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Electrokinetic Clarification of Colloidal Suspensions

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A novel apparatus has been developed for the electrophoretic clarification of concentrated colloidal suspensions. The laboratory equipment is capable of being scaled up to much larger capacity. The apparatus was designed to process suspensions in either batch or semicontinuous operation, and it has been used to demonstrate the removal of coal/clay colloidal matter from the effluent of a coal-washing facility. In addition, a parametric study of sodium−bentonite suspensions in batch operation was performed to elucidate the effects of initial colloid and electrolyte (NaCl) concentrations on the rate of clarification and the instantaneous power requirements. It was found that an increase in either the initial particle or electrolyte concentration (up to 50 mg of NaCl/L) caused a decrease in the instantaneous power levels but increased the time necessary to reach a given solution clarification. Typical electrophoretic clarification experiments resulted in 99.9% removal of the colloidal matter. In semicontinuous operation it was shown that a 0.2 wt % sodium−bentonite suspension could be clarified to 2.6×10^{-4} wt % in 13.5 h without the addition of flocculants or other chemicals.

Introduction

A significant problem in the preparation of coal for coal-fired power plants is the production of a polydisperse, colloidally contaminated aqueous suspension of coal and clay produced by coal washing. Due to the surface charge and the small size of the particles, a stable colloidal suspension is formed which will not undergo gravitational sedimentation at acceptable rates. The consolidation technique which is most often used for treating the effluent from coal washing and other contaminated streams is the addition of high molecular weight polyelectrolytes or other chemical additives. Grant et al. (*1*) used a surfactant (sodium dodecyl sulfate or cetyltrimethylammonium bromide) and found that the rate and extent of dewatering of iron oxide fines increased with increasing surfactant concentration. However, clarification via surfactant or polyelectrolyte addition can be expensive, and one must also be concerned about discharging the treated fluid to the environment after it has been chemically altered.

An alternative clarification technique, which has been known for several decades, and which is chemically noninvasive, and possibly less costly, is the application of an electric field to the colloidal suspension. When an electric field is applied to the suspension, the particles will move in the direction of the electric field lines. This phenomenon of particle movement relative to the surrounding stagnant fluid, due to an applied electric field, is known as *electrophoresis* and results from the fact that the colloidal particles become charged in water at the typical pH's encountered. In 1809 Reuss (*2*) observed this phenomena and the related phenomenon of *electroosmosis* in which the fluid moves through a stationary porous medium due to the applied electric field.

Since the 1930s electrokinetic phenomena have been applied to various processes ranging from the separation of proteins (*3*) to the stabilization of soft soils (*4*). In the 1960s Sprute and Kelsh (*5*) at the Bureau of Mines began to investigate the use of electrokinetics for the densification of mill tailings and coal-processing waste. Stanczyk and Feld (*6*), also at the Bureau of Mines, investigated electrokinetics for dewatering phosphate clays.

Since the Bureau of Mines investigations, several other researchers have tried to incorporate electrokinetics as a separation technique. Lockhart (*7*) was able to clarify clay suspensions and mine tailings, Wilmans and Van Deventer (*8*) treated kimberlite slimes from diamond mining, and Yoshida et al. (*9*) treated bentonite sludge. Sauer and Davis (*10*) were able to remove fine particles of coal and clay from the water of a coal-washing waste pond using a small laboratory apparatus. They determined that the electric field does not need to be applied throughout the entire course of the electrokinetic processing, thus lowering the costs associated with power consumption. Shang and Lo (*11*) made further improvements in reducing the power consumption of electrokinetic techniques in their investigation of the consolidation and dewatering of a phosphate clay suspension from a waste disposal pond. They showed that the efficiency of electrokinetic treatment was improved when the current was applied in cycles of 15 min ON and 5 min OFF, a 25% reduction in power consumption. Electrophoresis has also been applied to colloidal suspensions in nonaqueous media. Shih et al. (*12*) investigated the sedimentation of illite particles in toluene containing asphaltene, Lee et al. (*13*) studied the rate of sedimentation of α-alumina particles in xylene for various concentrations of a surfactant which controlled the surface charge on the particles, and Matsumoto et al. (*14*) removed oxidized aluminum and iron particles from kerosene.

