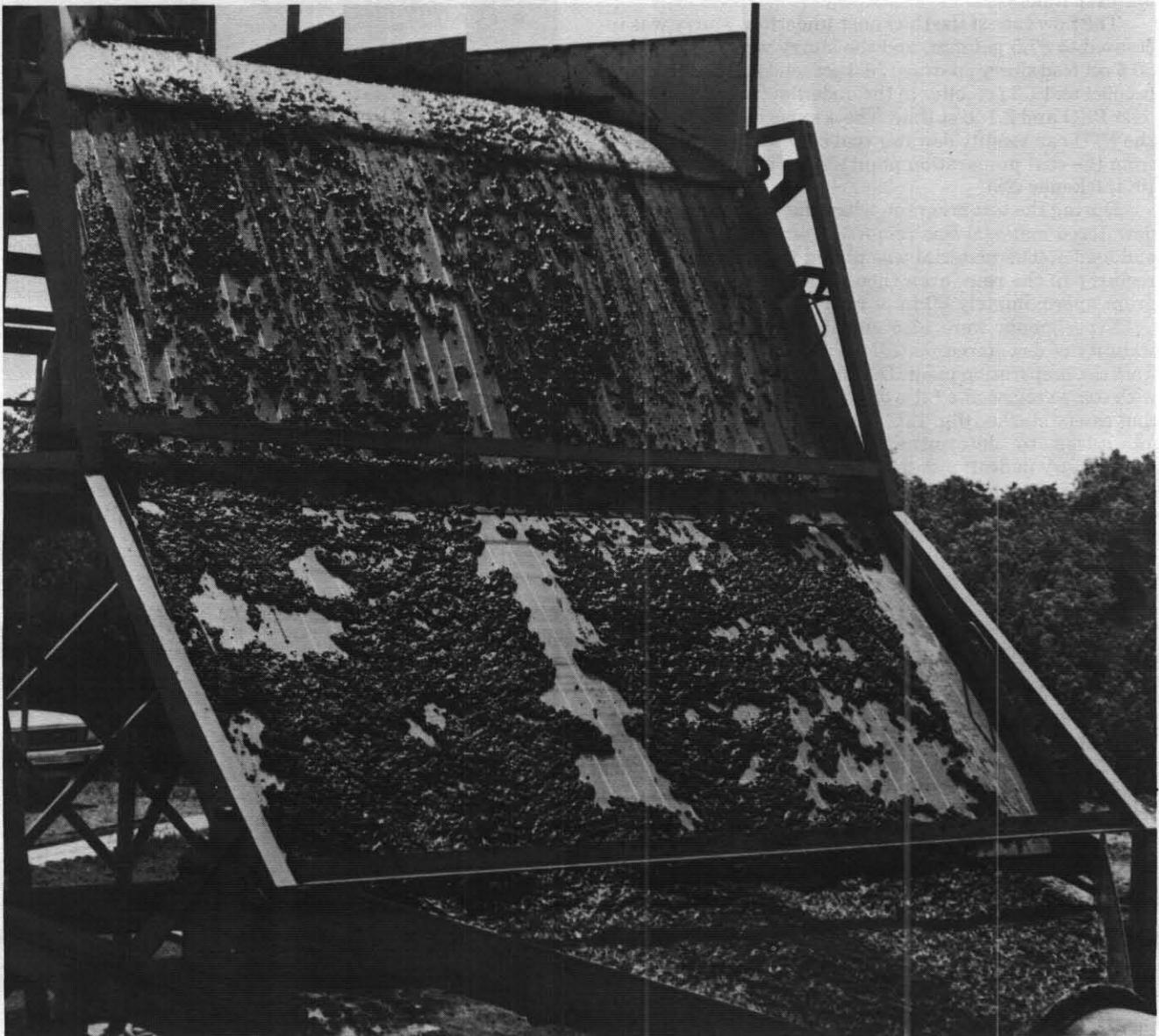


FIGURE 11.—FTU for dewatering coal-clay waste.

blade in the mixer, and the angles of the top and bottom sections of the screen. Using a stirrer blade with a 15.5-in radius, positioned 1 in from both of the mixers, a stirring speed of 40 r/min, the upper screen angle at 58°, and the bottom screen angle at 50°, a 3 pct feed slurry at a rate of 400 gal/min was dewatered to 63 pct solids with 0.7 pct solids in the underflow water. The solids content of this water settled rapidly, producing a solids-free product. These results were obtained repeatedly over several days of operation. Reagent consumption was 0.22 lb PEO and 1.5 lb lime per short ton of solids (28).

Field tests were also conducted on coal-clay waste obtained from the thickener underflow. The optimum operating conditions were the same as the conditions specified above, except for the stirring speed; 30 r/min was found to be optimum.

Using these conditions, the thickener underflow slurry, feeding at a rate of 64.5 gal/min, produced a product containing 57.8 pct solids and 1.4 pct solids in the underflow water. The reagent consumption was 0.09 lb/st PEO and 0.1 lb/st lime.

The flow rate of the thickener underflow slurry was increased to 87.5 gal/min, and the slurry was dewatered. A 20.5 pct feed slurry produced 58.3 pct solids in the dewatered product and 1.3 pct solids in the underflow water, using 0.12 lb/st PEO and 2.1 lb/st lime. These experiments show that the FTU can readily dewater coal-clay waste slurry either from the coal preparation plant's flotation circuit or from its thickener (28).

During the test program, when the pit became full, the dewatered material was removed from the pit by a front-end loader. The material was placed in a pile and did not reslurry in the rain, even though during the testing program approximately 60 in of rain fell.

Experiments were also conducted to determine the stability of dewatered material mixed with coarse refuse from the preparation plant. Dewatered material was mixed with coarse refuse at a ratio of 1:5 to form a stable material that can be stacked (fig. 12). These mixtures are now being monitored to determine their long-term stability. Preliminary indications are that the material is stable and can be disposed of using the same method now used to dispose of the coarse waste alone.

