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MICRO-STRUCTURE AND HYDRAULIC CONDUCTIVITY OF SIMULATED SAND-BENTONITE MIXTURES

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Abstract—This paper describes the relationship between the micro-structure and hydraulic conductivity of simulated sand-bentonite mixtures (SSBMs) prepared with powdered and granular bentonite. Glass beads were used to simulate sand grains because of their superior optical properties. The micro-structure of SSBMs was observed using optical micrography and scanning electron microscopy. For mixtures prepared with powdered bentonite, the indications are that bentonite coats the particles. As the bentonite content increases, the thickness of bentonite coating increases and reduces the area available for flow. For mixtures containing granular bentonite, the dry bentonite granules occupy the space between the particles and then swell to fill the void space. As the bentonite content increases, the number of granules increases, leading to more void spaces being filled with bentonite. At higher bentonite content (>8%), flow paths devoid of bentonite are unlikely, and the hydraulic conductivity appears to be controlled by the hydraulic conductivity of bentonite. The changes in micro-structure that were observed are consistent with the decrease in hydraulic conductivity that occurs with increasing bentonite content. However, the relationship between hydraulic conductivity and bentonite content differs depending on whether a mixture contains powdered or granular bentonite.

Key Words—Bentonite Distribution, Hydraulic Barrier, Hydraulic Conductivity, Micro-structure, Sand-Bentonite Mixtures, Optical Micrographs, Scanning Electron Micrographs.

INTRODUCTION

Sand-bentonite mixtures (SBMs) have been used successfully for construction of hydraulic barriers when clayey soils are not available. An empirical approach based on laboratory tests is commonly used to determine the amount of bentonite required to achieve the desired hydraulic conductivity (Kenney *et al*., 1992). Hydraulic conductivity testing, however, may take several months to perform, especially at higher bentonite contents. An alternative approach is to use mathematical models to predict the hydraulic conductivity based on the properties of the sand and the bentonite (Chapuis, 1990; Abichou, 1999).

The predictive models generally assume that the SBM is an "ideal mixture", as reported by Chapuis (1990), Kenney *et al*. (1992), and Mollins *et al*. (1996). The SBM is assumed to be a two-phase material consisting of sand and bentonite gel (Chapuis *et al*., 1990). The bentonite is assumed to be distributed uniformly throughout the void space and has a swelling capacity (*i.e*. volume of bentonite after swelling) that is greater than or equal to the volume of the void space in the sand. Because the sand particles are effectively embedded in a matrix of hydrated bentonite, the hydraulic conductivity of an ideal mixture is controlled by the hydraulic conductivity of the bentonite (Kenney *et al*., 1992).

Sand-bentonite mixtures used in practice normally cannot be assumed to be ideal mixtures because they do not contain enough bentonite to fill the voids in the sand matrix. Alternatively, the bentonite is not distributed uniformly throughout the sand and thus does not swell to fill all of the available voids. Models to predict the hydraulic conductivity of these non-ideal SBMs require knowledge of how the pore-space in the sand changes as the bentonite content varies. The objective of this study was to investigate how bentonite is distributed in SBMs with low bentonite content (<10%) to elucidate how increasing the bentonite content causes the hydraulic conductivity of non-ideal SBMs to change from that of sand to that of bentonite. For simplicity, sand-bentonite mixtures were simulated with mixtures of uniformly graded glass beads and bentonite. The behavior of these mixtures is assumed to be similar to that of sand-bentonite mixtures. Future efforts will focus on how particle-size distribution affects the hydraulic conductivity of SBMs.

BACKGROUND

Graham *et al*. (1989) examined the particle micro-structure of SBMs with a bentonite content of 50% using scanning electron micrographs (SEMs). In this paper,

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micro-structure is defined as the arrangement of bentonite and sand particles in a SBM. At low magnification, the SEMs showed that larger particles of sand are separated by a matrix of bentonite and finer sand particles. At larger magnification, the finer sand particles appeared to be separated by a matrix of bentonite. Gnanapragasm *et al.* (1995) obtained SEMs of SBMs containing 10% bentonite that were permeated with water or aniline, an organic base. Bentonite occupied the void space and adhered to sand grains in the SBMs permeated with water. Significantly less bentonite swelling was observed for the SBMs permeated with aniline. Neither Graham *et al.* (1989) nor Gnanapragasm *et al.* (1995) correlated hydraulic conductivity and microstructure for their SBMs.

