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ABSTRACT

Permeation grouting using weak grouts such as bentonite grout is one of the effective methods to mitigate liquefaction in loose saturated sand deposits. While a flow parameter of the grout such as yield stress determines its penetrability into the deposit and resistance to groundwater flow, a dynamic parameter such as critical storage modulus evaluates postgrouting performance of the treated soils under cyclic loading condition. However, the yield stress and critical storage modulus should be obtained through two different types of rheological tests: drag and oscillatory shear test. Although previous research has suggested a method to evaluate yield stress from an oscillatory shear test, the conventional method does not consider the time-dependent nature of bentonite grout, which is one of its crucial properties as a grout. In this study, flow and dynamic rheological properties of bentonite suspensions were measured using drag (stress ramp) and oscillatory shear (strain sweep) tests with a vane geometry for various weight fractions of bentonite suspensions (5, 7.5, 10, and 12 %) and resting times (0 to 480 h). At different resting times, elastic and crossover stresses from strain sweep tests were compared to yield stresses obtained from stress ramp tests. The results showed that both the elastic and crossover stresses from strain sweep tests were significantly lower (40 %-60 %) than the yield stresses measured by stress ramp tests. The comparison also showed a dependency on particle fractions. In order to evaluate yield stress from the oscillatory shear test, a time-independent relationship between yield stress and critical storage modulus was proposed. This study suggests an economical approach to evaluate an important design parameter ("undisturbed" yield stress) in permeation grouting using bentonite grout.

Keywords

permeation grouting, bentonite, yield stress, vane, storage modulus, thixotropy

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Nomenclature

COV =	Coefficient	of V	Variation

- G' = storage modulus
- G'' =loss modulus
- G'_{cr} = critical storage modulus
- R^2 = coefficient of determination
- t = time

YP = yield stress

- $\gamma =$ Shear strain
- $\gamma_{o} =$ Maximum applied shear strain
- $\delta = \text{phase angle}$
- $\tau =$ shear stress
- $\tau_{v} =$ yield stress
- $\tau_{y,drag flow}$ = yield stress measured from drag shear test
 - $\tau_{crossover} = crossover stress$
 - $\tau_{elastic} = elastic stress$
 - $\omega = angular frequency$

Introduction

Bentonite has been widely used in civil engineering such as drilling fluids, slurry walls, and permeation grouting due to its thixotropic nature (Weaver 1993; Kazemain and Huat 2009). Recently, Haldavnekar et al. (2003), El Mohtar et al. (2008,2013) and Rugg et al. (2010) have studied the possibility of using bentonite grouts to improve soil performance under static and cyclic loading conditions. The bentonite grouts are delivered into granular deposits by permeation without disturbing original soil structures and play an important role to mitigate liquefaction by retarding the excess pore water pressure generation. For this application, it is required to obtain both flow and dynamic parameters of the grouts (such as yield stress and critical storage modulus) to design and evaluate grouting works. Yield stress, a threshold shear stress where a fluid starts to flow, determines the penetration of grout into soils during injection (Raffle and Greenwood 1961; Jeffris 1992; Janczesz and Steiner 1994; Gustafson and Stille 1996) and the stability of the grout against "washing-out" in pore spaces (Cambefort 1964). Critical storage modulus, a constant storage modulus at small strain range (less than 10 %), represents the solid-like behavior of viscoelastic material. The modulus controls resistance of treated soils to cyclic loading after treatment (El Mohtar et al. 2008,2013). Specifically, the yield stress and critical storage modulus after varying resting times are useful parameters to evaluate the long-term performance of grout. However, the yield stress and critical storage modulus must be obtained by two different types of testing methods (drag and oscillatory shear tests, respectively), and thus leads to an uneconomical testing program for the time-dependent measurements that required a number of samples. These parameters should also be

measured at "undisturbed" state without bias from the measuring process (sample disturbance and wall slip); otherwise, the long-tem performance of the grout is significantly underestimated (Yoon and El Mohtar 2013). The objective of this study is to investigate time-dependent dynamic rheological behavior of bentonite grouts and propose a method to evaluate the aged "undisturbed" yield stress from the dynamic rheological parameter of bentonite grouts.