Upon completion of the testing at the No. 7 Mine at Brookwood, the FTU was moved to the Tennessee Valley Authority's (TVA) Paradise complex near Drakesboro in western Kentucky. The waste at Paradise is finer than that tested at the No. 7 Mine, as shown in table 4.

In initial experiments it was determined that 38 to 42 pct solids was typically obtained. This was considerably lower than the 55 to 60 pct obtained at the No. 7 Mine. To increase the solids content, a trommel screen 20 in in diameter and 8 ft long was added to the unit. Dewatered material exiting the static screen entered the trommel and was further dewatered (fig. 13). This further treatment increased the solids content of the dewatered material to 48 to 51 pct, with a PEO dosage requirement of 0.12 to 0.15 lb/st and a lime dosage of 1.3 lb/st. Investigations also indicated that by increasing the capacity of the trommel, an increase in dewatered solids amounting to several percent, could be obtained.

Dewatered material was placed in a pit and continued to dewater, reaching 69 pct solids in 1 week. This material was quite stable and could be handled with heavy equipment such as a front-end loader.

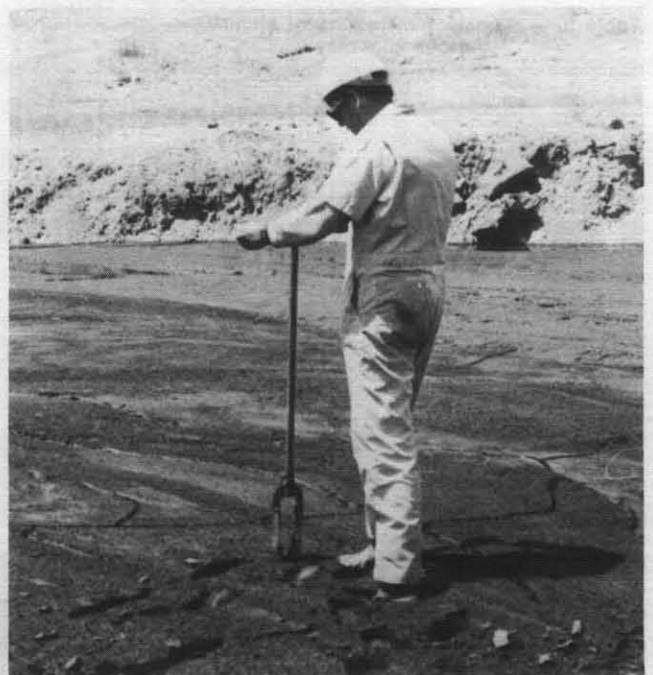


FIGURE 12.—Dewatered coal-clay waste material mixed with coarse refuse from preparation plant at 1:5 ratio.

Table 4. — Screen analysis of coal-clay waste, weight-percent

Screen size, mesh	No. 7 Mine	Paradise complex
Plus 28	13.2	2.0
Minus 28 plus 48	15.0	6.0
Minus 48 plus 100	13.3	16.9
Minus 100 plus 200	10.2	14.6
Minus 200 plus 325	4.7	8.6
Minus 325	43.6	51.9

Further testing was conducted in which coarse refuse from the preparation plant was mixed with dewatered material at a ratio of 4:1. A stable, stackable mixture resulted. At ratios below 3, the material did not stack and would slowly flow.

Based upon published results from the initial FTU experiments, a considerable number of samples were submitted by mine operators for bench-scale amendability testing. Typical results are shown in table 5. The results obtained varied depending on the character of the coal-clay waste sample.

Table 5. — Results of laboratory dewatering tests on coal-clay wastes

Waste sample	PEO dosage, lb/st	Solids content, pct	
		Initial	Dewatered product
Kentucky	0.23	34.1	55.2
Missouri A19	7.7	44.5
Missouri B35	24.8	48.4
Ohio A34	16.3	48.9
Ohio B35	10.7	48.2
West Virginia58	23.9	56.9



FIGURE 13.—FTU with trommel screen at Paradise complex.

SUMMARY

Coal-clay waste is readily flocculated with PEO. A technique utilizing a static screen or a combination of a static screen followed by a trommel has been successfully used to dewater PEO flocculated coal-clay waste. Field tests on two wastes have shown that solids contents in the 50 to 60 pct range can be obtained using 0.12 to 0.15 lb/st PEO. Dewatered material continues to dewater and reaches 65

to 70 pct solids in 3 to 7 days. Dewatered material has shown good stability and does not reslurry easily even when subjected to heavy rainfall. It has been demonstrated that dewatered material, when mixed with coarse refuse from the preparation plant, forms a stable product that can be disposed of by stacking on an available terrain. Data also indicated that the final solids content obtained by the PEO dewatering technique depends on the particle size of the waste being tested.

CHAPTER 3. — DEWATERING OF MISCELLANEOUS CLAY WASTE SLURRIES

Based upon the successful dewatering experiments and field assessment tests on phosphatic and coal-clay waste, a wide variety of other waste slurries was studied.

ULTRAFINE BENTONITIC WASTE

Bentonites are used in drilling muds, foundry sands, and pelletizing of taconite iron ores (29). The major source of raw bentonite is in Wyoming. Raw bentonite is mined and then refined to remove roots, coarse quartz, and bentonite particles larger than several micrometers. The rejected material from the refining process is discharged from the plant as an aqueous slurry and disposed of by impoundment. This waste slurry settles poorly, thus requiring large impoundment areas.

A sample of bentonitic waste containing 12 pct solids was obtained from a refiner in Texas. X-ray diffraction analysis of the material showed that it had major amounts of quartz and bentonite, with minor to trace amounts of calcite and mica and/or illite. It also contained trace amounts of plagioclase, zeolite, and cristobalite. The material was 100 pct minus 400 mesh.

The procedure for small-scale continuous testing consisted of pumping the slurry at the rate of 1 gal/min to an agitator-type mixer unit, where PEO was added. The flocculated material flowed from the mixer into a rotary trommel screen, where water was removed through the screen; the solids formed a roll and moved down the screen as shown in figure 14. The trommel screen was constructed of type 304 stainless steel mesh; it was 36 in long and had an 8-in diam and 10-mesh screen openings.