Komine and Ogata (1996) investigated the relationship between hydraulic conductivity of SBMs and swelling of the bentonite. A scanning electron microscope with temperature and vapor pressure control was used so that hydrated specimens could be observed. The SBMs having 5, 10, 20 and 50% bentonite content were evaluated. All mixtures were compacted to maximum dry unit weight and at optimum water content per standard Proctor compactive effort. The swelling of the bentonite was observed as bentonite adsorbed water by dew condensation. Scanning electron micrographs of each mixture were obtained before, during and after bentonite swelling. The SEMs show that voids were present in all mixtures before dew condensation and that these voids act as conduits or channels for water flow. The void size in the mixtures with 5 and 10% bentonite decreased as the bentonite adsorbed water; however, these mixtures still contained unfilled voids between the sand grains after swelling of the bentonite was complete. In contrast, no voids were present in the mixtures prepared with 20 and 50% bentonite content after swelling was complete.

Komine and Ogata (1996) correlated their microstructural observations to hydraulic conductivity. The hydraulic conductivity decreased by a factor of 20 (1×10^{-8} to 5×10^{-10} cm/s) as the bentonite content increased from 5 to 20% due to a reduction in size and quantity of unfilled voids between the sand grains. For bentonite contents >20%, the hydraulic conductivity decreased gradually by only a factor of five (5×10^{-10} to 1×10^{-10} cm/s) as the bentonite content increased from 20 to 50%. The mixtures with bentonite content >20% had smaller voids that were more readily filled by the swelling bentonite than the mixtures containing less bentonite. Increasing the bentonite content caused a smaller reduction in hydraulic conductivity for these mixtures because the bentonite in these mixtures filled all of the void space between the sand particles. As a result, increasing the bentonite content from 20 to 50% primarily resulted only in a reduction in the void ratio of the bentonite, which causes a more subtle change in the hydraulic conductivity.

Table 1. Mineralogy of powdered and granular bentonites.

Constituent	SS-100 (Powdered) (%)	Benseal® (Granular) (%)
Quartz	5	5
Cristobalite	14	2
Plagioclase feldspar	2	9
K-feldspar	–	2
Calcite	1	trace
Siderite	–	2
Analcime	–	3
Clinoptilolite	–	trace
Hornblende	–	trace
Montmorillonite	77	59
Illite/mica	1	1
Mixed-layer illite-smectite	–	17

MATERIALS AND METHODS

Mixtures

Simulated sand-bentonite mixtures (SSBMs) were prepared using glass beads to simulate sand grains and two types of bentonite: powdered and granular. Glass beads were used in place of natural sand to improve the optical properties of the mixtures. The difference in microstructure and hydraulic behavior obtained with glass beads instead of sand is assumed to be insignificant. The glass beads simulate a rounded-uniform sand with a median particle size of 1.2 mm. Glass beads have been widely used to simulate clean sands, such as filter media. For example, Chang *et al.* (1999) used glass beads to study head loss across filter beds and Hozalski and Bouwer (1998) used glass beads to study deposition and retention of bacteria in backwashed filters. Vallejo (2001) used glass beads to simulate sand grains in binary granular mixtures, and Boley and Overcamp (1998) used glass beads to investigate the displacement of non-wetting liquids from unsaturated sand.

The powdered bentonite was CETCO SS-100 (American Colloid Co., Arlington Heights, Illinois, USA), which is a Na-bentonite ground to 70% passing the No. 200 sieve. Benseal® (Baroid Corp., Houston, Texas, USA) was used for the granular bentonite. Benseal® is a Na-bentonite with a median particle size of 1.1 mm. The mineralogy of both bentonites, as determined by X-ray diffraction (XRD) during this study, is shown in Table 1. The free swell, as defined by ASTM D 5890, is 25 mL for the powdered bentonite and 32 mL for the granular bentonite.

Prior to mixing the glass beads and bentonite, the beads were lightly sprayed with water to simulate the natural moisture that commonly exists in the field and so that bentonite would stick to the surfaces of the glass beads. A known mass of bentonite (powdered or granular) was then added to achieve the desired bentonite content. The bentonite content was defined as the ratio of the weight of dry bentonite to that of the dry glass beads. The bentonite was added in increments to the beads as they were being mixed in a large container using a hand trowel. Once all of the bentonite had been added, the entire mixture was mixed thoroughly in the same container until it appeared uniform.

Two sets of specimens were prepared from each mixture. The first set was used to observe the swelling of bentonite in the presence of water and was prepared by tamping the mixture in a cylindrical container (50 mm in diameter and 6 mm deep). These specimens were then inundated with tap water to allow the bentonite to swell. Photomicrographs of these specimens, as described in subsequent paragraphs, were obtained before and after inundation. The second set of specimens was compacted at optimum water content and to maximum dry unit weight per standard Proctor (ASTM D698) in rigid-wall permeameters (~100 mm in diameter and 50 mm high). The desired dry unit weight was achieved by using the standard proctor hammer to compact a known weight of the mixture into the permeameter mold in two equal lifts. The compacted specimens were permeated with tap water, and then observed using photomicrographs and electron micrographs.