Successful laboratory studies have not yet led to widespread commercial application. This can be partly attributed to the low rates of particle migration in an electric field and partly to a poor understanding of particle motion in concentrated suspensions when an electric field is applied. The latter issue makes it difficult to scale-up results from the laboratory to commercial sizes.

Numerous analyses of single particle motion in an electric field have been published. The first analysis of electrophoretic motion was that of Smoluchowski (*15*) for a single sphere with a low surface potential and a nonpolarizable thin electric double layer moving in an infinite surrounding fluid. Smoluchowski obtained the particle mobility, U_e, given by

$$U_e = \frac{v_e}{E} = \frac{\epsilon_0 \epsilon_r}{\mu}\zeta \qquad (1)$$

in which v_e is the velocity of the particle in the stagnant fluid due to the applied electric field E, ϵ_0 is the permittivity of free space, ϵ_r is the dielectric constant of the surrounding fluid, μ is the viscosity of the fluid, and ζ is the zeta potential. The zeta potential must be less than $|\pm 25|$ mV for eq 1 to be valid.

A measure of the thickness of the electric double layer is given by κ^{-1}, where κ is the Debye−Hückel parameter. The

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TABLE 1. Summary of Colloidal Suspension Characteristics

	suspension	
	coal-washing[a]	**sodium−bentonite**[b]
vol av diam (μm)	1.07	3.0
specific gravity of particles	1.2	2.5
solids content (wt %)	0.2	0.2−0.3
electrophoretic mobility (m^2/V−s)	-3.2×10^{-8}	$-(2.15 \text{ to } 2.28) \times 10^{-8}$
initial pH of suspension	8.2	9.0
suspension conductivity (mS/m)	385.7	71.4−153.1

[a] Coal-washing suspension from Centralia Mining Co., Centralia, WA. [b] Sodium−bentonite particles from Whittaker, Clark, and Daniels, Inc.

Debye−Hückel parameter is a function of the concentration of ionic species, C_i^∞, in the bulk solution, and the valence of the ions present, z_i; that is

$$\kappa = \left(\frac{F^2 \sum_i C_i^\infty z_i^2}{\epsilon_0 \epsilon_r RT}\right)^{1/2} \tag{2}$$

Here F is the Faraday constant, R is the gas constant, and T is the absolute temperature. A thin electric double layer corresponds to large κa ($\kappa a > 100$), where a is the particle radius.

In an attempt to relax the restrictions in the Smoluchowski theory, Kozak and Davis (*16*) obtained an analytical solution for the electrophoretic mobility of a concentrated suspension of particles with a moderately thin and polarizable electric double layer ($\kappa a > 50$) in a single $z-z$ electrolyte solution. The distortion of the electric double layer is referred to as the relaxation effect and has been shown to be small for large κa ($\kappa a \geq 100$ for $|\zeta| \geq 100$ mV) or for small zeta potentials ($|\zeta| \leq 50$ mV when $\kappa a \approx 10$) (*17, 18*). By including the relaxation effect, Kozak and Davis obtained the solution

$$U_e = \frac{\epsilon_0 \epsilon_r}{\mu}\zeta - \frac{2\epsilon_0 \epsilon_r KRT}{\mu z F(1+K)}\Lambda_{2,0} \tag{3}$$

in which $\Lambda_{2,0}$ is defined by

$$\Lambda_{2,0} = \ln\left[\frac{1 + \exp(F|z\zeta|/2RT)}{2}\right] \tag{4}$$

The function K depends on the solids volume fraction, ϕ, and the diffusivity, D_-, of the anionic species (for $\zeta > 0$) and is

$$K = \frac{2(1+2\phi)}{\kappa a(1-\phi)}\left(1 + \frac{4C_\infty RT}{\mu D_- \kappa^2}\right)\exp\left(\frac{F|z\zeta|}{2RT}\right) \tag{5}$$

