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Beneficial Reuse of Fly Ashes in Geotechnical Engineering with Physicochemical and Electron Microscopic Methods

by

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NUTC R286

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Technical Report Documentation Page

1. Report No. NUTC R286	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle Beneficial Reuse of Fly Ashes in Geotechnical Engineering with Physicochemical and Electron Microscopic Methods		5. Report Date June 2013
		6. Performing Organization Code
7. Author/s Bate Bate		8. Performing Organization Report No. Project #00036853
9. Performing Organization Name and Address Center for Transportation Infrastructure and Safety/NUTC program Missouri University of Science and Technology 220 Engineering Research Lab Rolla, MO 65409		10. Work Unit No. (TRAIS)
		11. Contract or Grant No. DTRT06-G-0014
12. Sponsoring Organization Name and Address U.S. Department of Transportation Research and Innovative Technology Administration 1200 New Jersey Avenue, SE Washington, DC 20590		13. Type of Report and Period Covered Final
		14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract

The sedimentation behavior of fine grained soil is largely dependent on its pore fluid chemistry. Physicochemical properties of the pore fluid, such as ionic strength and pH, could greatly influence the micro structure of kaolinite which in turn influences the sedimentation behavior. Other than ionic effect, adding fly ash can also cause different sedimentation behaviors due to the change of gradation, increased ionic strength and pozzolanic reactions. There are several tests to characterize the kaolinite micro fabric, such as sedimentation test, grain size distribution test, viscosity test, zeta potential test, and scanning electron microscopy (SEM). The objective of this project was to investigate the influence of ionic concentration and fly ash on the sedimentation behavior of kaolinite. In addition, zeta potential and particle size distribution in supernatant and suspensions were measured and analyzed. The zeta potential of kaolinite was found closely related to the particle size, micro fabric and settling speed. It was found that an increase in the percentage of fly ash in the fly ash soil mixture could cause an increase in the settling speed. The addition of fly ash was found more efficiency than the ionic concentration because the fly ash can not only interact with kaolinite particles but also increase the ionic strength in the dissolution so that the kaolinite could flocculate and aggregate which in turn increases the settling speed.

17. Key Words Kaolinite; Sedimentation; SEM; Micro Fabric; Zeta Potential	18. Distribution Statement No restrictions. This document is available to the public through the National Technical Information Service, Springfield, Virginia 22161.		
19. Security Classification (of this report) unclassified	20. Security Classification (of this page) unclassified	21. No. Of Pages 17	22. Price

Form DOT F 1700.7 (8-72)

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Abstract

The sedimentation behavior of fine grained soil is largely dependent on its pore fluid chemistry. Physicochemical properties of the pore fluid, such as ionic strength and pH, could greatly influence the micro structure of kaolinite which in turn influences the sedimentation behavior. Other than ionic effect, adding fly ash can also cause different sedimentation behaviors due to the change of gradation, increased ionic strength and pozzolanic reactions. There are several tests to characterize the kaolinite micro fabric, such as sedimentation test, grain size distribution test, viscosity test, zeta potential test, and scanning electron microscopy (SEM). The objective of this project was to investigate the influence of ionic concentration and fly ash on the sedimentation behavior of kaolinite. In addition, zeta potential and particle size distribution in supernatant and suspensions were measured and analyzed. The zeta potential of kaolinite was found closely related to the particle size, micro fabric and settling speed. It was found that an increase in the percentage of fly ash in the fly ash soil mixture could cause an increase in the settling speed. The addition of fly ash was found more efficiency than the ionic concentration because the fly ash can not only interact with kaolinite particles but also increase the ionic strength in the dissolution so that the kaolinite could flocculate and aggregate which in turn increases the settling speed.

Keywords: Kaolinite; Sedimentation; SEM; Micro Fabric; Zeta Potential;

INTRODUCTION

Sediments, deposited in rivers, valleys, and highways, eventually lead to the formation of soil. Initially, sedimentation occurs when particles settle out from the suspension. As the sediments accumulate, the particles and flocs are brought together and formed soil deposits due to consolidation (Pane & Schiffman 1985). At this stage the material could be defined as a soil, with properties described by traditional parameters (Been & Sills 1981). Studying the process of sedimentation can help engineers understand the stress strain characteristics and hydraulic conductivity of a soil deposit. Understanding the sedimentation behavior of fine grained soil is also very important in many other engineering practices (Imai 1980; Klein 1999; Sridharan and Prakash 2001), such as, developing fly ash grouting (Markou 2002), designing mine tailing dams (Wardwell and Muzzy 1988), purifying containment water, analyzing the drilling mud filtration on borehole stability, studying the interaction between waste leachate and clay liners in landfill (Fam and Dusseault 1998), and determining the geotechnical properties of alluvial soils.