Observation methods

The SSBMs were examined using two methods: (1) photomicrographs using digital image acquisition software; and (2) scanning electron micrography (SEM). The objective was to observe the micro-structure of the mixtures as the bentonite content increased and as the bentonite hydrated. As stated earlier, the sand particles were simulated using glass beads to improve the optical properties of the mixtures.

The photomicrographs were obtained using a Navistar Macro-Zoom 18-108 mm lens coupled to a personal computer equipped with NI-IMAQ software interface developed by National Instruments Corporation. Scanning electron microscopy micrographs of the SSBMs were obtained using a Leo 1530 Field Emission Scanning Electron Microscope with an interface equipped for digital image acquisition and processing.

The SEM that was used requires that the specimens be dry for observation. To dry the specimens without destroying their structure, a freeze-drying method was used that consisted of directly immersing the specimens of SSBM in a container of dry ice. The freeze-dried specimens were then fractured and their exposed surfaces examined by SEM.

Hydraulic conductivity testing

Hydraulic conductivity tests were performed on the SSBMs using rigid-wall permeameters without swell rings following the methods in ASTM D 5856. The absence of swell rings confined the specimens vertically, preventing volume change. This condition does not simulate the field condition, where the stress is maintained constant instead of the volume. However, one intention of these tests was to study the void ratio of the bentonite as it swelled in the pore-spaces of a coarser material. If constant stress conditions had been used, non-uniform swelling would have occurred (specimens in rigid-wall permeameters typically swell more at the top) and precluded an accurate measure of the void ratio of the bentonite.

The falling head procedure was used, and no backpressure was applied. Tests were terminated when the hydraulic conductivity was steady, the last four hydraulic conductivity measurements were within 25%, and inflow equalled outflow. Hydraulic gradients were maintained between 28 and 32 and tap water was used as the permeant liquid. Typically, one pore-volume of water passed through the specimen during a test.

After the hydraulic conductivity tests were terminated, a suction of 1 m was applied by a hanging column to drain off free water. Optical and scanning electron photomicrographs were then obtained from the surface of the specimens, as described in the preceding section.

Hydraulic conductivity tests were also conducted on the powdered and granular bentonites alone to define the hydraulic conductivities of the bentonites and the anticipated hydraulic conductivities of ideal SBMs. Methods similar to those used for testing the SSBMs were employed. The hydraulic conductivity of the powdered bentonite was determined to be 7×10^{-9} cm/s at a void ratio of 4, whereas that of granular bentonite was found to be 9×10^{-9} cm/s at the same void ratio. Only one test was performed using each bentonite.

RESULTS AND ANALYSIS

SSBMs with powdered bentonite

Micro-structures. Photomicrographs of SSBMs prepared with powdered bentonite are shown in Figure 1. The mixtures correspond to different bentonite contents and were taken both after mixing and after inundation. Mixtures with 1, 2, 4 and 8% bentonite content are shown. The photomicrographs on the left correspond to as-mixed specimens and the photomicrographs on the right correspond to conditions after hydration by inundation with water.

At all bentonite contents, the bentonite coats the glass beads somewhat evenly during mixing and before introduction of water. The volume of bentonite coating around the glass beads increases as the bentonite content increases. When water is introduced, the bentonite adsorbs the water and swells, filling some of the void space between the glass beads. At a bentonite content of 8%, the swollen bentonite fills the entire void space and the mixture appears as a continuous matrix of bentonite with beads as inclusions. Flow pathways devoid of bentonite were not apparent. Under this condition, the hydraulic properties of the mixture should be controlled by the hydraulic conductivity of the bentonite.