For $\zeta < 0$, D_- should be replaced by D_+, the diffusion coefficient of the cationic species. The first term on the right side of eq 3 is Smoluchowski's result, and the second term accounts for particle−particle interactions, electric field retardation due to the relaxation effect, and other electric double-layer effects. Hydrodynamic interactions and electric double-layer effects reduce the mobility of the particles compared to the mobility determined with Smoluchowski's theory. Kozak and Davis (*19*) later extended their analysis to suspensions of colloidal particles with moderately thick electrical double layers ($\kappa a > 20$).

Despite the multiple factors that decrease the electrophoretic mobility, electrokinetic clarification techniques are still a viable alternative for colloidal solutions which can be treated over long periods of time, for example, the removal of colloidal contaminants from settling ponds and lakes. This paper focuses on a new electrophoretic apparatus which can process concentrated colloidal suspensions on either a batch or semicontinuous basis for application in which chemical alteration of the suspension is undesirable and where low clarification rates of the suspension are acceptable.

Colloid Properties

The colloidal suspensions studied were analyzed for electrical conductivity, particle size, and average electrophoretic mobility. The conductivities of the colloidal suspensions were determined by a Jenway 4320 conductivity meter. The volume average diameters of the particles were determined using a Horiba Model CAPA-500 centrifugal particle analyzer. By measuring the sedimentation rate by light transmission, the particle diameter can be determined from Stokes' centrifugal sedimentation equation. The electrophoretic mobility was determined using a Rank Brothers Mark II microelectrophoresis apparatus. The average electrophoretic mobility was taken to be the average of 20 measurements for a particular suspension. A summary of the characteristics of the suspensions is provided in Table 1.

The colloidal suspension of principal concern here is wastewater containing coal and clay colloids obtained from the coal-washing effluent at the Centralia Mining Co. located in Centralia, WA. This suspension contained particles with a volume average diameter of 1.07 μm and a solids content of 0.2 wt % of which approximately 64% was clay. The average electrophoretic mobility was found to be -3.2×10^{-8} m^2/(V·s). The suspensions introduced into the experimental apparatus exhibited no gravitational sedimentation and no flocculation in the absence of an electric field.

Since the colloidal matter was more clay than coal and since the effluent varied from day-to-day, separate experiments were performed using a well-characterized clay. The clay used as the model colloid suspension was montmorillonite (sodium−bentonite) obtained from Whittaker, Clark, and Daniels, Inc. No purification of the montmorillonite was performed. The montmorillonite came as a dry powder and was later suspended in reverse osmosis (RO) treated water as needed for the experiments. To obtain a fine dispersion of particles, the water was stirred with a magnetic stirrer while the clay was added slowly to prevent clumping. The suspension was then stirred for an additional 10 min to ensure a homogeneous suspension of particles. The volume average diameter of the particles was found to be 3.0 μm.

The experiments conducted with the montmorillonite varied in initial clay concentration and initial salt (NaCl) concentration in the aqueous solution. Two clay concentrations were investigated, 0.2 and 0.3 wt %, because these concentrations were similar to the coal-washing effluent studied. For these clay concentrations, the average electrophoretic mobility and conductivity were measured as a function of salt concentration (0, 25, 50, and 100 mg/L), and the results are presented in Figure 1a,b, respectively. For the 0.2 wt % suspension the pH varied from 9.1 to 8.5 for a NaCl concentration of 0 and 100 mg/L, respectively. For the 0.3 wt % suspension the pH varied from 9.4 to 8.8 for a NaCl concentration of 0 and 100 mg/L, respectively. The literature reports spontaneous flocculation for montmorillonite when the pH is less than 3.5 (*20*) and a critical coagulation