Due to isomorphous substitution, clay particles are usually negatively charged in aqueous environment. A diffuse double layer consisting of abundant positively charged ions neutralizes the negative charges of the clay particles. In a clay-electrolyte system, there are several types of attraction and repulsion forces among clay particles. Double layer repulsion is a force that develops between clay particles. When two particles approach each other, the magnitude of the repulsion force will rely on the interparticle distance and the thickness of the diffuse double layer (Santamarina et al., 2001). Electrostatic attraction is another force that develops between positively charged edges and negatively charged faces of particles. This type of force supports the edge-to-face flocculation fabric of kaolinite system (Van Olphen 1977). Van der Waals force is also an important attractive force in clay colloidal system (Israelachvili 2011). The grain size distribution, solid water concentration, clay mineralogy, and water chemistry were found to greatly influence the sedimentation behavior of fine grained soils (Sridharan and Prakash 1999). The fabric/structure of the slurry and the final sedimentation are largely dependent on these factors. Klein (1999) reported that as ionic concentration increased, the kaolinite fabric varied. In the case of a mixture of kaolinite and a fluid of low ionic concentration, edge-to-face flocculation is favored due to electrostatic attraction (Schofield and Samson 1954). A slight increase in ionic concentration decreases the thickness of the diffuse double layer, and both the edge-to-face attractive force and face-to-face repulsive force decrease. The resulting structure is edge-to-edge flocculated (Van Olphen 1951). At NaCl concentrations greater than 0.1-0.15mol/l, Van der Waals attraction dominates, the face-to-face aggregation occurs (Palomino and Santamarina 2005). These aggregates link through edge-to-edge and edge-to-face interactions that resulting in a high void ratio net work of kaolinite particles (van Olphen 1977; O'Brien 1971; Rand and Melton 1977). Therefore, a change in ionic strength could lead to a great change in the soil fabric. As a result, the sedimentation behavior will vary significantly. In general, if attractive force is large enough, the particles will collide and combine together which result in a rapid and homogeneous sedimentation process. On the other hand, when the repulsive force is large, each particle will settle at its own speed thus leading to a slow and heterogeneous sedimentation.

Fabric of kaolinite suspension, the sedimentation behavior, influencing factors, and clay mineralogy have been studied extensively by previous researchers (Klein 1999; Sridharan and Prakash 1999; Santamarina et al., 2001; Palomino and Santamarina 2005). The sedimentation behaviors of larger particles such as fly ash and fly ash-clay mixtures, however, were not commonly found in the literature. The fly ash particle size is about 1 to 100 micro-scale, which is 10 to 100 times larger than kaolinite particles. Thus adding fly ash to kaolinite colloidal suspension will very likely lead to different sedimentation volumes and settling speed. This report evaluates the sedimentation behaviors of kaolinite suspensions at different salt concentrations and fly ash-kaolinite mixtures at various ash-to-clay weight ratios. The effects of fly ash on the sedimentation behavior were then quantified by comparing with that of the ionic concentration of the added electrolytes. The preliminary test results may provide some engineering guidance in the application of fly ash on soil modification, ground improvement, and weak soil stabilization.

The microscopic fabric of kaolinite has been studied extensively in literature (O'Brien 1971; Van Olphen 1977; Rand and Melton 1977; Stawinski et al. 1990; Pierre et al. 1995; Ma and Pierre1999; Wang and Sui 2006, Du et al. 2006; Kim and Palomino 2009; Du et al. 2010). The formation of the micro fabric is influenced by the double layer repulsion and van der Waals attraction between the negatively charged face sites of kaolinite clay particles. The change of the net surface charge would result in the kaolinite suspension to have different fabrics, dispersion, flocculation, and aggregation. The flocculated and dispersed suspensions thus exhibited different sedimentation behaviors due to their inherent distinct fabrics. Typically, three micro fabric modes between kaolinite particles were reported: edge to edge (EE), edge to face (EF), and face to face (FF). The pH, ionic strength, ionic charge, polymer type, polymer strength, polymer charge, and polymer adsorption were all found to be the critical influencing factors on the formation of the clay fabrics.