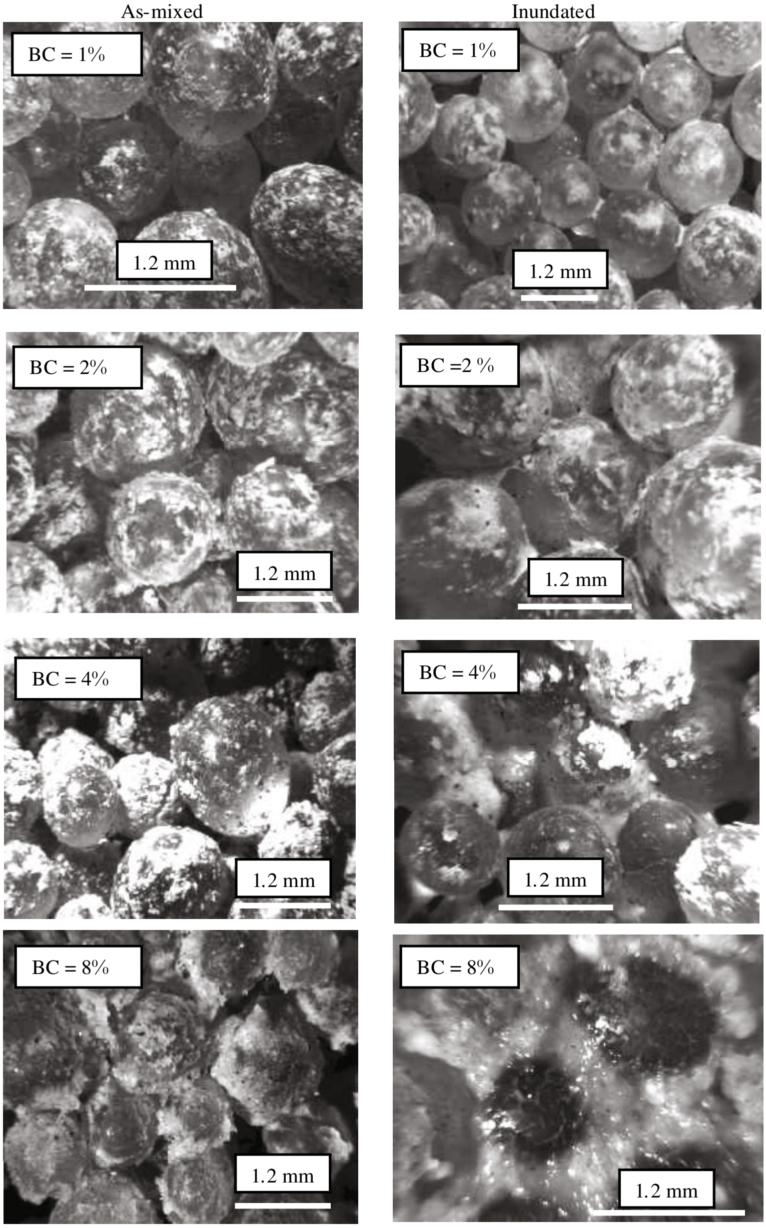

Figure 1. Photomicrographs of SSBMs prepared with powdered bentonite: (left) as-mixed and (right) after inundation with water.

Photomicrographs of SSBMs that were permeated are shown in Figure 2. The SSBMs contain 0, 2, 3 and 4% powdered bentonite. The mixture having no bentonite is provided for comparison. When the bentonite content is 2%, the beads are coated with bentonite and most of the spaces between the beads, which constitute the primary pathways of flow, are smaller relative to those in the glass beads alone. However, a number of large voids free of bentonite are available for water flow. As the bentonite content increases to 3%, the primary flow pathways become smaller as the thickness of the bentonite coating increases. The increase in thickness of the bentonite coating continues as the bentonite content increases to 4%. In the mixture with 4% bentonite, most of the visible flow pathways are filled with swollen bentonite. However, pores not filled with bentonite persist at bentonite contents of 3 and 4%.

The SEMs of freeze-dried SSBMs after permeation are shown in Figure 3. These specimens were freeze dried to preserve the micro-structure of the mixtures without shrinking the swollen bentonite. The mixtures were prepared with 1, 2, 3, 4, 6 and 8% powdered bentonite. As the bentonite content increases, the SSBMs change from a collection of glass beads with a light coating of bentonite to a matrix of bentonite with glass bead inclusions. As was observed in the photomicro-

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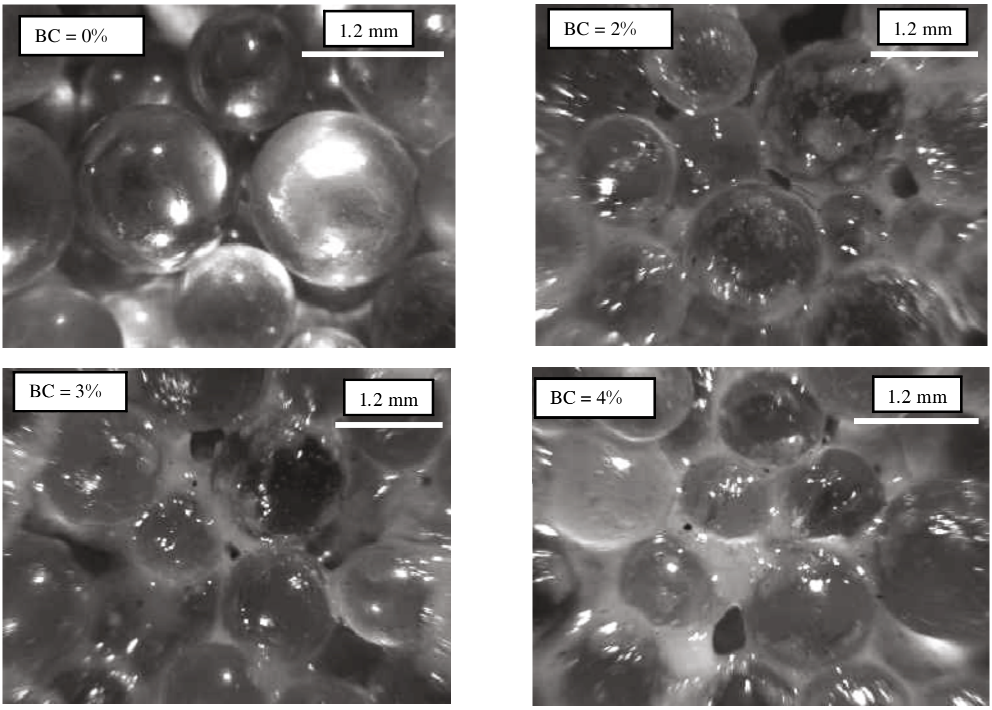