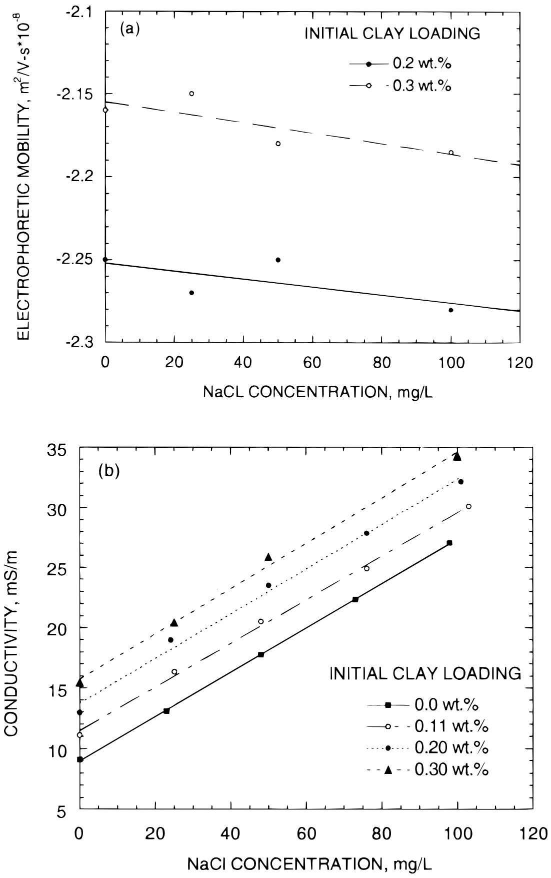

FIGURE 1. (a) Effects of NaCl concentration on the electrophoretic mobility of the sodium−bentonite colloid particles and (b) the effects of sodium−bentonite and NaCl concentrations on the conductivity of the colloidal suspension.

concentration (ccc) for a $|z| = 1$ electrolyte of 0.008 ± 0.006 mol/L (*21*). The pH values and the electrolyte concentrations used throughout these experiments never exceed these limits. Although the 100 mg/L (0.017 mol/L) concentration of NaCl is close to the lower limit of the reported ccc, no appreciable flocculation appeared to occur within 72 h in the absence of the applied electric field.

Experimental Apparatus

A benchtop electrophoretic apparatus was designed and constructed with industrial applications and scalability in mind. The apparatus, shown in Figure 2, consisted of a cubic acrylic tank with inner dimensions of 36.8 × 36.8 × 36.8 cm. The top electrode was a 0.64 cm thick carbon sheet with dimensions 36.3 × 33.3 cm. Because hydrogen gas is produced at this cathode by electrolysis of water, a number of 0.64 cm diameter holes were drilled through the carbon sheet to facilitate the escape of hydrogen gas. In the absence of these holes a layer of hydrogen formed at the lower surface, blanketing the electrode and preventing contact with the electrolyte solution. The cathode could be positioned at any height within the tank.

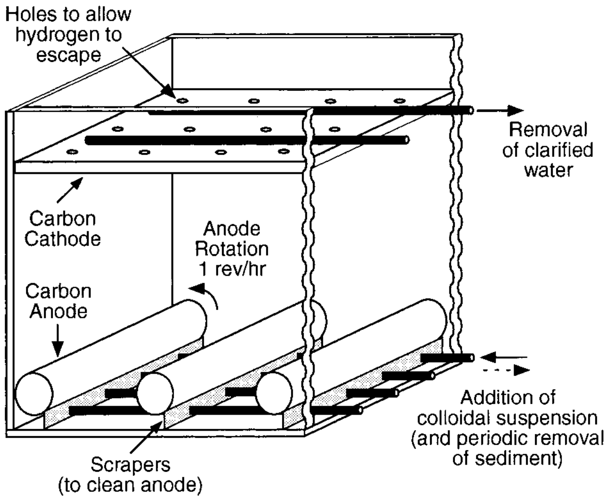

FIGURE 2. Experimental electrophoresis apparatus.