Initial batch experiments indicated that PEO formed weak flocs with the bentonite sample (a sodium form of montmorillonite). Previous research (12, 30) indicated that

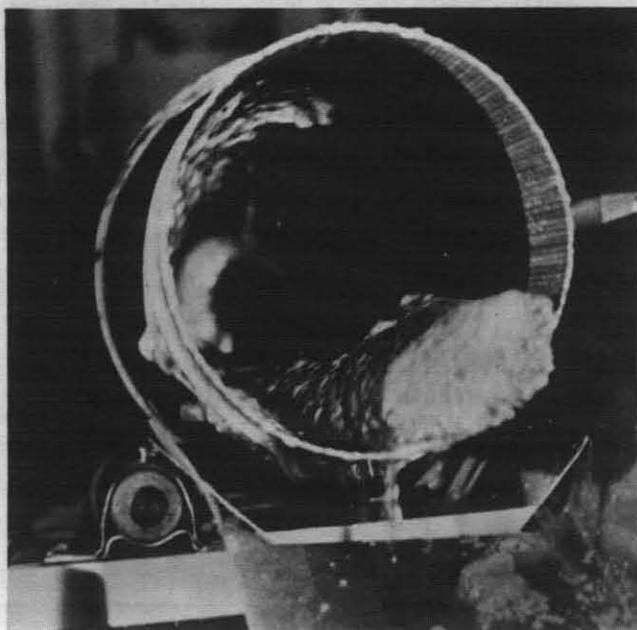


FIGURE 14.—Trommel screen dewatering bentonitic clay waste.

PEO forms stronger bonds with a calcium montmorillonite than it does with a sodium montmorillonite. Therefore, the bentonite slurry was treated with calcium ion in the form of lime or calcium chloride (CaCl_2). Strong flocs that dewatered readily were produced when PEO was added to the slurry.

Based upon the results of the preliminary batch experiments, small-scale continuous tests were conducted. In these experiments, the amount of CaCl_2 used was 20 lb/st, based on the dry solids content of the bentonite slurry, and the concentration of the PEO solution was 0.005 pct. Table 6 shows that all the dosages investigated produced trommel discharges that ranged from 38.7 to 41.5 pct solids. The water recovered from the trommel had flocculated solids that settled rapidly to produce water with a turbidity of 10 to 19 NTU, except for the 0.2-lb/st dosage, which produced water with 300 NTU turbidity. For discharge into a pristine environment, water is usually required to have a turbidity of 10 NTU or less.

Other experiments were conducted changing the PEO concentration from 0.05 to 0.01 pct and substituting lime for calcium chloride (30). These tests indicated that 0.05 pct PEO and CaCl_2 gave superior results.

Table 6.—Effect of PEO dosage on dewatering efficiency of 0.005-pct-PEO solutions for bentonitic waste

PEO dosage, lb/st	Trommel screen, pct solids		Final water turbidity, ¹ NTU
	Discharge	Underflow ²	
0.52	38.7	0.09	10
0.40	41.5	.14	13
0.34	40.9	.50	NAP
0.26	41.0	.35	19
0.20	41.0	5.26	300

NAP Not applicable.

¹Turbidity measurement taken after 5 min, thus allowing suspended solids to settle.

²Water recovered from trommel screen.

POTASH-CLAY WASTE

Potassium, supplied as potash, is one of the three basic nutrients, along with nitrogen and phosphorus, used to promote plant growth and increase crop yield. About 95 pct of the domestic consumption of potash is for fertilizers, and the remaining 5 pct is for chemicals (31). In 1983, U.S. consumption of potash totaled 9.7 million st, of which 7.3 million st was imported. Production data indicate that 85 pct of the total domestic production in 1983 was from the Permian Basin region near Carlsbad, NM (32).

Over the past 40 yr, vast tonnages of the high-grade New Mexico potash ores have been mined and processed. However, the high-grade deposits have now been virtually depleted, leaving high-clay-bearing, low-grade ore reserves.

When high-clay-bearing deposits are processed, the ore is deslimed by washing with saturated brine solution; the clay particles are removed as a suspension in the saturated brine. The clay-slime waste slurries are thickened to approximately 8 pct solids so that part of the brine can be recovered for recycling to the processing plant. Since the brine is also saturated with potassium oxide (K_2O) values, maximum recovery is advantageous. When treating a high-grade ore containing 20 pct K_2O and 1 pct clay slimes, a typical potash plant loses only about 3 st of K_2O values in

the slimes for every 100 st of concentrate produced. However, a decrease to 16 pct K_2O and an increase to 4 pct clay slimes increases losses to 14 st of K_2O values in the slimes for every 100 st of concentrate produced (33).

The problem of K_2O losses has grown worse in recent years. The average K_2O content of the Carlsbad ores decreased from 17.5 pct in 1966 to 13.8 pct in 1979, and then still further, to 13.6 pct in 1980 (32). Consequently, the loss of K_2O values in processing continues to increase, which has brought about greater interest in brine recovery techniques.

Both laboratory and small-scale continuous experiments were conducted on a potash-clay waste brine obtained from an operating potash producer in Carlsbad, NM. The sample contained 7.4 to 8.6 pct clay solids along with undissolved NaCl and minor amount of KCl. The aqueous part of the slurry contained considerable amount of dissolved chlorides of sodium and potassium and had a specific gravity of 1.22.

In laboratory experiments, the concentration and molecular weight of the PEO were varied. The results are shown in table 7. The data indicate that a 0.01-pct-PEO solution resulted in the lowest dosage. The data also show that with a dilute solution of PEO, a majority of the undissolved halides in the slurry were dissolved during flocculation.

Small-scale continuous tests were conducted using 0.01-pct PEO made using various amounts of the brine solution, as shown in table 8. Solids contents ranging from 13.8 to 16.4 pct were obtained; however, these values were considerably lower than those obtained in laboratory experiments. In attempts to increase the solids content, the discharge from the trommel screen was treated on a belt-roller device (34), as shown in figure 15. This increased the solids content to the 19- to 20-pct range.