Figure 2. Photomicrographs of SSBMs prepared with powdered bentonite after permeation.

graphs, the bentonite coats the grains reducing the area available for flow. As the bentonite content increases, the thickness of the coating appears to increase and more of the area available for flow appears to be blocked by the additional bentonite. One might theorize that blockage occurs in the contact regions first (regions where glass beads touch each other), followed by pore-throats (regions between three neighboring glass beads) and then the pores (inside the void space formed between four glass beads). At a bentonite content of 8%, the majority of the void space between the grains appears to be filled with swollen bentonite.

Hydraulic conductivity. Hydraulic conductivity *vs.* bentonite content for SSBMs prepared with powdered bentonite is shown in Figure 4. The hydraulic conductivity decreases as the bentonite content increases, as has been observed by many investigators (*e.g.* Chapuis, 1990; Kenney *et al.*, 1992; Mollins *et al.*, 1996; Komine and Ogata, 1996). The decreasing hydraulic conductivity is characterized by three regions. In Region A, the hydraulic conductivity decreases only slightly (factor of 8) as the bentonite content increases from 0 to 2%. In Region B, the hydraulic conductivity drops dramatically (by more than four orders of magnitude), as the bentonite content increases from 2 to 5%. Thereafter, the hydraulic conductivity again decreases gradually with increasing bentonite content. At the highest bentonite contents, the hydraulic conductivity of the SSBM approaches that of bentonite alone (7×10^{-9} cm/s).

The changes in hydraulic conductivity of SSBMs shown in Figure 4 appear consistent with the changes in micro-structure shown in Figures 2 and 3. The behavior can be explained by the following hypothesis. When the mixture has no bentonite, the voids between the particles are large and act as preferential flow paths. The hydraulic conductivity is dictated by the geometry of the void space. As the bentonite is first introduced into the SSBM (BC = 0 to 2%), the bentonite coats the particles and causes only a modest reduction in the cross sectional area of the flow and a modest decrease in hydraulic conductivity. As more bentonite is introduced (2 to 5%), the thickness of the coating increases, fills the pore-throats between particles, and eliminates some flow paths responsible for conducting flow through the mixture. Eliminating these flow paths has a more direct influence on the hydraulic conductivity, and therefore the hydraulic conductivity starts to decrease rapidly with a slight increase in bentonite content.

When the bentonite content is high enough so that nearly all of the void space between the sand particles is filled by hydrated bentonite (BC >5%), the hydraulic conductivity of the mixture is controlled by the bentonite. Addition of more bentonite causes the hydraulic conductivity of the SSBM to decrease, primarily due to a reduction in void ratio of bentonite. The decrease in hydraulic conductivity is gradual, however, because the reduction in void ratio results in smaller and more tortuous flow paths, but not elimination of flow paths as occurs at lower bentonite contents.