The increasing of the electrolyte concentration (for example, Na^{+}, Ca^{2+}, and Fe^{3+}) was found to promote a higher degree of flocculation, because the double layers are compressed and the Van der Waals attractions become dominant, which leads to a formation of face to face type fabric from scanning electron microscopy (SEM) results (Wang and Sui 2006, Pierre et al. 1995; Ma and Pierre1999; Stawinski et al. 1990; Faas and Crocket 1983). Klein (1999) summarized that as ionic concentration increased, the kaolinite fabric varied accordingly. At low NaCl ionic concentration, edge to face (EF) flocculation is favored due to electrostatic attraction (Schofield and Samson 1954). A slight increase in ionic concentration will result in an edge to edge (EE) flocculation fabric (Van Olphen 1977). At NaCl concentrations greater than 0.1 - 0.15mol/L, Van der Waals attraction dominates, the face to face (FF) aggregation occurs (Palomino and Santamarina 2005). Other than inorganic ions, organic ions, such as polymers, can also be adsorbed to the clay surface and interacted with kaolinite particles. SEM observations have been used successfully in studies of the structure of soils (Chen et al. 1980; Bisdom 1981; Tarchitzky et al. 1984). The aim of this project was to apply the SEM technique to the understanding of the microstructure of the kaolinite suspension. A literature review of the past SEM data was presented in Figure 1. Microscopic fabric of kaolinite was clearly imaged. For example, FF aggregation fabric was shown in Figure 1d at 1.0mol/L salt concentration. SEM studies were also carried out on clay-polymer system (Du et al. 2006; Kim and Palomino 2009; Du et al. 2010). It was reported that the increasing of nonionic polymer molecular mass and concentration could result in an increase in the FF aggregation in kaolinite polymer system.

The sedimentation behavior, fabric of the suspension, influencing factors, and clay mineralogy have been studied extensively by previous researchers. However, studies about the sedimentation behavior of fly ash clay mixtures were not commonly found in the literature. This project attempts to evaluate the sedimentation behavior of kaolinite slurry at different ionic strength and kaolinite fly ash mixtures at various ash to clay weight ratios. The effects of ionic concentration and the fly ash to kaolinite weight ratio on the sedimentation behavior were studied and compared. Grain size distribution test, zeta potential test, SEM technique, and sedimentation tests were used to characterize the micro fabric of kaolinite and understand the fundamental interactions behind sedimentation.

Materials and Methodology

Materials

Georgia kaolinite (RP-2, Active Minerals International) was used in this project. Kaolinite was Na^+ homoionized using procedure similar to the one described in Palomino and Santamarina (2005). The fly ash used in this project was shipped from Lafarge power plants, Wisconsin. The fly ash particles are in round shape (Figure 2).

Kaolinite samples were immersed in NaCl and hexadecyltrimethylammonium bromide (HDTMA Br) solutions with solid content of 10 g/100 mL. NaCl solutions ranged from 0.0001mol/L to 1.0 mol/L and HDTMA solutions ranged from 0.0000125mol/L to 0.01mol/L. In addition, fly ash was added to kaolinite slurry at weight ratios of 1%, 5%, 10%, 20%, 30%, and 50%.

Sedimentation, SEM, zeta potential, and grain size distribution tests were performed on kaolinite slurries and fly ash-kaolinite mixtures.

Sedimentation Test

The sedimentation process of kaolinite slurry was studied in 100 mL graduated cylinder. The interfaces between supernatant and suspension, and between suspension and sediment (Figure3) were recorded as a function of time (Pane and Schiffman 1997). The test procedures, modified from that of Fam and Dusseault (1998), were used in this project. Detailed test procedures are presented in Kang et al. (accepted).

Zeta Potential Test

Zeta potential of the kaolinite particles was measured by Malvern Zetasizer Nano ZS90 zeta potential analyzer. Malvern Zetasizer was calibrated prior to measurement by using the standard calibrating solution. The micro electrophoresis cell was rinsed with deionized water 3 times to prevent cross-contamination between samples before each measurement. The test temperature was set at 23.5 ^{o}C during the experiments.

Grain Size Distribution Test

The grain size distribution of kaolinite particles in the supernatant and suspension were measured by dynamic light scattering (DLS) technique, using the Malvern Zetasizer Nano ZS90 instrument.

Samples in supernatant and suspension of the sedimentation cylinder were extracted by pipette and transferred into the cuvette. Each sample was preconditioned to 25 ^{o}C before testing.

RESULTS

SEM study was carried out on the kaolinite and fly ash particles, the results are presented in Figure 2. The diameter ranges from a few microns to a hundred microns. Small fly ash particles usually attached to large particles, which makes the contact area vary significantly. However, kaolinite particles are generally stacked and in platy-shaped. They are randomly scattered, aggregated, and coagulated.