The anode was a series of nine 2.54 cm diameter solid carbon rods centered 3.8 cm above the bottom of the tank. The carbon electrodes were obtained from Graphite Sales, Inc. The carbon rods were designed to be rotated to clean the electrodes as the experiment proceeded. To do this, a shaft was extended from the end of each carbon rod through the tank wall. On each shaft a 24-pitch bronze worm gear was placed. The pitch diameter was 2.12 cm. A steel shaft with nine, appropriately placed, 24-pitch steel worms meshed with the bronze worm gears. The pitch diameter of these worms was 1.27 cm. The steel shaft was rotated by means of a 10−50 in.-lbs 12 V DC stepper motor. The power supply for the stepper motor was a Hewlett-Packard 6218C DC supply. As the carbon rods rotated they were cleaned by coming into contact with a flexible plastic scraper which touched the bottom of each carbon rod.

The power supply connected to the electrodes was a Hewlett-Packard 6555A DC supply. The connection to the nine carbon rods was made by contacting a lead from the positive terminal of the power supply to the steel shaft on which were mounted the nine steel worms. The charge was then transferred to the bronze worm gears and then through a wire running from the bronze gear through the shaft in the tank wall and finally to the carbon rods.

For the semicontinuous electrophoretic experiments a fresh colloidal suspension was added in the sump below the carbon rods while the clarified water was removed from the space above the carbon sheet. The addition and removal of solution was done by a Masterflex peristaltic pump with two pump heads, one pump head for the colloidal input tube and one pump head for the clarified solution output tube. To evenly distribute the addition of the colloid suspension, the input tube from the first pump head was split into four 0.64 cm i.d. Tygon tubes which were placed in the sump. Each of the four tubes had 1.0 mm holes drilled every 3.8 cm along the axis of the tube for the addition of the colloidal suspension. The clarified solution was extracted through two 0.64 cm i.d. Tygon tubes with 1.0 mm diameter holes drilled every 3.8 cm. These two output tubes were then connected to a single tube which led to the appropriate pump head.

As the particles settled due to the electric field, a clear zone formed above the sedimenting clay particles. To determine the final weight percent of particles remaining in the clear zone, an optical system based on turbidity was setup. The light source was a 20 mW HeNe laser, and the detector was a Newport Research Model 815 photosensor connected to a Newport Research Model 815 power meter. The laser and the photosensor were placed on opposite sides of the apparatus at a height which was the midpoint between the top and bottom electrodes. Upon calibration, the power meter readings could be directly correlated to the solids content of the clarified solution at this point. Although a concentration gradient of particles exists during the clarification process, it is assumed that the solids content at the midpoint adequately represents the solids content of the bulk clarified solution after long times.

Sedimentation Experiments

The electrophoresis apparatus was filled with the suspension to be studied. The electric field was then applied as soon as any observable convective motion ceased. The experiments were performed under constant current conditions (100 mA or 0.827 A/m^2) with the carbon cathode located 14 cm above the top of the carbon rods. With this electrode configuration, the test volume was 24 L. The maximum current density of 0.827 A/m^2 was chosen because at higher current densities the generation of oxygen at the anode due to electrolysis was excessive and caused convective mixing of the colloidal suspension as bubbles rose through the suspension. The suspensions to be studied contained either the coal-washing wastewater or the model montmorillonite colloidal suspension with various particle and/or salt concentrations.

Effects of Anode Surface Cleaning. To ensure that the rotating anode rods did not hinder the rate of clarification, two experiments were performed with a 0.2 wt % montmorillonite suspension in RO water. In one experiment, the carbon rods were rotated at a slow rate (1 revolution/h) to minimize convective motion within the apparatus. In the other experiment, the carbon rods remained stationary. There appeared to be a slight advantage in the rate of clarification when the rods were rotated.