Background

In both drag and oscillatory shear tests, the plate type geometry (either cone and plate or parallel plates) is typically used for measuring yield stress. However, using such geometries for measuring the undisturbed properties of specimens after long resting periods becomes challenging because the samples should remain in the setup for the entire resting time after preshearing. Otherwise, the samples are subject to disturbance during testing setup (Yoon and El Mohtar 2013). Drying of the edges of the specimens also could occur during long resting periods when using the plate type geometries.

Vane geometry measures the yield stress of thixotropic fluids such as bentonite suspensions without the effects of disturbance (from sample loading) and wall slip (during shearing) on the measured yield stress (Keentok 1982; Cheng 1986; Barnes and Carnali 1990; Barnes and Nguyen 2001; Stokes 2004; Yoon and El Mohtar 2013). Although the vane induces some disturbance during its insertion into the sample, such disturbance is considered minimal compared to that caused by other geometries (Dzuy and Boger 1983). In addition, the end effect is ignored by assuming the cylindrical shear surface for practical purposes (Barnes and Canali 1990). The vane has been used for the oscillatory shear test (Yanez et al. 1996; Zhang et al. 1998; Saak et al. 2001; Walls et al. 2003) to measure dynamic properties.

For the drag tests, stress ramp technique, which is a stresscontrolled test, is used to measure the yield stress with both geometries. In this technique, a constant level of shear stress (τ) is applied in an increasing stepwise, at a given interval of time, and the corresponding shear strains (γ) are recorded at the end of each interval. Initially, the shear strain increases linearly with shear stress at a slow rate (solid-like behavior); this rate increases significantly when the shear stress reaches sufficiently large values and the slope of the stress (τ)-strain (γ) curve becomes close to zero (liquid-like behavior). Two straight lines are extended from the solid-like (elastic) and liquid-like (viscous) regimes; the shear stress value at the intersection between these two lines is determined as the yield stress (Zhu et al. 2001; Clarke 2008; Yoon and El Mohtar 2013).

Oscillatory shear tests apply ramping up oscillatory strains at a constant level of frequency (Eq. 1). The shear stress is shifted by a phase angle δ with respect to the applied strain and

is expressed as the sum of an elastic component (in-phase, τ') and a viscous component (out-of-phase, τ'') with the applied strain (Eq. 2). Similarly, the shear modulus (Eq. 3) can be separated into an elastic and a viscous component, often referred to as storage (G') and loss (G'') modulus, respectively (Eq. 4).

 $G = \tau / \gamma$

(1) $\gamma = \gamma_o \cdot \sin(\omega t)$

(2)
$$\tau = \tau' + \tau''$$

(3)

(4)
$$\tau = \gamma_o \cdot [G' \sin(\omega t) + G'' \cos(\omega t)]$$

where:

 $\gamma =$ the shear strain,

 γ_o = the maximum amplitude of the shear strain,

 $\omega =$ the angular frequency, *t* is the time, and

G' and G'' = the storage and loss modulus, respectively. The critical storage modulus (G'_{cr}) is the storage modulus in elastic strain range where G' is essentially constant.

Yang et al. (1986), Yanez et al. (1996), and Pai and Khan (2002) determined yield stress as the maximum stress in the elastic range, which is expressed as follows:

(5)
$$\tau' = G' \gamma = \gamma_o \cdot [G' \sin(\omega t)]$$

Shih et al. (1999) suggested a method to estimate yield stress as the crossover stress. At such stress, storage modulus and loss modulus become equal and the phase angle (δ) is equal to 45°. This stress represents the phase transition from solid-like ($\delta < 45^\circ$) to liquid-like behavior ($\delta > 45^\circ$).

Previous research proposed relationships between the yield stresses measured using drag and oscillatory tests (Yang et al. 1986; Yanez et al. 1996; Shih et al. 1999; Pai and Khan 2002; Wall et al. 2003). However, the time-dependent behavior of thixotropic fluids, such as bentonite grouts, was not taken into account in any of these studies.

