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TIME EFFECTS ON TOTAL SUCTION OF BENTONITES

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ABSTRACT

This paper presents a study on time effects on total suction of bentonite-based materials under constant water content conditions. Three different types of bentonite (i.e., MX80, Calcigel I, and Calcigel II) and a natural expansive clay (i.e., London Clay) were used. Total suction was measured using chilled-mirror hygrometer technique. The measurements were performed on specimens aged 1 hour, 6 hours, 1 day, 2 days, 1 week, 2 weeks, 1 month, and 2 months. The results show that the true equilibrium state for two types of bentonite (i.e., MX80 and Calcigel I) used is time dependent. Total suction increases with time and this behaviour is controlled by the characteristics of the bentonites. Total suction of the other specimens (i.e., Calcigel II and London Clay) is not affected by curing time. The changes in total suction with time as the specimen's age are attributed to non homogeneity of the water content distribution in the micro- and macropores of the bentonites and inaccuracy of the device used.

Key words: bentonite, chilled-mirror hygrometer, time effect, total suction (IGC: B10/D1/D2/D3/D4)

INTRODUCTION

Soil suction is an important stress state variable in unsaturated soils. Measurement of soil suction is crucial for understanding the engineering behaviour of unsaturated soils. In semiarid and arid regions, soils can be so dry that the total suction may reach several hundreds of MPa. The measurement of high suction becomes more important when dealing with bentonite that can be used in hazardous and radioactive waste containment facilities and remediation systems, or as alternative covers for waste containment.

Total suction of most soils is measured using thermocouple psychrometers. However, this method can only be used to measure total suction from 100 to 8000 kPa (Likos and Lu, 2003; Agus and Schanz, 2005). Chilled-mirror hygrometer technique, on the other hand, has been extensively used for measuring the total suction of soils up to values higher than the upper limit (e.g., Leong et al., 2003; Albrecht et al., 2003; Thakur et al., 2005; Patrick et al., 2007; Leong et al., 2007).

Patrick et al. (2007) compared total suction measured using the chilled-mirror hygrometer technique and the filter paper method for expansive soils with liquid limits from 42–64%. General agreement with insignificant discrepancies were obtained between the total suction measured using the chilled-mirror hygrometer technique and that measured using the filter paper method. It was stated that the discrepancies were due to incomplete equilibration in the chilled-mirror hygrometer and variations of zero-water content intercepts in the calibration curves of the filter paper used. From both investigations and also similar investigations performed by other researchers (e.g., Leong et al., 2003; Albrecht et al., 2003; Leong et al., 2007), it seems apparent that no time effects were considered important in the study. It is understood since the materials used in the study were generally with low plasticity.

Highly plastic clays such as bentonite exhibit double porosity structure. The structure consists of inter-aggregate pores (or macro-pores) and intra-aggregate pores (or micro-pores) (Gens and Alonso, 1992; Yong, 1999). For these clays, the true equilibrium state is defined by the hydraulic equilibrium between the pores of different levels (Alonso, 1998). Using scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP) tests, Delage et al. (2006) observed the time-dependent changes in the micro-structure of a compacted sodium-type bentonite (MX80) as a result of the redistribution of porewater in the specimen. Agus and Schanz (2005) compared the total suction data of bentonite-sand mixtures obtained from the chilled-mirror hygrometer measurement and those measured using the filter paper method. The results show that total suction measured using the chilled-mirror technique was lower than that measured using the filter paper technique. The difference was attributed to

different ages of the specimens used. After enough curing time (i.e., approximately 2 weeks), it was found that total suction measured using the filter paper method and that measured using the chilled-mirror hygrometer was similar.

The concentration and type of absorbed cations in bentonites usually control the basic fabric unit of the bentonites. Sodium bentonite particles can separate into unit layers and interwoven network of filmy particles may form whereas calcium bentonite particles are usually made up of several unit layers (Mitchell, 1993). Pusch et al. (1990) observed that sodium bentonite has higher swelling pressure and lower permeability than calcium bentonite. From their studies, it was expected that bentonites with different type of cations would exhibit different time dependent behavior since the time to reach the true equilibrium state is influenced by the permeability of the bentonite (Delage et al., 2006).