Table 7.—Debrining of potash-clay waste slurry¹

PEO Conc., pct	Dosage, lb/st	Debrined solids		Density of recovered brine
		pct	Clay content, pct	
5-MILLION-MOL-WT PEO				
0.25	19.4	37.9	89.7	1.24
.10	9.0	39.2	94.9	1.20
.05	6.1	38.6	93.8	1.19
.01	2.6	33.7	95.0	1.13
8-MILLION-MOL-WT PEO				
0.25	27.3	38.3	97.9	1.20
.10	12.2	37.1	97.0	1.19
.05	6.6	37.8	99.5	1.17
.0	1.8	35.4	98.6	1.15

¹Initial clay solids; 7.39 pct of total solids.

Table 8.—Small-scale continuous tests on potash-clay waste slurry

Brine content of PEO solution, pct	PEO dosage, lb/st	Trommel, pct clay	
		Discharge	Underflow
0	1.50	16.4	0.07
34	1.10	15.4	.05
54	2.21	15.7	.02
74	2.03	13.8	.25

TALC SLURRY

Talc, a hydrated magnesium silicate, is a major constituent of soapstone, which is used in the manufacture of thermal and electrical insulators. Talc also finds applications as filler for use in the paper, rubber, and textile industries;

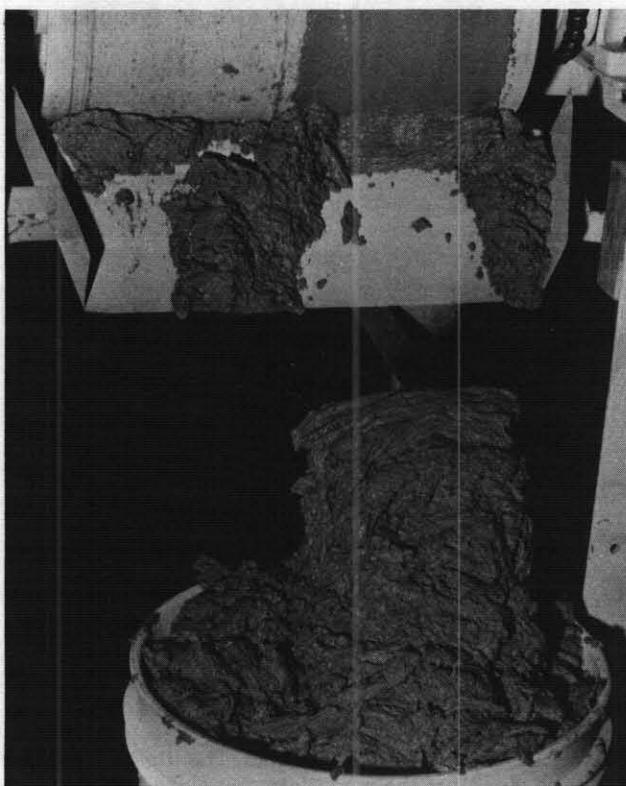


FIGURE 15.—Discharge of debrined potash-clay waste solids from belt-roller device.

in the preparation of soap, cosmetics, lubricating and special polishing agents; as well as in the paint and ceramic industries. Domestic production of talc in 1980 totaled well over 1.4 million st (35). In processing talc, ultrafine waste materials, which respond poorly to conventional separation and dewatering techniques, are often produced. The waste materials are usually impounded in a series of disposal ponds, but even after flowing through as many as nine ponds, the water is still too turbid for reuse or discharge into streams.

Samples of talc waste slurry containing 3.5 to 9.7 pct solids were obtained from an operating plant in Vermont. Examination of the solids by X-ray diffraction identified talc and chlorite as major components, magnesite and dolomite as major-to-minor components, and kaolinite as a minor component. Laboratory tests were conducted on the samples (36). Table 9 shows the results of dewatering tests using PEO of two different molecular weights as flocculants on slurries of 3.5, 6.3, and 9.7 pct solids. In these tests, 0.05-pct

Table 9.—Laboratory tests using PEO to dewater talc slurries (PEO concentration, 0.05 pct)

Initial solids content of slurry, pct	PEO dosage, lb/st	Solids content of dewatered product, pct	Turbidity, NTU		
			5 min	1 h	4 h
5-MILLION-MOL-WT PEO					
3.5	1.02	50.5	14.0	15.0	14.0
6.3	.22	54.8	12.0	10.0	9.0
9.7	.14	56.2	18.0	11.0	9.5
8-MILLION-MOL-WT PEO					
3.5	0.84	49.0	20.0	16.0	15.0
6.3	.34	50.5	21.0	16.0	15.0
9.7	.16	53.9	27.0	11.0	16.0

solutions of the polymers were used to flocculate the slurries.

The data show that when using a concentration of 0.05 pct, the dosage of PEO required to dewater the slurry decreased as the initial solids content increased. Maximum dewatering (highest solids content of dewatered product) was achieved at higher initial solids contents and lower dosages without any significant change in water quality.

Although the dosage required to dewater talc slurry with PEO was nominal and the solids content of the dewatered product was very high, the dewatered product was dry and brittle. Small-scale continuous dewatering tests using a trommel screen as the dewatering device were unsuccessful due to the brittle nature of the flocs formed with PEO (37).

MICA-CLAY TAILINGS

The major source of flake (scrap) mica in the United States is that which is recovered as a byproduct or coproduct from domestic feldspar or kaolin beneficiation (38). A sample of waste slurry from a mica producer in North Carolina was obtained for testing purposes. X-ray diffraction analysis of the sample indicated that the waste was essentially a mixture of fine mica particles and kaolin clay. Initial tests indicated that PEO flocculated the slurry, but clear supernate was not obtained. In a second series of tests, additions of lime and CaCl_2 were investigated. The lime did not improve the clarity of the supernate, but CaCl_2 did—it gave a clear supernate. Based on these results, a series of tests was conducted in which the amount of CaCl_2 was varied as shown in table 10. The data indicate that with an addition of 15 lb/st CaCl_2 followed by PEO flocculation, a clear supernate and strong flocs were obtained.