Effects of Electrolyte Concentration. The presence of electrolytes in the aqueous phase has an effect on the electrophoretic mobilities of clays. First, the measured electrophoretic mobility of montmorillonite in the presence of no salt is in quantitative agreement with Swartzen-Allen and Matijevic (*20*). Next, the magnitude of the electrophoretic mobility tends to decrease for an increase in the concentration of electrolyte and corresponding decrease in pH, as shown in Figure 1a. This is in agreement with past research on a variety of clay particles (*20*–*22*).

To investigate the effects of electrolyte concentration on the power requirements and time necessary to clarify the solution, experiments were conducted with varying amounts of sodium chloride in a 0.2 wt % montmorillonite suspension. The effects of increasing salt concentration had the expected result of lowering the instantaneous power requirements during the experiment, as shown in Figure 3a.

It should be pointed out that the electrolyte solutions corresponding to no NaCl addition contained ions originating from the colloidal particles as well as ions formed by dissolution of CO_2 from the ambient air. No attempt was made to eliminate CO_2 or control it. The data shown in Figure 3a for lower ion concentrations show a relative maximum in the instantaneous power consumption at approximately 10 h. This time-dependent power consumption results from the fact that a clear zone forms above the suspension as sedimentation proceeds. The conductivity of the expanding clear zone is less than in the suspension, and as the particle concentration increases, the conductivity increases. The result is that the instantaneous power consumption initially decreases, then goes through a relative maximum, then sharply decreases, and finally slowly increases to a steady-state value.

Figure 3b, which presents information on the solids removal as a function of time, indicates that as the salt level increased, the clarification rate decreased. This is in agreement with the result of a decrease in electrophoretic mobility with a increase in salt concentration. The decrease in mobility is due to the decreased thickness of the electric double layer, as predicted by eq 2, which results in decreasing the repulsive force of the double layer between particles. This leads to an increase in particle–particle interactions and a reduced clarification rate. The increase in the rate of clarification for the experiment with 100 mg/L NaCl compared to the experiment with 50 mg/L NaCl is probably due to the ability of the clay particles to overcome the repulsive forces of the compressed electric double layer, due to electrophoretic motion, leading to particle–particle aggregation and an increased rate of clarification due to gravitational sedimentation.

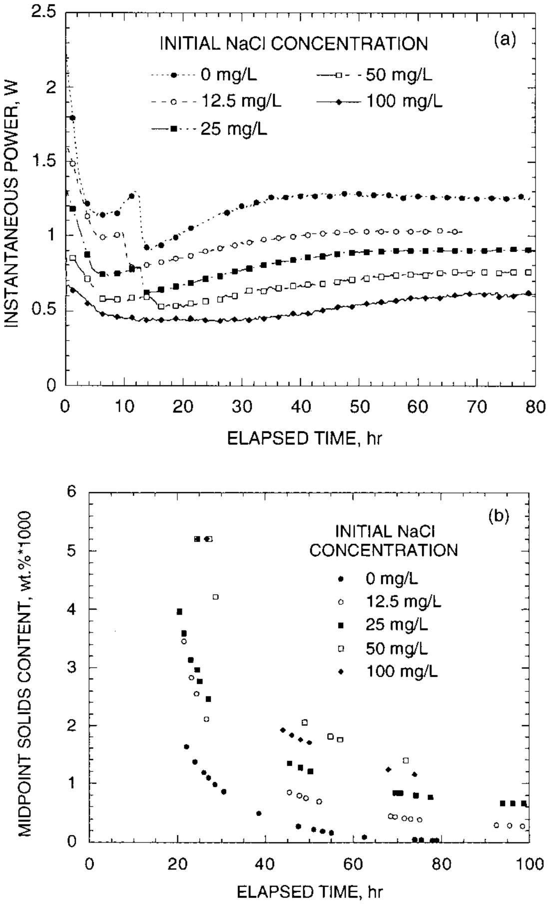

FIGURE 3. Effects of NaCl concentration on (a) the instantaneous power requirements and (b) the rate of solution clarification as measured by the remaining solids content in the clarified solution for batch operation.