To date, there have been no comprehensive studies on this aspect with rigorous total suction measurements. This paper presents a study on time effects on total suction of bentonites. The chilled-mirror hygrometer technique was utilized to measure total suction of three different types of bentonite and a natural expansive clay.

MATERIALS USED

Three types of bentonite (i.e., MX80, Calcigel I, and Calcigel II) and a natural expansive soil (i.e., London Clay) were used in this study. The physical, chemical, and mineralogical properties of the material used are summarized in Table 1. It can be seen from the table that MX80 has the highest values of plasticity, SSA, CEC, and montmorillonite content. The lowest values belong to London Clay, while Calcigel I and Calcigel II have values in between the highest and the lowest values. The bentonites used have different main exchangeable cations (i.e., Na^+

for MX80 and Ca^{2+} and Mg^{2+} for Calcigel I and Calcigel II).

In particular, it can also be seen that MX80, Calcigel I, Calcigel II, and London Clay have hygroscopic water content of 10.8, 9.2, 6.2, and 4.6, respectively. Hygroscopic water content is water absorbed from the atmosphere and held very tightly by the soil particles. Such water can only be removed from the soil specimen by heating. In this research, hygroscopic water content was the initial water content of the specimen taken from its pack and determined using ASTM standard D2216 (ASTM, 1997). It is clear from the table that the values of hygroscopic water content decrease by decreasing plasticity, SSA, and montmorillonite content of the specimen. It suggests that there is a correlation between the hygroscopic water content and the physical, chemical, and mineralogical properties of soils.

TECHNIQUES AND PROCEDURES

Chilled-Mirror Hygrometer

The chilled-mirror hygrometer used was a water activity meter (type 3TE, Decagon Devices, Inc., Pullman, WA). The device is shown in Fig. 1. It consists of a mirror and a photodetector cell, a temperature sensor, a fan, a sealed chamber, and a digital display. The soil specimen is fed to the device by placing it in a small container (Fig. 1(a)), which in turn is placed on a tray of a drawer, and pushing the drawer into the device. Vapor from the specimen then fills the headspace above the container. Air in the chamber is circulated by the fan to spread vapor evenly during the measurement. The condensation of water vapor at its first appearance is detected by the mirror and reflected to the photodetector cell. The temperature sensor measures temperature at which the condensation occurs. Using the temperature, the water activity or relative humidity is automatically calculated and is shown in the

Table 1.   Summary of physical, chemical, and mineralogical properties of material used

Properties	MX80[¥]	Calcigel I	Calcigel II	London Clay
Specific gravity	2.76	2.80	2.75	2.73
Liquid limit (%)	411 ± 10	180	84	76
Plasticity index (%)	374 ± 10	124	48	49
Specific surface area (SSA) (m^2/g)[†]	562	525	400	234
Basic exchangeable cation Na^+, Ca^{2+}, Mg^{2+}, K^+ (meq/100 g)[‡]	62.4, 7.4, 3, 0.2	3, 50, 21, 2	2, 50, 13, 1	—
Total cation exchange capacity (CEC) (meq/100 g)[‡]	73	76	66	—
Montmorillonite content (%)[*]	75	55	45	32[§]
Hygroscopic water content (%)	10.8	9.2	6.2	4.6
Total suction at hygroscopic water content (kPa)	89465	152607	216738	126655