Experimental Program

MATERIALS AND SAMPLE PREPARATION

The physical properties of the sieved bentonite are summarized in **Table 1**. Wyoming sodium-bentonite (CP-200, CETCO, WY) was used in this study. The particles are platy in shape and less than 1 or $2 \mu m$ in diameter (Mitchell 1993; Luckham and Rossi 1999). The raw bentonite initially included impurities such as

TABLE 1	Physical	properties	of Wyoming	bentonite	(CP-2	200)
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Plastic Limit	38 %	Cation Exchange Capacity ^a	91 meq/100 g
Liquid Limit	440~%	Specific area ^b	712 m²/g
Specific gravity (Gs)	2.7	pН	9.3
Initial water content	8.3 %	Swelling capacity	16 ml/2 g

^aMethylene blue adsorption test.

^bCalculated the method suggested by Santamarina et al. (2002).

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plagioclase feldspar, orthoclase, and muscovite minerals and large particles (> 75 μ m); therefore, the bentonite was screened through a No. 200 sieve to minimize the impurities and their effects on the results (Abend and Lagaly 2000; Clarke 2008). This process produced approximately 95 % of particles smaller than 25 μ m and 50 % of particles smaller than 1 μ m (ASTM D422). No further purification or gradation was applied to simulate mass production of bentonite for field applications.

Since Na/Ca ratio is a critical parameter controlling the time-dependent rheological properties of bentonite suspensions (Brandenburg and Lagaly 1988), a chemical analysis was performed using Philips/FEI XL30 Environmental Scanning Electron Microscope (FEI Company, Hillsboro, OR) equipped with energy diffraction analysis of X-ray (EDX) to characterize chemical composition of the sieved bentonite powder. A Gaseous Secondary Electron (GSE) detector was utilized with frame and spot mode of the EDX. The Na/Ca ratio of the sieved bentonite was 1.9. The complete chemical composition of the sieved bentonite is presented in Table 2. An average pH of the suspensions measured using JENCO 60 pH meter was 9.3.

The weight fraction of bentonite suspension was calculated as the weight ratio of dry bentonite to the total weight of suspension. In the calculation of the bentonite fraction, the initial water content of bentonite was taken into account. The screened bentonite powder was mixed with de-ionized water, having a constant ionic strength of 2×10^{-5} mM, in three steps; each step consisted of 5 min of high shear mixing (Hamilton Beach 950 spindle mixer, Southern Pines, NC) of the suspension followed by manual scraping of the sides and base of the mixing cup to remove any attached bentonite flocs.

A Physica MCR 301 rheometer (Anton Paar, Granz, Austria) was used in this study. The vane used had six-blades and the thickness and length of each blade were 1 mm and 16 mm, respectively. The vane radius was 11 mm, resulting in a 3.46 mm of gap between the cup and the edge of the vane blades. For samples rested for extended periods of time before testing, the suspensions were poured into cups manufactured specifically for this study. The cups were 80 mm in length and 29 mm in internal diameter. Sample volume was maintained at 37 ml, which allowed the vane to penetrate approximately twice its length in the suspension. After pouring the suspension into

TABLE 2	Chemical a	analysis of V	Vvominc	hentonite	(CP-200)	wt %
			VVUIIIII			. VV L. 70.

0	53.05	K	0.52
Na	2.27	Ca	1.2
Mg	1.52	Ti	0.15
Al	9.07	Cr	0.11
Si	28.77	Mn	0.2
Р	0.46	Fe	2.31
S	0.43	Total	100
Cl	0.09	Na/Ca	1.9

Copyright by ASTM Int'l (all rights reserved); Tue Dec 10 15:15:50 EST 2013 Downloaded/printed by University of Texas at Austin pursuant to License Agreement. No further reproductions authorized. the storage cups, mineral oil was added on top of the bentonite suspensions to reduce any evaporation from the samples, and then the cups were tightly sealed afterwards and stored away. All suspensions were rested for a given time without any shearing and isolated from any vibrations. The mineral oil was removed with an eyedropper just prior to testing. The end effect of vane was considered small enough to ignore for practical purposes (Barnes and Carnali 1990). All tests were performed at $22^{\circ}C$ ($\pm 0.03^{\circ}C$); the temperature was controlled using the built-in Peltier temperature control system in the rheometer.