Table 10.—Dewatering of mica-clay waste slurry

CaCl_2 , ¹ lb/st	PEO dosage, ² lb/st	Dewatered material, wt pct
10	31.45	29.7
11.6	41.31	30.2
15	1.23	27.9
20	1.30	28.2
30	1.35	28.1
50	51.77	NP

NP No product.

¹ CaCl_2 added as a 10-pct solution.

²0.01-pct-PEO (8 million-mol-wt) solution was used.

³Solution was cloudy.

⁴Solution was yellow.

⁵Flocs broke down.

URANIUM MILL TAILINGS

A sample of uranium mill tailings from a New Mexico mine was furnished by the Colorado School of Mines

Research Institute. The material had been cycloned to remove the sand fraction prior to dewatering experiments. The resulting material had a solids content of 15.4 pct and was 66 pct minus 325 mesh. X-ray diffraction analysis showed 60 pct quartz, 20 pct plagioclase, 15 pct orthoclase, and 5 pct unidentified substances. A series of dewatering experiments was conducted, and the results are summarized in table 11 (36).

Decreasing the PEO concentration from 0.25 to 0.01 pct reduced the dosage from 1.13 to 0.39 lb/st for the 5-million-mol-wt PEO with little ill effect on the dewatered product, and from 1.02 to 0.18 lb/st for the 8-million-mol-wt PEO, with increased solids content at 0.18 lb/st. The results indicate that uranium tailings are easy to consolidate, especially with the higher molecular weight polymer, since 67 pct solids was obtained with a PEO dosage of only 0.18 lb/st.

Table 11.—Effect of PEO concentration on PEO dosage for uranium tailings

PEO conc., pct	Dosage, lb/st	Solids content of dewatered product, ¹ pct
5-MILLION-MOL-WT PEO		
0.25	1.13	46
0.10	.46	49
0.05	.35	47
0.01	.39	42
8-MILLION-MOL-WT PEO		
0.25	1.02	64
0.10	.50	60
0.05	.48	62
0.01	.18	67

¹Initial solids content, 15.4 pct.

SUMMARY

The PEO dewatering technique has been shown to be effective in dewatering a wide variety of mineral wastes. For bentonitic waste, dewatered material containing 38.7 to 41.5 pct solids was obtained using 0.20 to 0.52 lb/st PEO. To obtain good flocculation for the bentonitic waste, calcium ion had to be added prior to PEO addition. Potash-clay waste was dewatered to 19 to 20 pct solids using 1.10 to 2.21 lb/st PEO and a trommel screen followed by treatment on a belt-roller device. Talc waste slimes were dewatered to 56.2 pct solids using a PEO dosage of 0.14 lb/st in laboratory tests, but the flocs formed were not amenable to dewatering on a trommel screen. Waste from a mica ponding plant was dewatered to 27.9 pct solids using an addition of 15 lb/st CaCl_2 and 1.23 lb/st PEO. Tailings from a uranium recovery mill were dewatered to 67 pct solids, using a PEO dosage of 0.18 lb/st PEO.

CHAPTER 4.—THE MECHANISM OF PEO-CLAY INTERACTION

The results obtained during this research program indicate that PEO forms strong flocs that can be dewatered on a static screen and rotating trommel. During various stages of the research program, other polymers that have been successfully used in industry to flocculate clay waste, such as polyacrylamides, were compared with PEO. The flocs formed with these polymers were not strong enough to be dewatered using static screen and trommel screen. These flocs would disintegrate during screening. Also, when PEO was used as the flocculant, the dewatered material had a higher solids content than that obtained with other commercially used flocculants. For example, for phosphate-clay waste, polyacrylamides produce 12 pct solids in a thickener versus more than 20 pct solids for PEO-flocculated material subjected to screening (39). Coal-clay waste thickened with polyacrylamide produces 10 to 30 pct solids (22), whereas PEO produces 50 to 60 pct solids. To determine why PEO is unique, considerable fundamental research was conducted.

In the early stages of the Bureau's dewatering research program, experiments were conducted to determine if additives that change the zeta potential of phosphatic clay waste and additives that could participate in hydrogen bonding could be used to decrease the dosage of PEO required (40). However, the results indicated that such additives had a negligible effect on PEO flocculation. These results led to the conclusion that more than classical surface chemistry techniques would have to be used to understand the PEO flocculation system.

RHEOLOGICAL STUDIES

It is generally believed that PEO flocculates clay slurries clay by the development of "bridges" between flocs. Tattersall (41) developed an equation that was modified by Lapasin (42) which describes the breaking of links between particles in portland cement paste. This equation was further modified for studies in which calcium-exchanged montmorillonite clay was flocculated and subjected to shear (43). The calcium-exchange form was chosen for this research since it is the major constituent of Florida phosphatic clay waste (1). The investigation showed that small flocs are formed by interaction of PEO with the clay. These smaller flocs are then bridged into larger aggregates. Also, it was shown that when shear is applied to the flocculated clay, the large aggregates break down first, resulting in small flocs. To further degrade the small flocs, considerable shear is required.

In another series of experiments, viscosity measurements were made on ion-exchanged montmorillonite slurries of the following cations: Na^+ , Li^+ , NH_4^+ , Ca^{2+} , Mg^{2+} , and La^{3+} . The data were analyzed and the measured viscosities were separated into Newtonian and non-Newtonian components using the Ree-Eyring equation (44). The magnitudes of the Newtonian and non-Newtonian viscosity components were found to be dependent upon the floc volume, a parameter controlled by the degree of particle interaction, and the thickness of the individual montmorillonite platelets (tactoids). It was also determined that the number of tactoids was directly related to the hydrated-radius-to-charge ratio, as shown in figure 16. The floc volume was also shown to be related to the hydrated-radius-to-charge ratio, as shown in figure 17.