[¥] Müller-Vonmoos and Kahr (1982).
[§] Dineen (1997).
[†] determined using the Ethylene glycol monoethyl ether (EGME) method (Cerato and Lutenegger, 2002).
[‡] determined using Silver Thiourea (AgTH) method (Dohrmann, 1997) and Ammonium Acetat method (Müller-Vonmoos and Kahr, 1982) for Calcigel specimens and MX80, respectively.
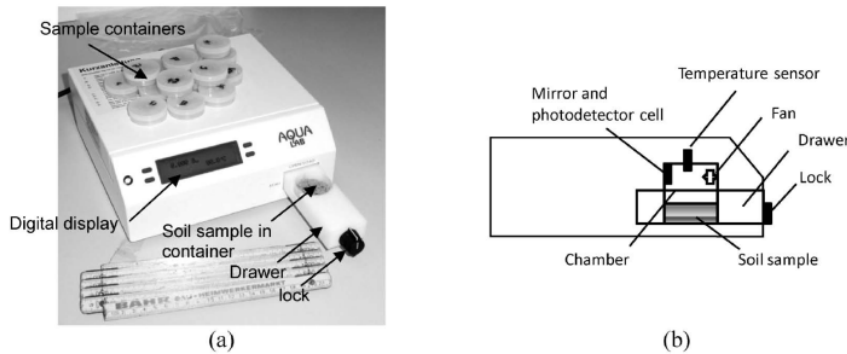[*] determined using X-ray diffraction method.

Fig. 1.   Photo and schematic picture of the chilled-mirror hygrometer

display.

The total suction is calculated using the thermodynamic relationship between soil suction (or the free energy of the soil water) and the partial pressure of the pore water (Eq. (1)).

$$s_t = -\frac{RT}{M_w(1/\rho_w)} \ln\left[\frac{RT}{100}\right] \qquad (1)$$

where s_t is total suction in kPa, R is the universal gas constant (i.e., 8.31432 J/mol K), T is the absolute temperature in Kelvin, M_w is the molecular weight of water (i.e., 18.016 kg/kmol), ρ_w is the unit weight of water in kg/m^3 as a function of temperature, and RH is the relative humidity. The derivation of Eq. (1) is given in Lu and Likos (2004).

The device was first checked for its accuracy using standard salt solutions provided by the manufacturer. The salt solutions used were 0.5 molal KCl, 6.0 molal NaCl, 8.57 molal LiCl, and 13.41 molal LiCl giving water activity (a_w) or RH of 0.984, 0.760, 0.500, and 0.250, respectively (Decagon Devices Inc, 2002). The salt solution used is close to the range of the suction measured. The readings obtained for the standard salt solutions were found to vary less than 0.3% RH with a response time of no longer than 3 minutes.

Specimen Preparation

The experimental work commenced by mixing the specimen with deaired-distilled water to reach a target water content of 14% and 23%. The specimens were then placed in glass containers with fastened cap and left to age. The air space in the container was minimized. In this way, condensation in the container due to change in temperature particularly for specimen with very low total suction could be prevented. Total suction measurements were performed after the specimens reached ages of 1 hour, 6 hours, 24 hours (1 day), 48 hours (2 days), 168 hours (1 week), 336 hours (2 weeks), 720 hours (1 month), and 1440 hours (2 months).

Total Suction Measurement

The choice of the initial condition of the benonite (i.e., whether it is loose or compacted) has no effect on the total suction measurement (Yahia-Aissa et al., 2000; Delage and Cui, 2008). Loose specimen was used in this study. The first total suction measurement was performed on the specimen aged for 1 hour.

Approximately one-half of the specimen container in the device is filled with the soil specimen (Fig. 1(a)). After the container is inserted into the device, the temperature difference between the specimen and the air space in the chamber is shown on the display. An isothermal equilibrium between the specimen and the chamber is reached when the temperature difference shown on the display is almost zero. The measurement is started by locking the drawer in its position. Water activity or relative humidity and temperature are shown in the display for no longer than 3 minutes.

The measurement was repeated until at least 3 readings showed the same magnitude of relative humidity. Time to reach this condition was recorded in order to investigate the time for total suction measurement using the equipment. Water content of the specimens was determined according to ASTM D-2216 (ASTM, 1997) immediately after the total suction measurement. The tests were performed in a temperature-controlled room at 20 ± 0.5°C. The same procedures were applied to specimens at ages of 6 hours, 24 hours (1 day), 48 hours (2 days), 168 hours (1 week), 336 hours (2 weeks), 720 hours (1 month), and 1440 hours (2 months).