Effects of Particle Concentration. An increase in the number density of particles in the suspension should cause a decrease in the electrophoretic mobility due to particle–particle hydrodynamic interactions (*16*). This is demonstrated in Figure 1a, which shows an average reduction in electrophoretic mobility of 2.2% for an increase in particle concentration from 0.2 to 0.3 wt %. The magnitude of this reduction in electrophoretic mobility is not predicted by eq 3, which only predicts a reduction of 0.1% for a given ζ potential. Thus, it appears that the reduction in the electrophoretic mobility is due to the increased electrolyte content due to the additional particles. The increase in the electrolyte content is shown by the increase in suspension conductivity with increasing particle concentration in the absence of added salt (Figure 1b).

To investigate the effects of particle concentration on the power requirements and the time necessary to clarify the solution, experiments were conducted with varying amounts of montmorillonite particles in a solution having an NaCl concentration of 12.5 mg/L. Figure 4a shows the increase in time necessary for clarification of the suspension when the initial number density of particles is increased. The corresponding change in instantaneous power required is presented in Figure 4b. The increase in initial conductivity of the suspension with increased solids content lowers the instantaneous power levels for constant current operation.

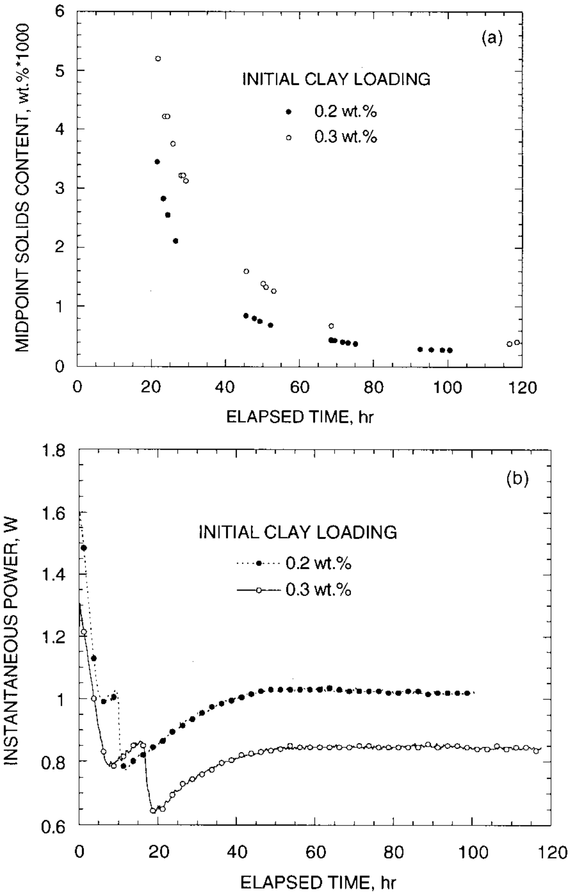

FIGURE 4. Effects of sodium−bentonite concentration on (a) the rate of solution clarification and (b) the instantaneous power requirements for batch operation.

Semicontinuous Experiments. The experiments performed with a batch operation demonstrate the feasibility of clarifying colloidal suspensions electrokinetically, but continuous or semicontinuous operation is preferred for large-scale processing. To demonstrate semicontinuous operation, experiments were performed with a particle concentration of 0.2 wt % and no NaCl added to the RO water.