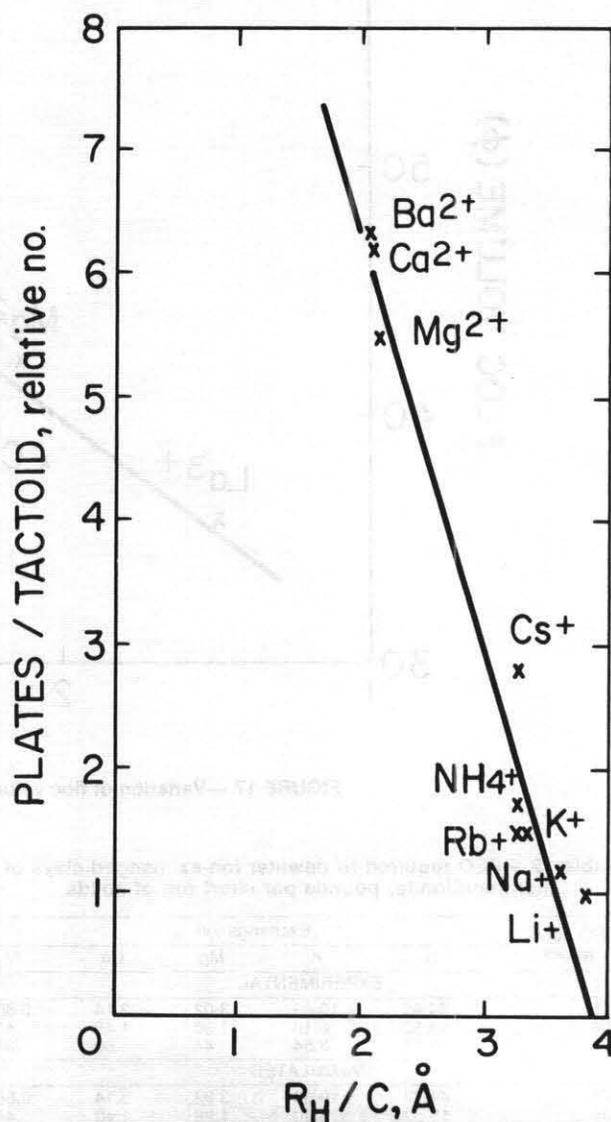


FIGURE 16.—Linear relationship between relative number of plates per montmorillonite tactoid and hydrated-radius-to-charge ratio (R_H/C) for some common exchange ions.

EFFECT OF CLAY EXCHANGE ION ON DEWATERING

As previously mentioned, the addition of calcium ion in the form of CaO or CaCl_2 improved the flocculation of many clay wastes with PEO. To determine the effect of exchange ion on flocculation, experiments were conducted in which the sodium, potassium, calcium, aluminum, and magnesium clays of montmorillonite and attapulgite were dewatered with PEO. It was determined that dewatering techniques could be described by the following empirical relationship (45-46):

$$A^n = kC + b,$$

where A is the amount of polymer, C is the concentration of the polymer solution used for dewatering, and n , k , and

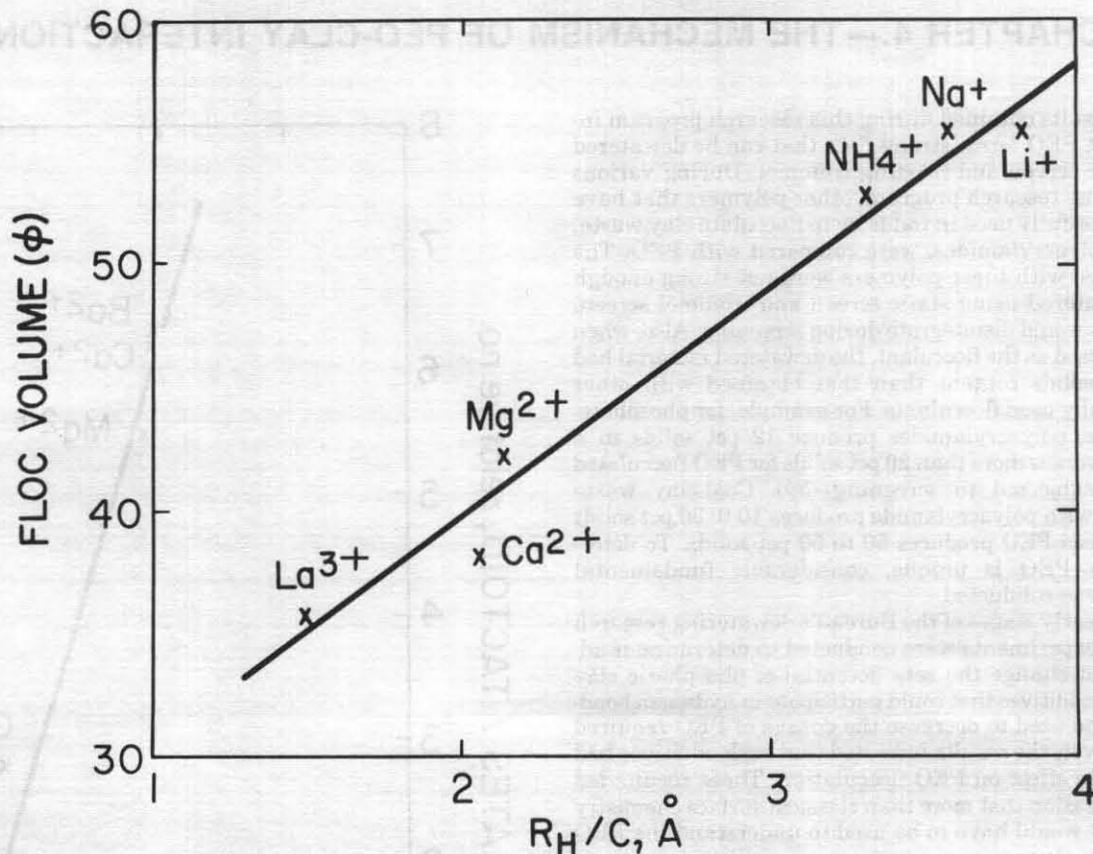


FIGURE 17.—Variation of floc volume with hydrated-radius-to-charge ratio.