RESULTS AND DISCUSSIONS

Total Suction Measurement

Figure 2 shows typical curves obtained from the total suction measurements using chilled-mirror hygrometer technique. The curves present both relative humidity (RH) and temperature as a function of time. Generally, RH values tend to increase with time and reach constant value after several readings. For example, specimens aged for 1 hour reached constant RH values (i.e., 68.7%) in 12 minutes. The time to reach constant RH values for all

specimens tested is listed in Table 2.

For MX80 with 25% water content, the time to reach constant RH values are different for different specimens with different ages. No discernable trends are obtained with respect to correlation between time to reach stable RH readings and aging or the characteristics of bentonites.

An analysis was performed to investigate possible errors due to variation of temperature during total suction measurement. As shown in Fig. 2, the temperature in the chamber during measurements varied in the range of 21.2–21.4°C. These variations occurred only during measurements of 2 days and 2 months. The temperature altered from 21.3 to 21.4°C and from 21.4 to 21.3°C for the specimens at 2 days and 2 months age, respectively. It implies that the temperature variation in the chamber was about 0.1°C. These variations were mainly due to differences in the initial temperatures of the specimens and the chamber. Similar pattern was also observed for the other specimens.
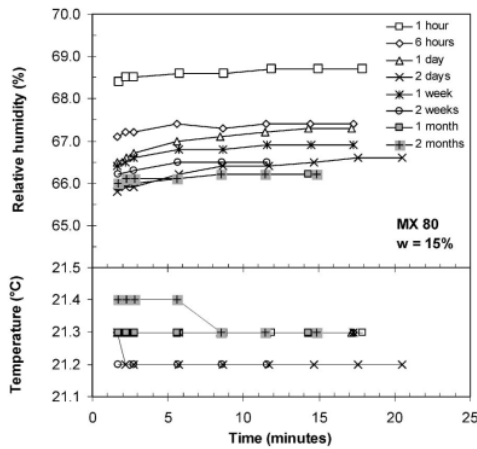
The total suction measurement error due to temperature variations can be computed using the first derivative of Eq. (1) with respect to temperature (Agus and Schanz, 2006) (Eq. (2)).

$$\frac{d(\ln s_t)}{dT} = \frac{1}{T} - \frac{1}{P_o \ln (RH)} \frac{d(P_o)}{dT} \qquad (2)$$

P_o varies with T and the variation of P_o with T can be found, for instance, in Lide and Frederikse (1994). By introducing the temperature variation or gradient (dT) occurring in the device (i.e., 0.1°C), the change in total suction (ds_t) encountered for a minimum measured total suction of 1000 kPa is about 2 kPa. Therefore, measurement of total suction higher than 1000 kPa would expectedly exhibit negligible errors due to temperature variation.

Error in total suction measurement may also arise due to inaccuracy of the device used. The change in suction due to the inaccuracy of the device can be calculated from the first derivative of Eq. (1) with respect to relative humidity (Eq. (3)).

$$\frac{ds_t}{d(RH)} = \frac{RT}{M_w(1/\rho_w)RH} \qquad (3)$$

Since $d(RH)$ is essentially the inaccuracy of the device (i.e., $\pm 0.3\% RH$; Decagon Device Inc., 2002), the change in total suction (ds_t) encountered at 20°C (or 293 K) is about ± 405 kPa.

Time Effect on Total Suction

Figures 3(a) and (b) show total suction as a function of time for the MX80 specimen at $w = 25\%$ and $w = 15\%$, respectively. The measurement error due to the inaccuracy of the device is also plotted as a band of total suction versus time curve. The error is not obvious in Fig. 3(b) for the MX80 specimen with 15% water content since the error value is too small compared to the measured total suction.