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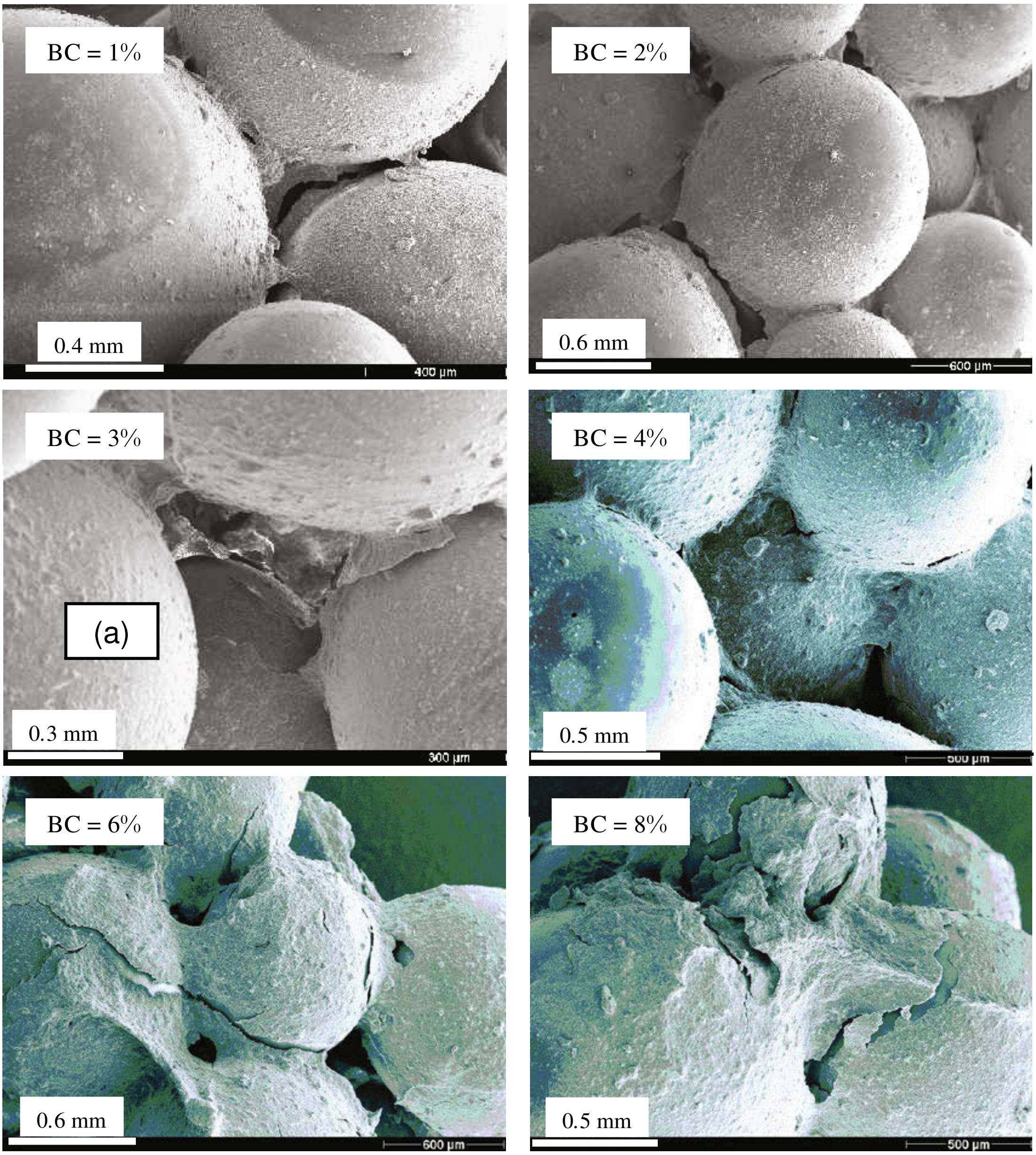

Figure 3. SEM images of freeze-dried SSBMs prepared with powdered bentonite.

SSBMs with granular bentonite

Micro-structures. Photomicrographs of SSBMs prepared with granular bentonite are shown in Figure 5. These SSBMs were permeated before the photomicrographs were obtained. The SSBMs have bentonite contents of 2, 4 and 6%. An SEM of a freeze-dried specimen of an SSBM prepared with 5% granular bentonite is shown in Figure 6. This SEM was observed before the SSBM was permeated, and provides a close-up of the distribution of bentonite in SSBMs prepared with granular bentonite.

The photomicrographs show that the mechanism of void filling is different for SSBMs prepared with granular bentonite. The bentonite granules occupy the spaces between the granular particles, and only a small portion of the bentonite coats the grains. During hydration, the bentonite granules swell and fill pores with hydrated bentonite, but little grain coating occurs. As the bentonite content increases, the number of granules increases, leading to more pores between the sand being filled with bentonite.

The use of uniform glass beads probably exaggerates the differences between mixtures prepared with powdered and granular bentonite. Sands tend to be more angular and more broadly graded than the glass beads,

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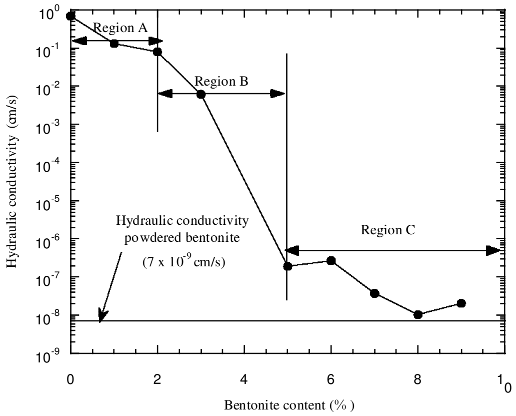

Figure 4. Hydraulic conductivity *vs*. bentonite content for SSBMs prepared with powdered bentonite.