Sedimentation test results of kaolinite in NaCl solutions at different concentrations are presented in Figure 3. At low concentration (0.00002 to 0.003 mol/L), kaolinite suspensions settled in a dispersed form, which was very slowly, and the suspension volume was very large. The final sediment volume (the bottom solid portion) increased as the time increased. As the ionic concentration increased (> 0.003 mol/L), however, the sedimentation behavior changed significantly. The suspension settled in a flocculated form. The supernatant sediment interface appeared soon after test started and the sediment volume started decreasing as time increased. This change was attributed to the flocculated and aggregated fabric that formed under high salt concentrations (Figure 1). The flocculation and aggregation phenomenon observed in this project agrees well with the results by other studies (Klein 1999, Sridharan and Prakash 1999, 2001; Blewett et al 2001). Figure 4 showed the sedimentation behavior of kaolinite in HDTMA solutions. Similarly as those observed in NaCl solution, the sedimentation behavior could be categorized into two groups, the flocculated sedimentation (above 0.001mol/L), and dispersed sedimentation (below 0.001mol/L), where the threshold concentration of 0.001mol/L is the critical micelle concentration (CMC) of HDTMA.

Sedimentation behaviors of fly ash-kaolinite mixture were presented Figure 5. A very clear trend was observed that as the fly ash weight ratios increased, the settling rate of kaolinite fly ash mixture became faster. The supernatant sediment interface appeared very early and the sedimentation speed increased as the fly ash content increased (Figure 5). The increase of sedimentation speed was more drastic when fly ash was larger than 5%, where the sedimentation ceasing time decreased to approximately 1000 s. at 1% FA content, the mixture settled in a dispersed form, and the sediment volume increased gradually. The conductivity of fly ash colloidal system at different fly ash contents was measured. The measured conductivity was then correlated with the concentration of NaCl solution. The relationship between the fly ash concentration and the corresponding NaCl concentration was plotted in Figure 6. It is found that when fly ash content at about 5.0g/l, the conductivity was about 0.009mol/l. This value was larger than the EF flocculation threshold of NaCl concentration (~ 0.002 - 0.003mol/L). Therefore the mixture settled in a flocculated form. Below 5.0g/l, the conductivity of the mixture was less than 0.003mol/l, and hence the mixture settled in a dispersed form.

Zeta potential, which is also known as electrokinetic potential, is the electrical potential at the shear plane. Changes in pH, ionic strength, type of ions in bulk fluid, and temperature will lead to the change of the net surface charge (Hunter 1981), which can be measured by the corresponding changes in zeta potential. Therefore, zeta potential is a good indicator of the net surface charge of fine grain particles. The zeta potential was measured at the end of each sedimentation tests. Figure 7 shows the change of the zeta potential of kaolinite in NaCl and HDTMA solution as a function of the concentration. The zeta potential varied with the variation of salt concentration. As the concentration of NaCl/HDTMA increased, the zeta potential of Kaolinite decreased (became less negative). The adsorption of Na^+ and HDTMA monomers on kaolinite particles would compress the DDL layer, thus resulting a lower zeta potential. Similarly, adding fly ash could also compress the DDL, and cause the zeta potential to decrease. Figure 8 shows the zeta potential of kaolinite as a function of the fly ash weight ratio. As the fly ash content increased, the zeta potential lowered gradually. The zeta potential indicates the repulsive force among kaolinite particles. the lower the zeta potential, the smaller the repulsion force, which means that kaolinite particles tend to attract and flocculate with each other, thus the colloidal system is not stable, and settles fast. On the other hand, if the zeta potential is high, which means the repulsive force among particles is large, so the colloidal system is stable and the particles settle slow or even do not settle. Similarly, the addition of fly ash could break the stability of the kaolinite colloidal system and thus cause the particles to settle fast.

Sedimentation behavior of kaolinite is closely related to its grain size distribution. The grain size of kaolinite flocs in a colloidal system depends on the thickness of the DDL, thus, the ionic concentration in the system could largely influence the grain size distribution of the kaolinite. Figure 9 shows the measured grain size distribution of kaolinite in 0.003mol/L NaCl solution. Particles in supernatant have smaller size and particles in suspension have slightly larger size (Figure 9). The average size in supernatant and suspension were 146.9nm and 184.3 nm, respectively. Figures 10 to 11 show the grain size distribution in NaCl and HDTMA solutions. As the salt concentration increased, the measured particle size decreased gradually. This could be understood by the changing of the zeta potential. At high salt concentration, the Van der Waals attraction force is dominant. Particles flocculated with each other and thus settled faster, only small particles left in the supernatant. However, at lower salt concentration, DDL repulsion force is dominant. The DDL repulsion force could prohibit the particles settling, thus the measured particle size was larger.

SUMMARY

The sedimentation behavior, micro fabric, zeta potential and grain size distribution of kaolinite and fly ash mixture in different salt solutions were investigated in this project. The ionic effects and grain size distribution on the sedimentation behavior was studied and correlated with the zeta potential of kaolinite suspension. The high quality SEM images of the micro fabric obtained in this project agreed well with the literature results, which supported that the SEM technique is a good tool to directly verify the micro fabric of kaolinite. The influence of ionic concentration and fly ash on the sedimentation behavior of kaolinite was studied. It was found that an increase in the percentage of fly ash in the mixture could cause an increase in the settling speed. The addition of fly ash was found more efficiency than the ionic strength because the fly ash could not only interact with kaolinite particles but also increase the ionic strength so that the kaolinite could flocculate and aggregate which in turn increases the settling speed. In addition, zeta potential and particle size distribution in the supernatant and suspensions were measured and analyzed. Zeta potential was found closely related to the particle size, micro fabric of kaolinite and sedimentation behavior.