Based on Fig. 3(a), the total suction after 1 hour of aging is 12000 kPa. The total suction shows almost constant values up to a period of 1 week (or ± 168 hours). After aging for 2 weeks (or ± 336 hours), the total suction of the specimen increases reaching 13000 kPa after 2 months

Fig. 2.   Relative humidity and temperature versus time for MX-80 with water content of 15%

Table 2.   Summary of time to reach constant RH in the total suction measurements

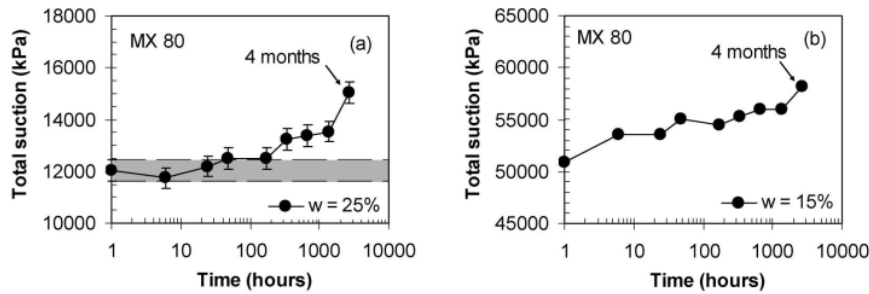| Material	Water Content (%)	Time to reach equilibrium (minutes)							
		1 hour	6 hours	1 day	2 days	1 week	2 weeks	1 month	2 months
MX80	25	13	5	9	17	11	14	11	6
MX80	15	12	12	14	18	12	6	9	9
Calcigel I	23	13	13	18	16	13	9	11	11
Calcigel I	14	19	6	12	6	12	15	9	12
Calcigel II	22	9	13	12	18	11	13	8	13
Calcigel II	14	9	9	16	12	13	12	15	6
London clay	22	5	15	13	11	5	6	11	13
London clay	13.5	11	10	8	5	8	5	8	8

Fig. 3.   Total suction as a function of time for the MX80 specimens with: (a) water content of 25% and (b) water content of 15%
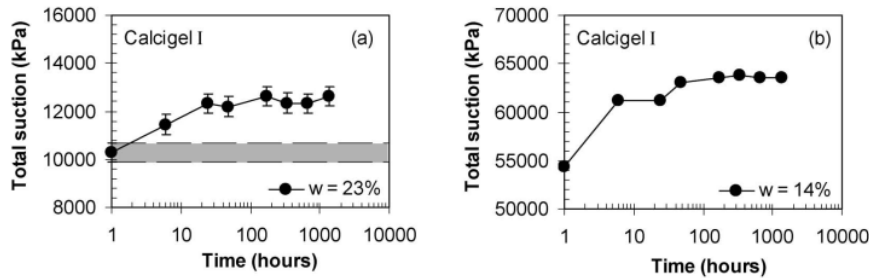

Fig. 4.   Total suction as a function of time for the Calcigel I specimens with: (a) water content of 23% and (b) water content of 14%
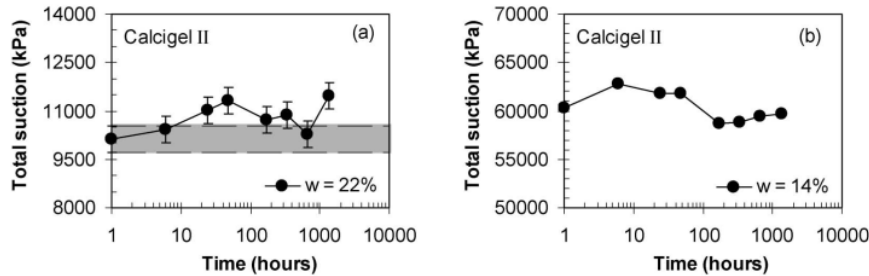

Fig. 5.   Total suction as a function of time for the Calcigel II specimens with: (a) water content of 22% and (b) water content of 14%

(or ±1400 hours). For a confirmation, an additional test has been done with an independent specimen with an age of 4 months (or ±2900 hours). The result shows that the total suction of this specimen was 15000 kPa or 25% higher than at the age of 1 hour. Similar phenomena were also observed in the MX80 specimen with 15% water content as shown in Fig. 3(b). However, the increase in total suction appears to be more rapid than that in the specimen with 25% water content. The total suction of the specimen starts to increase after aging for 6 hours. An additional test was also performed for this water content after aging for 4 months (or ±2900 hours). The total suction obtained was 58200 kPa or 15% higher than that at the age of 1 hour (i.e., 50900 kPa).