EXPERIMENTAL PROCEDURES

Two types of tests were performed in this study to determine the yield stress: the stress ramp (drag test) and the strain sweep (oscillatory test). The stress ramp technique, which is a stresscontrolled test, was utilized in this study. Each stress level was maintained for 12s and the strain was recorded at the end of the interval. The tests were programmed such that the samples rested for 2 min after inserting the vane to provide a consistent initial condition. The short resting time before shearing allows partial recovery of structures disturbed during the placement of the vane, although the disturbance is thought to be minimal. Preshearing was not applied prior to the measurements to simulate field condition (under no flow condition) and investigate a time-dependent change in yield stress from the initial mixing state. The ramp rates of 1 and 3 Pa/step were applied since those ramp rates produced reproducible yield stresses at the range of tested bentonite fractions (Yoon and El Mohtar 2013).

A series of strain sweep (oscillatory shear) tests were conducted at a constant oscillation rate of 1 Hz over the range of strains (0.01 to 1000 %). The frequency was selected since it was typical frequency used in cyclic tests (i.e., cyclic simple shear and cyclic triaixal test) for liquefaction evaluation. Although the critical storage modulus can be affected by the applied frequency, depending on materials, Geier (2004) observed that the behavior of bentonite suspensions was frequency independent at the range of 0.1 to 100 Hz. The pre-oscillatory shear stress at the strain of 0.01 % was applied for 2 min before testing, followed by increasing strains on a log scale. The storage and loss modulus were recorded at a rate of six measurements per log cycle. The pre-oscillation was applied to reduce the disturbance effect that occurs during inserting the vane into the suspensions (Clarke 2008).

Results and Discussion

YIELD STRESS FROM DRAG SHEAR FLOW

Figure 1 shows the yield stress for bentonite fractions of 5 %, 7.5 %, 10 %, and 12 % immediately after mixing. The yield stresses increased with the increase of bentonite fraction, indicating that yield stress is a function of particle fraction ($R^2 = 0.998$). The presented yield stresses are the average values





from at least three measurements and the coefficient of variation (COV) ranged from 0.02 to 0.07. Figure 2 depicts the thixotropic increase in yield stresses of 5, 7.5, 10, and 12 % bentonite suspensions. The yield stresses increased from 3.4, 25.8, 135.6, and 457.1 Pa to 33.6, 138, 423.4, and 984.1 Pa, respectively, after 480 h. The time-dependent increase in yield stress is attributed to the fact that the 3D networks develop in the suspensions with time. The 3D networks increase with bentonite fractions and resting times (Yoon and El Mohtar 2012). The initial yield stress increased rapidly, but the rate of the increase was reduced gradually as time elapses. The rapid increase in yield stress at short resting times (less than 24 h) is mainly due to the hydration of bentonite particles. The adsorption of water molecules between the interlayers of the clay platelets causes swelling of the bentonite, producing higher repulsive forces and weak structures are formed by the delaminated platelets. These platelets approach each other under the influence of Brownian motion building up network structures with resting time, resulting in a consistent thixotropic increase in yield stress with



200

300

Resting time (hrs)

400

500

600

0

100



resting time (Luckham and Rossi 1999). The yield stresses in this study were measured without preshearing stage to capture the change in yield stress from flow condition (mixing) to atrest condition (after injection) for the proposed application. The concentrated suspensions produced less increase in yield stress than those of the diluted suspensions. This reduced thixotropy is because Brownian motion of particles is constrained in the concentrated suspensions so that the time-dependent increase in yield stress is reduced compared to that of the diluted suspensions.