Initially the experiment was conducted as if it were a batch process. That is, the apparatus was filled with 24 L of the 0.2 wt % colloidal suspension and the electric field was applied. After 48 h the particles had moved below the surface of the anode rods. At this point, more of the 0.2 wt % colloidal suspension was injected beneath the anode rods at a rate of 50 ± 5 mL/min. This method of colloidal addition was thought to be advantageous for two reasons. First, the negative particles would be entering near the anode, and second, the entering particles will have an increased probability of colliding with particles already present in this area. The increased probability of particle collisions increases the chances of particle−particle aggregation which leads to gravitational sedimentation.

Due to the inflow of the added colloidal suspension, the height of the sediment layer rose. Because of this, the addition of the colloidal suspension was periodically stopped every 2 h for a 2 h duration to allow electrophoresis to recompact the sediment. Once the sediment layer dropped to a level near the top of the anode rods, the addition of the colloidal suspension was recommenced. After 13.5 h, 18 L of a 0.2 wt % colloidal suspension had been added to the apparatus and, therefore, 18 L of clarified water with an average solid content of 2.6×10^{-4} wt % had been removed. At this time the sediment layer had increased in height to 8.8 cm above the bottom of the tank compared to 2.7 cm at the onset of the semicontinuous operation. By continuing to apply the electric field for an additional 10.5 h, the sediment height reduced to 5.7 cm, resulting in a solid content of 0.757 wt %, an increase of 279% from the original 0.2 wt % suspension. The instantaneous power essentially remained constant at 1.3 W throughout the semicontinuous operation.

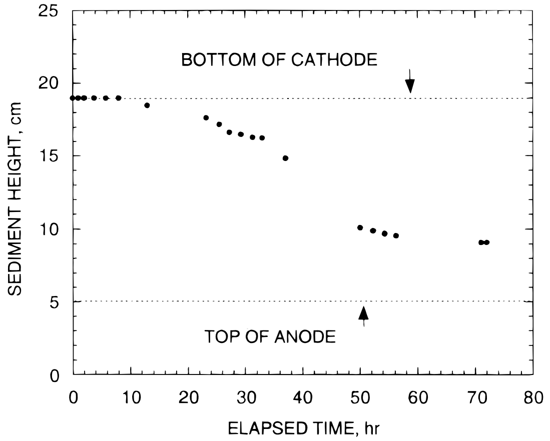

FIGURE 5. Sediment height as a function of time for the processing of the coal-washing wastewater.

It is important to note that eventually the region near the anode will become too concentrated in sedimented particles to achieve effective separation. At this point, the pump can be reversed in order to withdraw the sediment through the inlet tubes.

The benefits in developing a semicontinuous electrokinetic clarification system can be observed from a cost comparison. The operating costs associated with electrokinetic clarification for a system with a flow rate of 25 mL/min, an average power consumption of 1.3 W, and a cost of 12¢/kW·h is approximately 0.010¢/L. Of course, there are additional and unknown costs associated with maintaining the electrokinetic process. Compare this to a typical industrial clarification technique at a coal-washing site which spends about $1.5 M/year on polyelectrolytes to clarify a suspension with an average flow rate of 2270 L/min to give a cost per liter of 0.126¢/L. The primary disadvantage of electrophoretic processing is the rate of clarification, but for cleaning sediment ponds and lakes this is not a serious problem provided the power requirements are low.

Coal-Washing Suspension. To demonstrate that the coal-washing wastewater could be electrophoretically clarified, it was added to the apparatus and clarified in a batch operation. Figure 5 shows the sediment height vs time which resulted in a clarified solution containing 0.018 wt % remaining solids compared to the initial particle concentration of 0.2 wt %. The power level throughout the experiment remained essentially constant at 0.45 W. In this case a sharply defined clear zone did not form, for the suspension remained diffuse as clarification proceeded. As shown in the clay experiments, for higher salt concentrations, the remaining solids content in the clarified solution increases and the power level decreases for higher initial electrolyte concentrations. Therefore, the higher solids content in the clarified solution and the lower power requirement for the coal-washing effluent experiment, compared to the clay experiments, is probably due to the high salt concentration (350 mg/L NaCl) in the wastewater.

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