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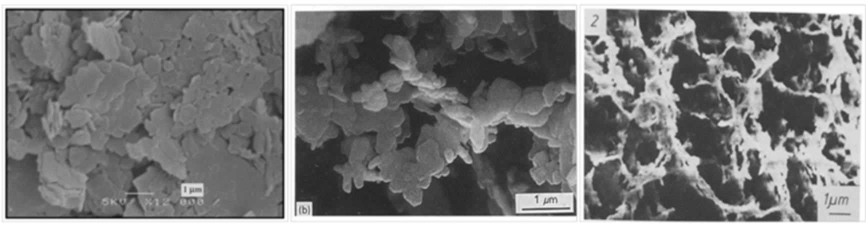
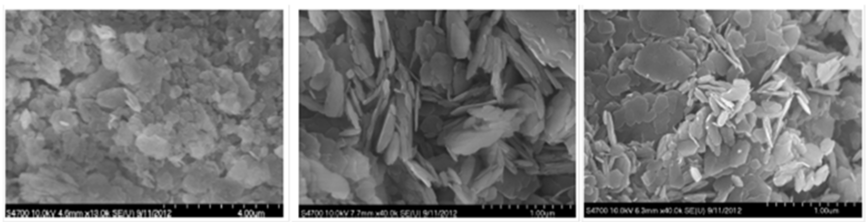
Fig. 1.
SEM Images of Kaolinite at Different NaCl Concentrations



(a) (b) (c)

Wang and Siu (2006) FF fabric (a), Pierre et al. (1995) EE fabric (b), and Stawinski et al. (1990) EF fabric (c)



(d) (e) (f)

1.0mol/L NaCl, FF fabric (d), 0.02mol/L EE fabric (e), and 0.003mol/L EF fabric (f)

Fig. 2.
SEM Image of Fly Ash (left) and Kaolinite Particles (right)

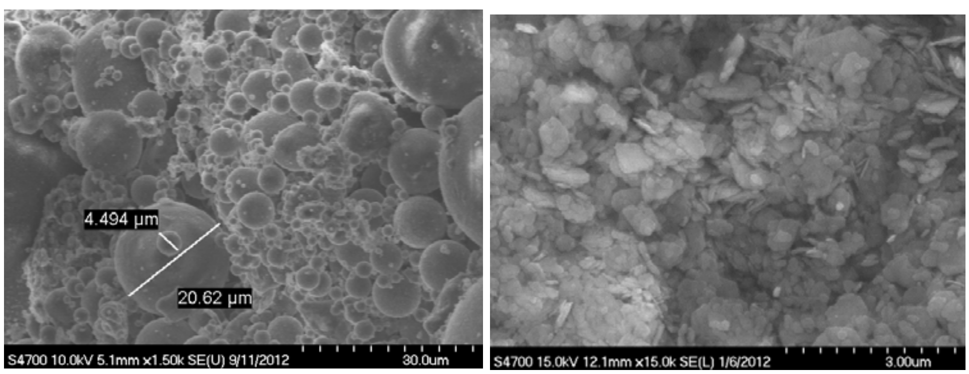


Fig. 3.
Sedimentation Behavior of Kaolinite Suspension at Different Ionic Concentrations