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MODULUS AND STRAIN FROM OSCILLATORY SHEAR FLOW

Elastic stress is affected by the change in both storage modulus and the corresponding strain (see Eq. 5), thus the change of both parameters with time is important to evaluate the elastic stress. Figure 3(a) shows a time-dependent increase in storage modulus of 10 % bentonite suspensions. Storage modulus increased gradually at small strains with increasing resting times, but tended to converge to an equilibrium state at high strains (over 100 %). This implies that the 3D networks in the suspensions are broken at a threshold strain, and then degrades to a residual structure which cannot be further broken at high strains (Yoon and El Mohtar 2013). Figure 3(b) depicts a time and bentonite-fraction dependent variation of the elastic strain (strain at the maximum elastic stress). The elastic strain increases with the decrease in bentonite fraction and increases with the increase in resting time. The elastic strains range from 464 % to 32 %, but remain relatively constant with time for the concentrated suspensions (10 and 12 %). Additionally, Yoon (2011) reported that the phase angle (δ) of bentonite suspensions within the elastic strain range, where the phase angle is relatively constant, decreases with the increase of resting time and clay content. These observations imply that the suspensions become more solid-like material with an increase in bentonite fraction as well as resting time. Figure 3(c) displays the modulus (G' or G'')-strain (γ) curves for 10 % suspensions at the resting time of 0 and 480 h. Both the storage and loss modulus increase with resting time. The crossover strain decreased over resting time similar to the elastic strain.

COMPARISON OF YIELD STRESS FROM DRAG AND OSCILLATORY SHEAR FLOW

Elastic and crossover stresses were evaluated from strain sweep tests and compared to yield stresses determined from stress ramp tests based on resting times. Figure 4(a) and 4(b) depict the variation of the normalized stress (elastic or crossover stress from strain sweep test divided by the yield stress from stress ramp test for the same bentonite fraction and resting time) with resting time. As the normalized stress approaches unity, elastic, and crossover stress become close to yield stress. At a given resting time, the normalized stresses decreased with increasing bentonite fraction. As time elapses, the ratios were reduced by approximately 15 to 70 %, reaching the ratios of approximately 0.4 to 0.6. The diluted suspensions (5 and 7.5 %) decreased more than the concentrated suspensions (10 and 12 %) with the resting times. The normalized stresses for the concentrated suspensions remained relatively constant with resting times. This implies that the determination of yield stress based on elastic or crossover stress could significantly underestimate the yield stress and would vary with particle fractions and resting time. The normalized stress for the concentrated suspensions showed less dependency on bentonite fraction and resting time



compared to the diluted suspensions. This is attributed to the fact that the elastic and threshold strains in the concentrated suspensions were relatively independent of resting time and bentonite fraction (see Fig. 3(b)).

CRITICAL STORAGE MODULUS

Critical storage modulus is defined as a constant value of storage modulus at small stain range (<10 % strain for bentonite suspensions). At such stain range, the storage modulus is constant and bentonite suspensions display a linear viscoelastic response. Beyond this range, the nonlinear viscoelastic responses are observed. This limiting character of the critical storage modulus is similar to that of yield stress; below the yield stress, the suspensions show a linear viscoelastic behavior that quickly changes into a nonlinear viscoelastic behavior. Figure 5 shows the variation of critical storage modulus at bentonite fractions of 5, 7.5, 10, and 12 %. The critical storage moduli presented in Fig. 5 are the average values from at least three measurements and the coefficient of variation (COV) ranges from 0.03 to 0.09. The overall behavior of the critical storage modulus was similar to that of yield stress. The critical storage modulus increases with the increase of bentonite fractions, suggesting



that critical storage modulus is also a strong function of particle fractions in the suspensions. Mahaut et al. (2008) observed similar phenomenon with the mixture of bentonite and glass beads. Figure 6 shows the time-dependent increase in the critical storage modulus. The critical storage moduli increased from 3.1, 73.9, 571.5, and 1965 to 52.3, 423.5, 1770, and 5460 Pa, respectively, after 480 h. The critical storage modulus increases in a similar way to the yield stress, indicating that the time-dependent buildup of 3D network structure in bentonite suspensions also governs the critical storage modulus of the suspensions with resting time. Similar to yield stress, 3D network structures build up with resting time under the influence of Brownian motion of the delaminated particles, resulting in a consistent increase in critical storage modulus with resting time.