Figures 4(a) and (b) present the plot for total suction versus time for the Calcigel I specimens with 23% and

14% water content, respectively. The total suction at the age of 1 hour was 10300 kPa and it increased up to the age of 24 hours (or 1 day). After 2 days, total suction of the specimen flattened. The specimen's total suction after aging for 2 months (or 1440 hours) is 12636 kPa or 23% higher than that after 1 hour. For the specimen with 14% water content, the total suction after aging for 1 hour was about 54300 kPa and increased from 6 hours. The total suction of this specimen remained constant after 48 hours (2 days). The total suction after aging for 2 months was about 63500 kPa or about 17% higher than that after 1 hour.

Figures 5(a) and (b) show the evolution of total suction of the Calcigel II specimen with curing time with 22% and 14% water content, respectively. Figure 5(a) indicates that total suction after aging for 1 hour was about
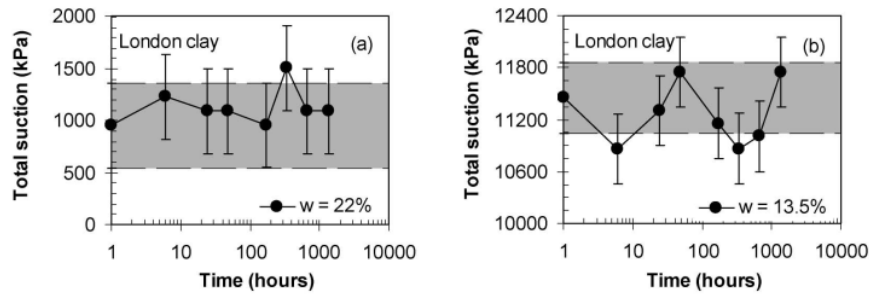
Fig. 6. Total suction as a function of time for the London Clay specimens with: (a) water content of 22% and (b) water content of 13.5%
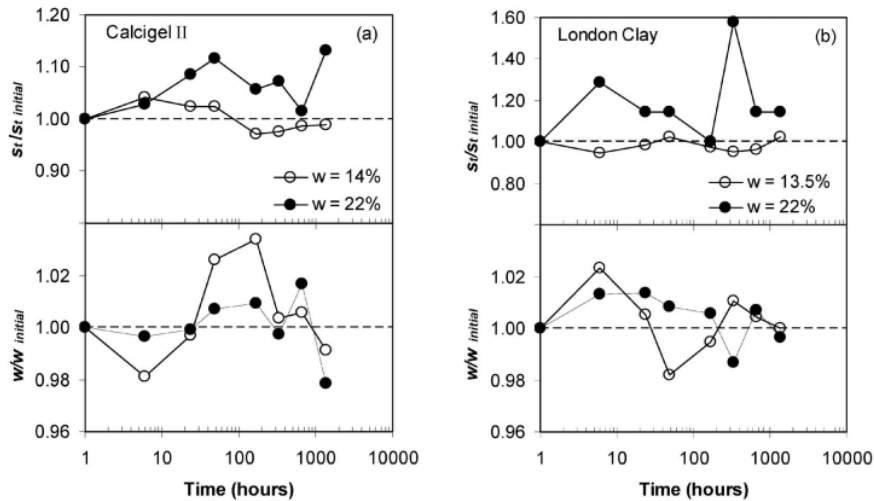

Fig. 7. Normalized water content and total suction versus time (a) for the Calcigel II specimens and (b) for the London Clay specimens

10100 kPa and increased with time to 11300 kPa after 48 hours. The total suction versus time plot shows a decrease in total suction down to 10300 kPa when the specimen reaches the age of 1 month (or 720 hours). Similar phenomena are shown in Fig. 5(b) for the specimen with 14% water content. The total suction of this specimen at the age of 1 hour was about 60300 kPa and increased to 60852 kPa after aging for 6 hours before decreasing to 58600 kPa after aging for 1 week (or 168 hours). At the end of the test, the total suction (2 months or 1440 hours) was about 59700 kPa which is close to the total suction after aging for 1 hour. The evolution of total suction with time as shown in Figs. 5(a) and (b) are not considered to be due to the effects of pore water distribution since the trends are different from those shown in Figs. 3 and 4. The total suction changes measured with time for the Calcigel II specimens fall within the error band. Thus, these changes seem to be due to measurement errors.