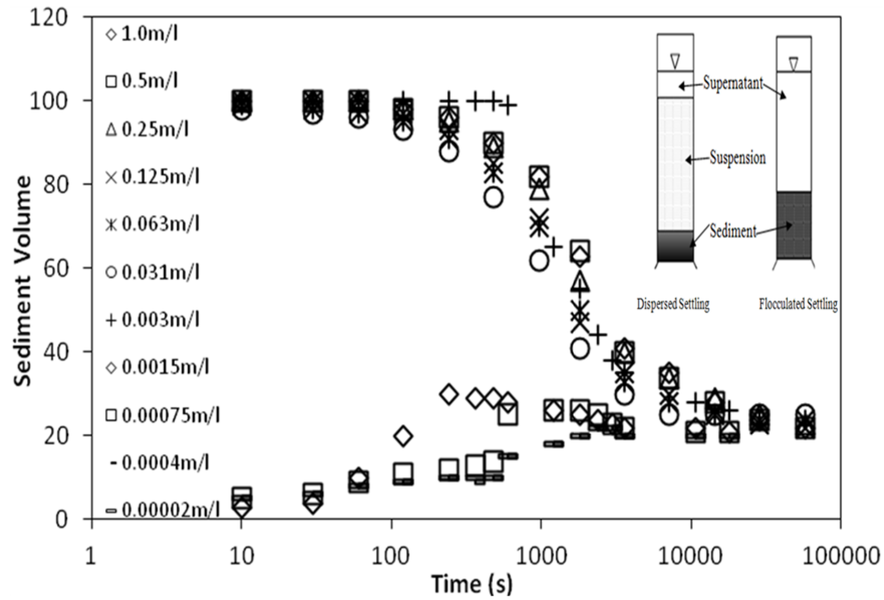


Fig. 4.
Kaolinite Sedimentation Behaviors at Different HDTMA Concentrations

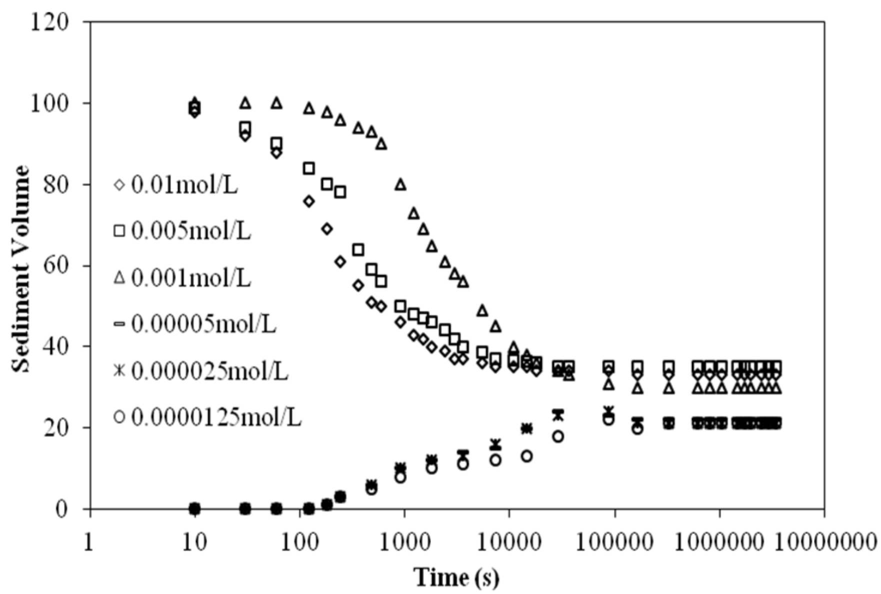


Fig. 5.
Kaolinite-fly Ash Mixture Sedimentation Behaviors at Different Ash to Kaolinite Weight