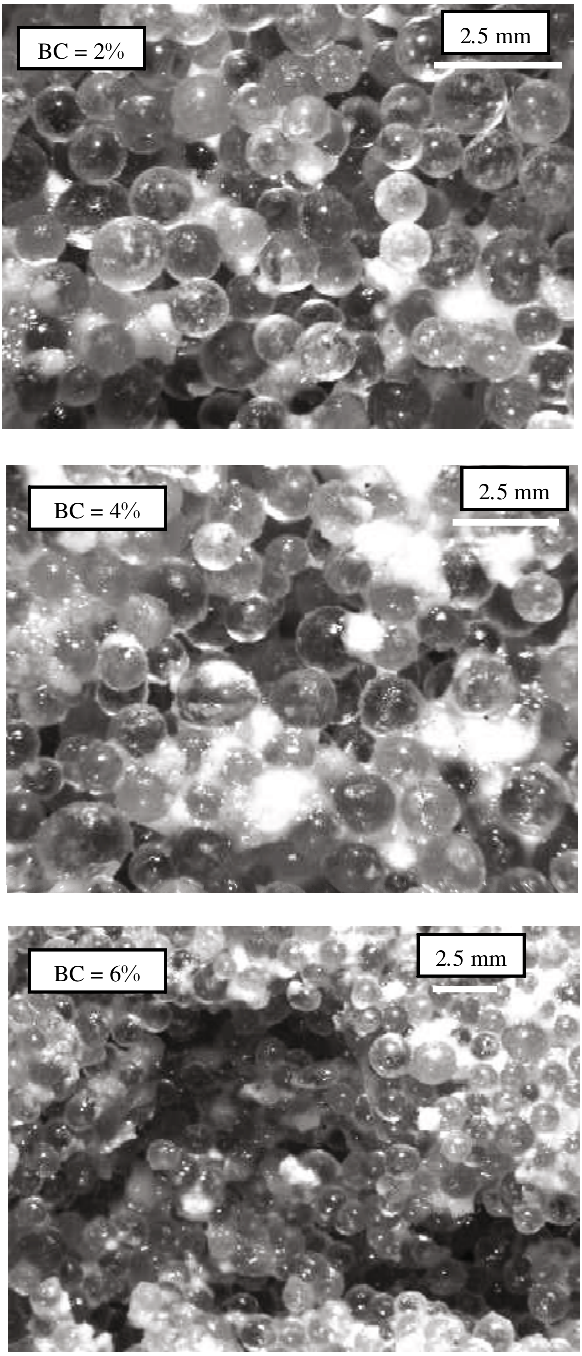

Figure 5. Photomicrographs of SSBMs prepared with granular bentonite after permeation.

and thus bentonite granules are more likely to be crushed into finer particles during compaction of mixtures prepared with sands.

Hydraulic conductivity. The hydraulic conductivity of SSBMs prepared with granular bentonite *vs*. bentonite content is shown in Figure 7. As with the SSBMs prepared with powdered bentonite, the hydraulic conductivity decreases with increasing bentonite content in a step-like manner. The hydraulic conductivity decreases very little (factor of 2) as the bentonite content increases from 0 to 2% (Region A). The hydraulic conductivity then decreases by more than two orders of magnitude as the bentonite content increases from 2 to 3%, which is followed by a gradual decrease in hydraulic conductivity (one order of magnitude) as the bentonite content increases from 3 to 8% (Region B). From 8 to 9% bentonite content (Region C), the hydraulic conductivity drops by almost three orders of magnitude. No data are available for SSBMs with bentonite contents >9%, but the hydraulic conductivity at 9% is similar to that of bentonite alone (9×10^{-9} cm/s) suggesting that the SSBM has approached an ideal mixture.

The hydraulic conductivity of SSBMs containing granular bentonite can be correlated to their microstructure. As the bentonite content increases from 0 to 2%, bentonite granules are introduced into the spaces between the grains. The bentonite granules swell and fill the space between the grains, blocking any flow path passing through these voids. However, the mixture still has an abundance of other flow paths that are responsible for conducting flow through the specimen (Figure 5), and therefore the hydraulic conductivity remains almost unchanged. As more bentonite granules are introduced into the mixture, many direct flow paths are blocked (Region B). As a result, the flow becomes controlled by more tortuous flow paths and the hydraulic conductivity drops abruptly as the bentonite content increases from 2 to 3%. As more bentonite is introduced into the mixtures, more and more flow paths are blocked, leading to a gradual decrease in hydraulic conductivity as the bentonite content increases from 3 to 8% (Region B). At a bentonite content > 8%, apparently all of the primary flow paths become blocked and the hydraulic conductivity becomes controlled by the bentonite and as a result the hydraulic conductivity of the mixture approaches that of pure bentonite (Region C).