Figures 6(a) and (b) show the total suction versus time for the London Clay specimens with 22% and 13.5% water content, respectively. The total suction after aging

for 1 hour was about 950 and 11500 kPa for 22% and 13.5% water content, respectively. The total suction versus time curve is also erratic as changes fall within the error range due to device inaccuracy as shown in the plot as a gray band.

It seems that total suction of the Calcigel II and London Clay specimens is not time dependent. This can be attributed to the soil being less active compared to MX80 and Calcigel I. Internal redistribution of moisture in the specimen did not really occur and as such no significant suction changes were observed.

A further analysis was performed by normalizing the total suction data with the total suction after aging for 1 hour. The water content of the specimens at each age was also normalized in order to prevent non homogeneity of water content in the mixture. Figures 7(a) and (b) show the normalized total suction and water content as a function of time for the Calcigel II and London Clay specimen, respectively. The water content data located below the unity line as shown in dash line should result in the total suction located above the unity line, and vice versa.

The normalized total suction data of the Calcigel II specimen with 14% water content as shown in Fig. 7(a) are more consistent than those of the specimen with 22% water content. For specimen with 22% water content, the inconsistency occurs at the age of 48 and 168 hours.

Similar phenomena are also observed for the London Clay specimen as shown in Fig. 7(b). The specimen with 13.5% water content shows a consistent trend. For the specimen with 22% water content, the inconsistent trend occurs between 6 and 168 hours and at 1440 hours ages.

Discussion

From the results presented, it can be seen that the total suction values of the MX80 and Calcigel I specimens are time dependent, that is they increase with time. Figure 8 shows a possible mechanism for indicating how the total suction of a specimen changes with time under constant water content condition after the specimen is subjected to a wetting process. Before mixing with water or at the initial condition, the specimen is in the hygroscopic water content state (point 1). The measurement using the chilled-mirror hygrometer on the specimen immediately after the wetting process (point 2) only gives the total suction as the state of the macro-pores of the specimen. This value is not the true equilibrium total suction, but rather the total suction at a quasi-equilibrium state. When the specimen is kept for some period of time at constant water content (from points 2 to 3), an internal redistribution of pore water is expected to occur due to unbalanced total suction between the micro-pores and the macro-pores.

Delage et al. (2006) observed an increase in the volume of tiny pores not intruded by mercury in the MIP test for specimens cured at constant water content. Since water flows from high water potential, this was shown by measuring lower total suction immediately after water was mixed, total suction increases as water flows to the micro-pores under suction gradient effects. Combining the finding obtained by Delage et al. (2006) and this research, the plausible mechanism of pore-water redistribution and internal swelling postulated is that when soil is mixed with water, most of the water added is located in the macro-pores. This state is referred to as a quasi-equilibrium state since the water potential equilibrium only exists in the macro-pores. With time, pore-water is redistributed to the micro-pores driven by suction gradient resulting in higher total suction being measured. The pore-water redistribution increases the volume of micro-pores in soil as indicated by the increase in unintruded pores in the MIP test.