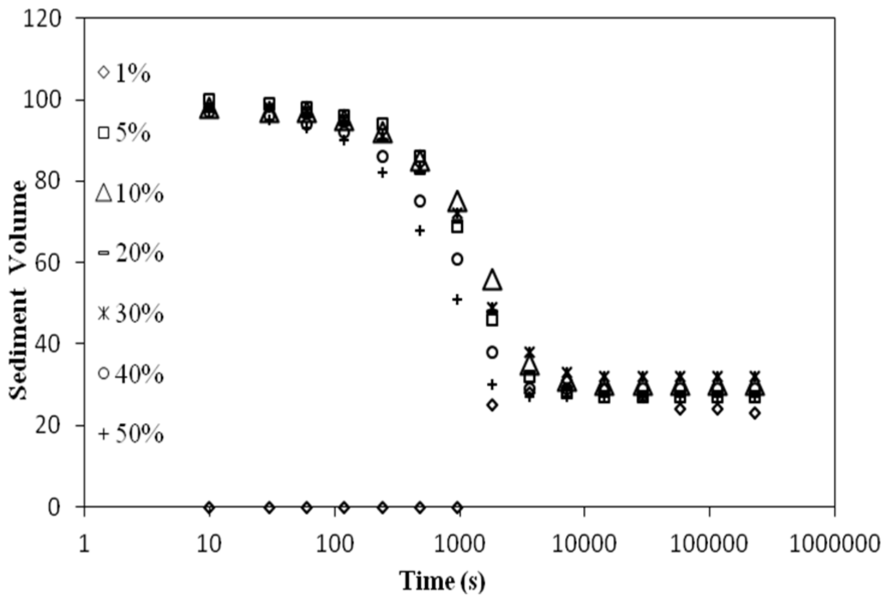


Fig. 6.
The Equivalent NaCl Concentration versus Fly Ash Content

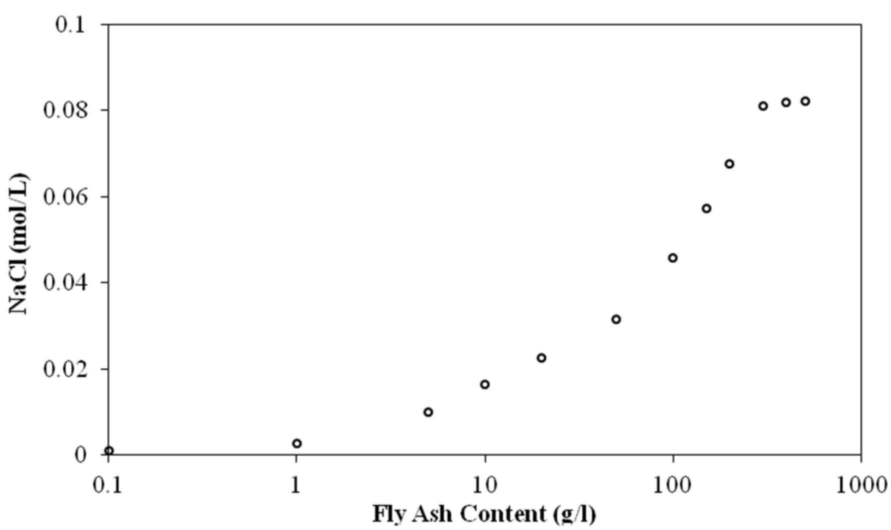


Fig. 7.
Zeta Potential versus NaCl /HDTMA Concentration of Kaolinite (at neutral pH)