Figure 7 shows the normalized yield stress and critical storage modulus, which represent the ratio of time-dependent increase in both parameters at a given resting time compared to their value at the initial state. The increase in critical storage



FIG. 5 Critical storage modulus of 5, 7.5, 10, and 12 % (wt./wt. %) bentonite



modulus is slightly larger than that in yield stress. Although yield stress and storage modulus are both a strong function of 3D network structures in bentonite suspensions, the critical storage modulus build-up was 18 to 40 % higher than that of yield stress. The discrepancy becomes larger for concentrated suspensions compared to diluted suspensions.

EVALUATION OF YIELD STRESS FROM CRITICAL STORAGE MODULUS

Figure 8(a) and 8(b) depict the comparison of yield stress and storage modulus at various particle fractions and resting times. Since critical storage modulus showed a similar tendency to yield stress with particle fractions and resting times, yield stress and critical storage modulus are directly correlated. As shown in Fig. 8(a), yield stresses increased with the increase of critical storage modulus regardless of particle fraction. Moreover, no significant effect from different resting times was observed (Fig. 8(b)), implying that yield stress is strongly correlated to critical storage modulus regardless of resting times. This is attributed to the fact that both the critical storage modulus and yield stress are controlled by the state of 3D networks in bentonite suspensions. This uniqueness of the relationship parameters (for a given material, bentonite in this study) is critical in that the dynamic parameter can be directly converted to the flow parameter regardless of both particle fractions and time-dependency. Regression analyses were performed to develop a representative relationship between yield stress and critical storage modulus and a power function was selected based on the previous observations. The empirical correlation is expressed as follows:

(6)
$$\tau_y = \alpha \Big(G'_c \Big)$$

where τ_{v} is the yield stress from stress ramp tests, G'_{cr} is the critical storage modulus from strain sweep tests, α and β are the empirical constants (1.43 and 0.75, respectively, $R^2 = 0.995$).



Comparison of yield stress and storage modulus at various (a)

FIG. 8

The proposed model was tested with 9 and 11 % bentonite suspensions at the resting times of 0, 0.5, 1, 24, and 48 h. Figure 9 shows the predicted versus the measured values. The predicted yield stresses agreed well with the measured values. The average ratio of predicted and measured yield stress was 0.96 with the standard deviation of 0.17 and COV of 0.18. The result







shows that the "undisturbed" yield stress can be evaluated with a good accuracy using the proposed correlation. This is beneficial in that the stability of grouts after any resting times can be estimated from the oscillatory shear test. However, it should be noted that this correlation was developed for the typical range of bentonite fractions in the suspensions (5 %–12 %) used in permeation grouting. Above and below this range, bentonite suspensions are not applicable in permeation grouting due to low efficiency (in case of low bentonite content, which is not enough to retard ground water flow/washing or excess pore water generation), a mixing problem, and low penetration depth (in case of high bentonite content).

Conclusion

A time-dependent drag and oscillatory behavior of bentonite grouts were investigated to evaluate yield stress from dynamic rheological tests. The conventional methods underestimated yield stress by 40 %–60 % with resting times because of the time-dependency of bentonite grouts, which is one of the crucial properties required in permeation grouting. The estimated yield stresses also varied with particle fractions. This implies that yield stress from drag flow test could not be accurately evaluated with the conventional methods.

The proposed framework evaluates the aged "undisturbed" yield stress from critical storage modulus measured by oscillatory shear test. Critical storage modulus showed similar trends to those observed in yield stress. The critical storage modulus increased with particle fractions and resting times, but the rate of time-dependent increase of storage modulus is higher than that of yield stress.

Regression analyses of yield stress and critical storage modulus, at zero resting time, showed that the yield stress and critical storage modulus have a unique relationship regardless of particle fractions. It was found that the relationship is also valid for the values measured at various resting times. Using this relationship, "undisturbed" yield stress from critical storage modulus could be reasonably predicted. Although the proposed model was developed for bentonite suspensions, it is expected that the same model can be used for different types of weak grouts. This framework is attractive in terms of an economical and cost effective determination of the "undisturbed" yield stress of bentonite grouts from a dynamic rheological parameter.

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