Table 12.—PEO required to dewater ion-exchanged clays of montmorillonite, pounds per short ton of solids

PEO conc., wt pct	Exchange ion				
	Na	K	Mg	Ca	Al
EXPERIMENTAL					
0.25	44.46	10.86	3.02	3.14	0.68
0.05	13.52	6.16	1.30	1.46	.44
0.01	9.74	3.54	.44	.50	.34
CALCULATED					
0.25	44.72	10.86	3.02	3.14	0.68
0.05	15.52	6.20	1.28	1.40	.44
0.01	7.80	3.46	.46	.62	.34

b are empirical constants. An example of how the PEO dosages obtained experimentally compare with those obtained using this equation for different exchange ions of montmorillonite clays is shown in table 12. Similar correlations between experimental and calculated values were obtained for the different ion-exchange forms of attapulgite clay.

Evaluation of the data revealed that the exponent n was related to the charge on the exchange ion of the clay divided by the hydrated radius of the exchange ion. It was also determined that k was a function of the square of the ionic radius divided by the charge on the exchange ion.

These observations, when applied to the PEO-dewatering sequence, provide insight into the mechanism of how

PEO flocculates the clay particles. The flocculation technique as practiced by the Bureau is a two-stage process. First, the PEO is mixed with the clay waste, forming strong flocs, and water is released. In the second step, the flocs are screened to remove the released water and in the process are mechanically altered on the screens. The coefficients n and k describe this two-stage process. The exponent n relates to the process of uncoiling the random PEO coil under the influence of the exchange ion and shear. The coefficient k is important in the second stage of the dewatering process, during which the initial open floc structure is formed into an edge-to-edge and face-to-face interaction between the clay particles. Since PEO is nonionic, the initial bond between the clay waste and the PEO is a water-bridge type. An understanding of how this water bridge interacts with the exchange ion on the clay will lead to a mechanistic picture of the PEO flocculation process.

The hydration of montmorillonite is illustrated in figure 18 (47). The initial water bridge between the clay particle and PEO is not strong; the flocs formed disintegrate if the released water is not removed (7-9). Therefore, the initial bond is probably between the filling water and PEO. As the water is removed and the flocs are screened, a stronger bond is formed with the hydration shell of the cation exchange ion of the clay, as shown in figure 19. Once this stronger bond is formed, the floc is resistant to breakup.

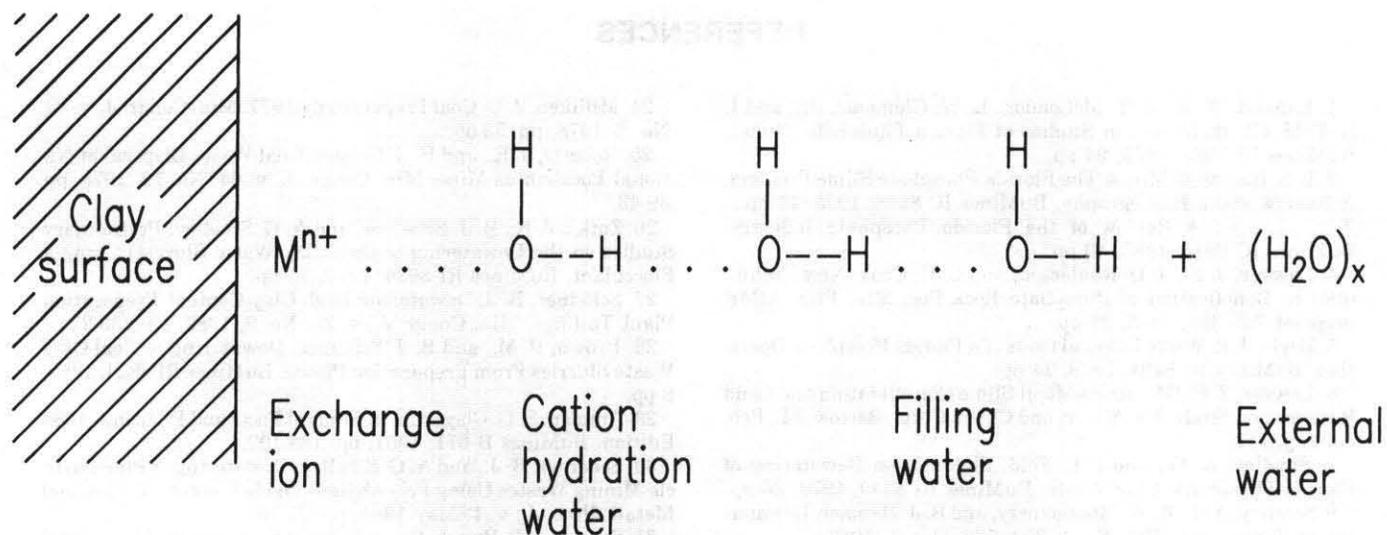


FIGURE 18.—Water structure of montmorillonite clay.