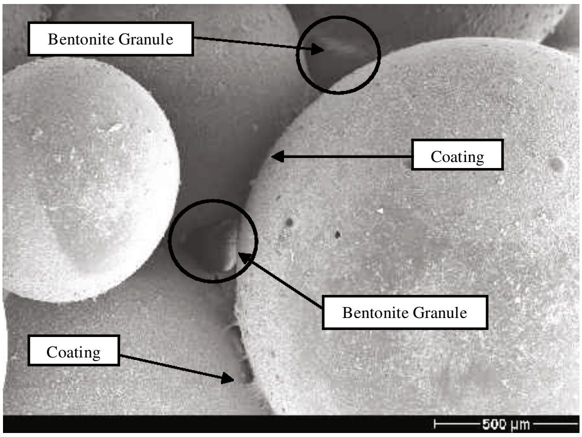

Figure 6. SEM image of SSBM prepared with granular bentonite at a bentonite content of 5%.

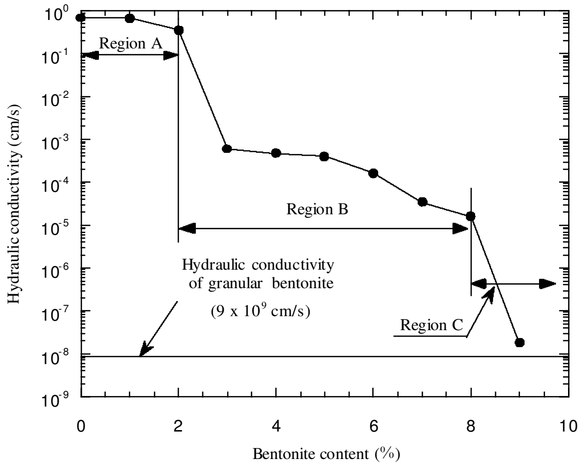

Figure 7. Hydraulic conductivity *vs.* bentonite content for SSBMs prepared with granular bentonite.

Hydraulic conductivity and void ratio

Hydraulic conductivity of the SSBMs *vs.* void ratio of bentonite is shown in Figure 8. Hydraulic conductivities of the powdered and granular bentonite at a void ratio of 4 are also shown in Figure 8.

The void ratio of bentonite in the mixtures was estimated after the hydraulic conductivity tests by weight-volume calculations after drawing off the excess water with a 1 m suction. This suction was assumed to draw off the excess water held by capillarity and not retained by bentonite. The remaining water was assumed to fill the voids in the swollen bentonite. The volume of the remaining water, along with the volume of dry bentonite introduced into the mixture, was used to calculate the void ratio of the swollen bentonite.

Ideal mixture theory suggests that the hydraulic conductivity of the mixtures should be lower than that of bentonite alone. However, Figure 8 shows that the mixtures have greater hydraulic conductivities when their bentonite has a void ratio of 4 than the hydraulic conductivity of pure bentonite at the same void ratio. Thus, at lower bentonite contents (in this case <4% for powdered bentonite, and <8% for granular bentonite), ideal mixture theory should not be used to predict the hydraulic conductivity of SBMs.

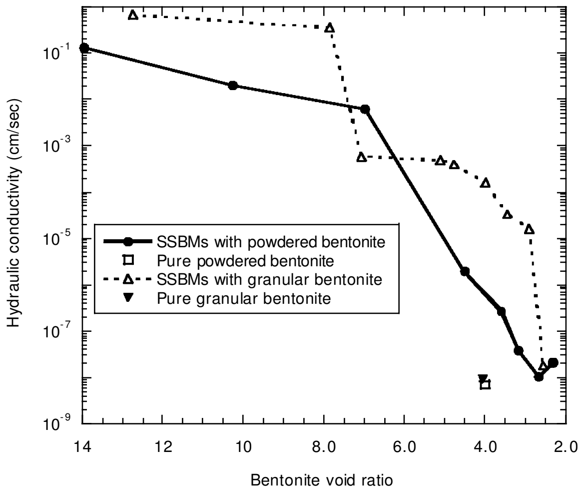

Figure 8. Hydraulic conductivity *vs.* void ratio of bentonite for all tests performed during the study.

CONCLUSIONS

Optical and SEM micrographs show that particle coating is the primary mode by which bentonite fills the pores in SSBMs prepared with powdered bentonites. A different mechanism controls void filling in SSBMs prepared with granular bentonite. Little grain coating occurs as shown in SEMs and optical photomicrographs of SSBMs containing granular bentonite. Instead, the bentonite granules occupy the space between the sand particles and then swell and fill the space with bentonite. The difference in micro-structures of mixtures prepared with powdered *vs.* granular bentonite was also reflected in the pattern of decreasing hydraulic conductivity with increasing bentonite content of the mixture.

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