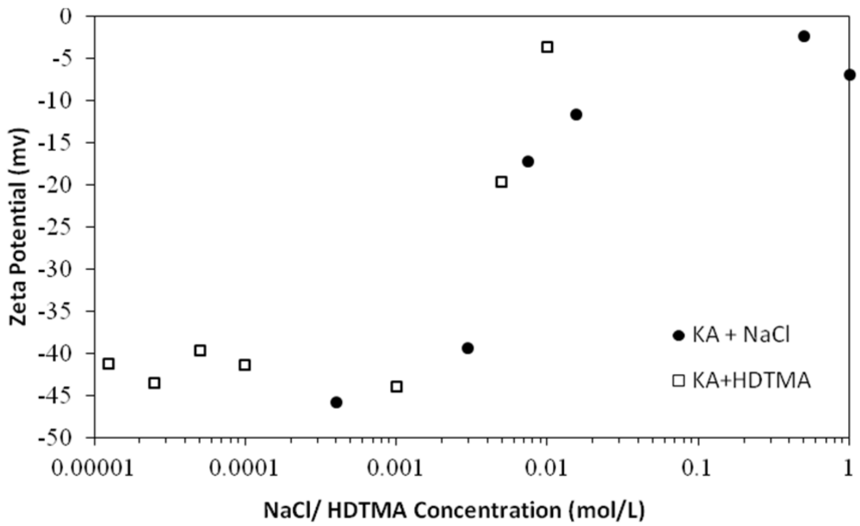


Fig. 8.
Zeta Potential versus Fly Ash Content

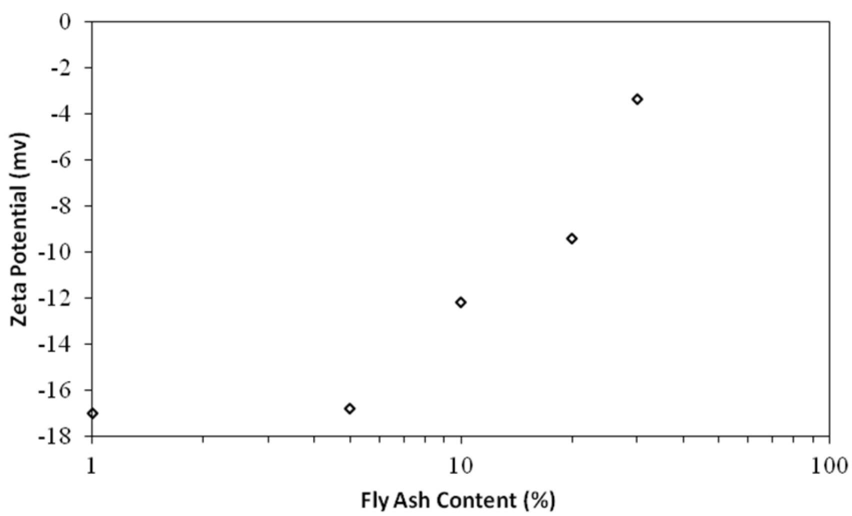


Fig. 9.
Grain Size Distributions in 0.003M NaCl Solution

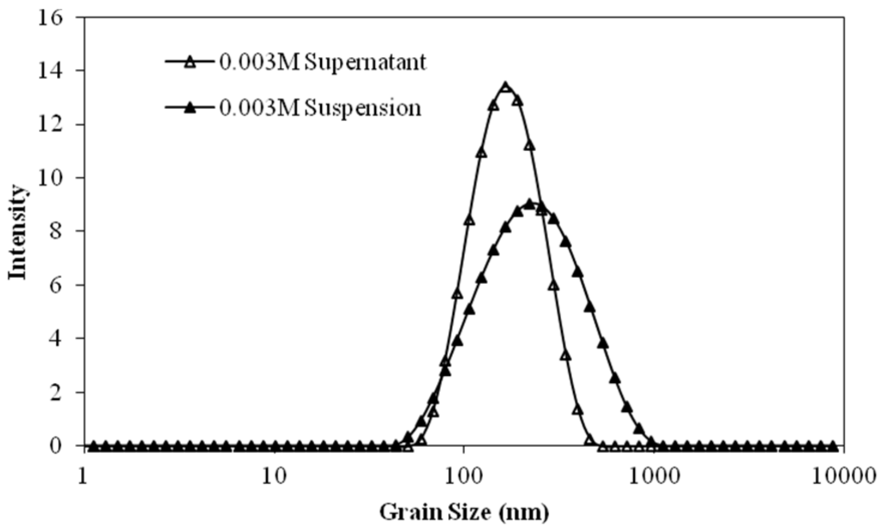


Fig. 10.
Grain Size Distribution of Kaolinite Supernatant at different NaCl Concentrations

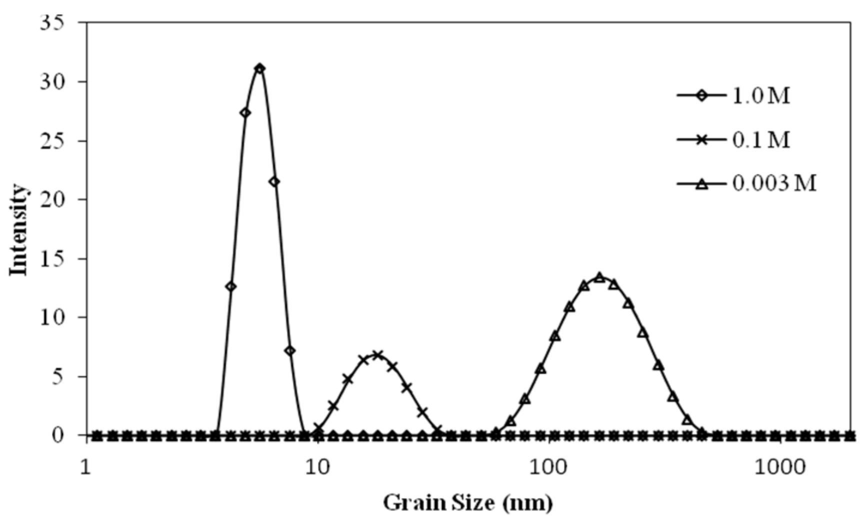
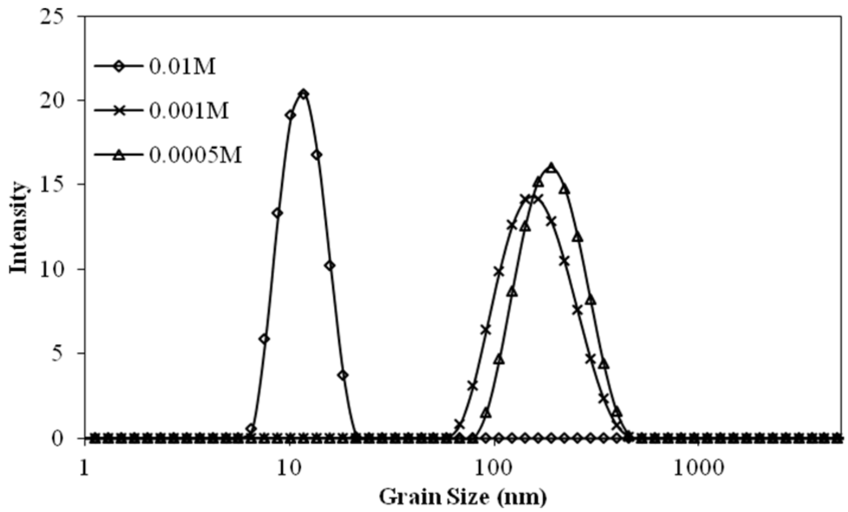


Fig. 11.
Grain Size Distribution of Kaolinite Supernatant at different HDTMA Concentrations


25
20
15
10
5
0
Intensity
0.01M
0.001M
0.0005M
1
10
100
1000
Grain Size (nm)