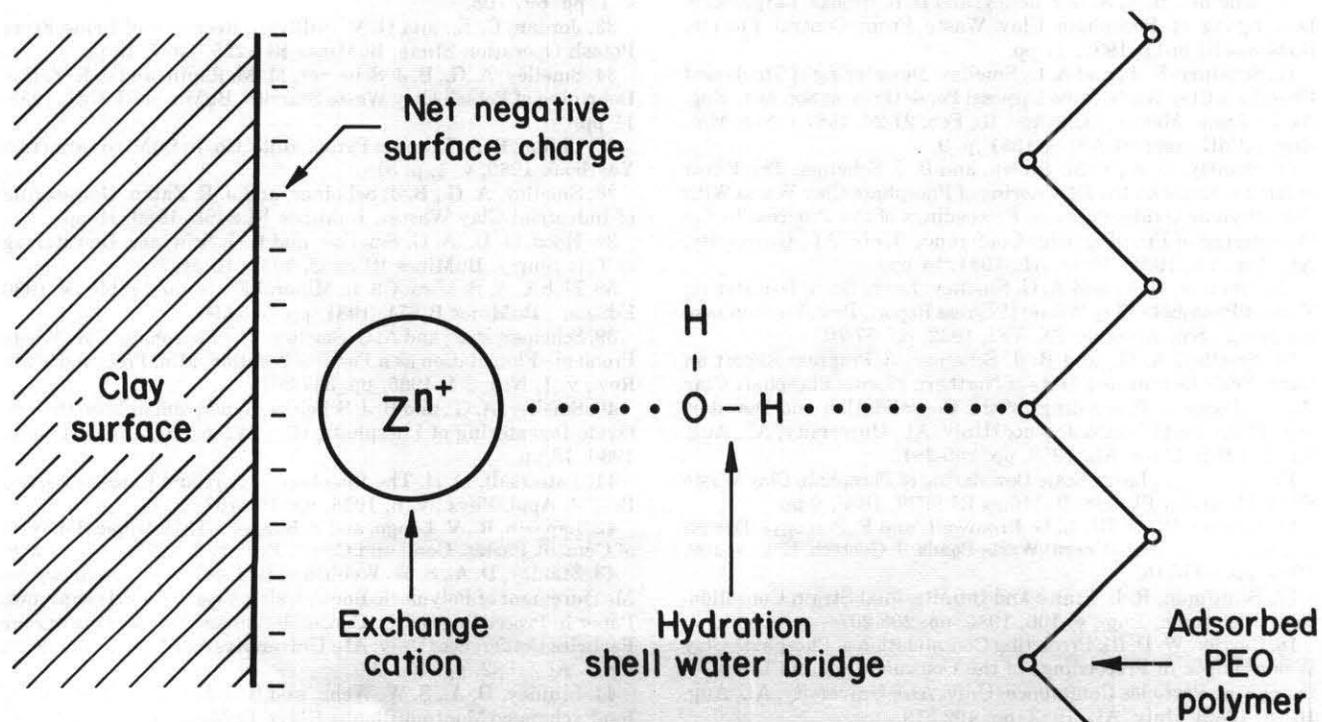


FIGURE 19.—Proposed mechanism for attachment of PEO to montmorillonite.

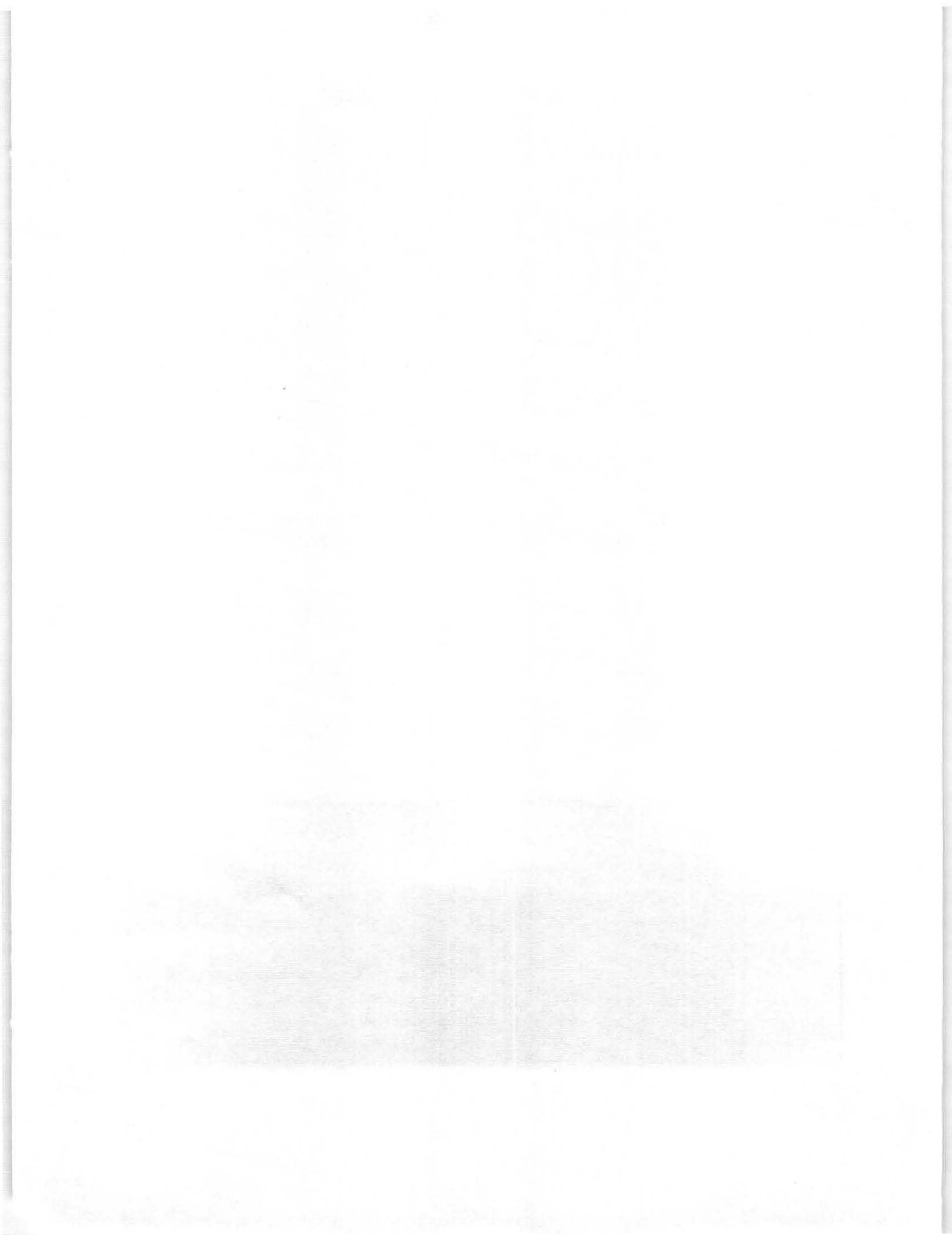
SUMMARY

Fundamental studies using surface chemistry, rheology, and ion-exchange techniques have provided insight into how PEO interacts with waste slurries containing clay or claylike particles. Initially, PEO forms small flocs that are subsequently bridged into larger flocs. These larger flocs

are strong enough to undergo mechanical deformation on the screens. The strength of the flocs comes from strong interaction between the oxygen molecules of the polymer and the exchange ion found in the clay. Bond strength increases inversely with the radius of the exchange ion divided by the charge.

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