Figure 9 shows the distributed suction, which is defined as the difference between initial total suction and total suction at 8 weeks or (1440 hours) versus water content of all specimens. The distributed suction represents the suction gradient between the macro- and micro-pores. The higher the distributed suction, the greater the suction gradient between the macro- and micro-pores. As shown in Fig. 9, the distributed suction in the MX80 and Calcigel I specimens is higher than that in the Calcigel II and
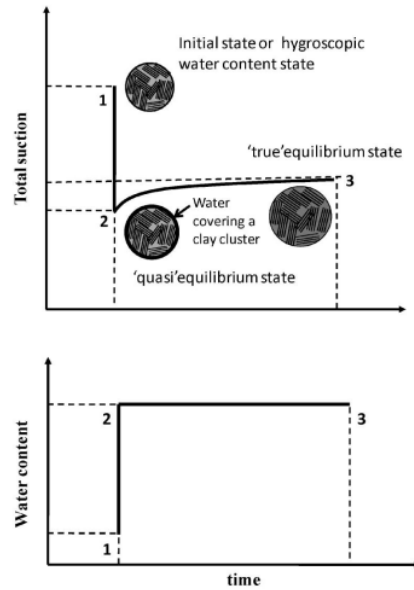

Fig. 8.   Effect of internal water distribution on total suction magnitude
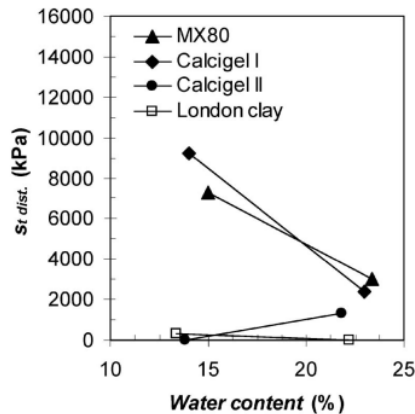

Fig. 9.   Distributed suction versus water content

London Clay specimens. The distributed suction is higher at lower water content indicating a higher suction gradient. Figure 9 only indicates the magnitude of the suction gradient that exists in the specimens. However, the state of the specimens relative to the standard state under hygroscopic conditions is not represented.

Pore-water distribution in the specimen is also influenced by the difference between the suction at hygroscopic water content and that at the equilibrium suction since the bentonite particles are already at equilibrium at hygroscopic water content before mixing. The ratio between the distributed suction and this difference represents the activity of the bentonite. Figure 10 shows the ratio between distributed suction and the
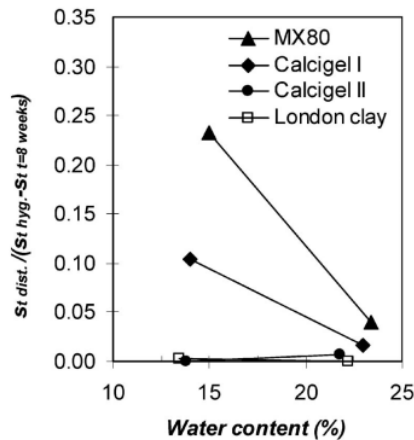
Fig. 10.   Normalized distributed suction versus water content

difference between the hygroscopic suction and the "equilibrium" suction versus water content of the specimen. The figure shows that MX80 is the most active material as compared to the other bentonites. In addition, Saiyouri et al. (2004) reported the difference of hydration mechanism between sodium bentonite (i.e., MX80) and calcium bentonite (i.e., FoCa7). At hygroscopic water content, the particles of materials used in the study were composed of 350 and 100 elementary layers for MX80 and FoCa7, respectively. The number of layers per particle decreases to 10 layers at the highest water content of the material at 1 kPa suction. These result in particle size changing with ratio of 35 for MX80 and 10 for FoCa7. This finding also shows that the sodium bentonite is more active than calcium bentonite.

CONCLUSIONS

This paper discusses results of an experimental study on the time-dependent changes in the total suction of three bentonites (i.e., MX80, Calcigel I, and Calcigel II) and London Clay observed from the measurement data via the chilled-mirror hygrometer technique. The results show that the true equilibrium state is significantly delayed for the MX80 and Calcigel I specimens. The total suction increases with time. The time to reach the true equilibrium state depends on the magnitude of total suction gradient between different levels of pores in the specimens and controlled by the characteristics of the bentonites. The total suction of the Calcigel II and London Clay specimens is not time dependent. The apparent changes in total suction of these soils are possibly due to the inaccuracy of the device used.

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FINAL GRADE

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GENERAL COMMENTS

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