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Total Suction Measurement of Unsaturated Artificial Gypsified Soil Using the Chilled-Mirror Hygrometer Technique

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ABSTRACT

Total suction is the sum of the matric and the osmotic suction of the soil. Various methods are used to measure the total suction of the soil. One of the methods is the chilled-mirror hygrometer technique. The chilled-mirror device employs a dew-point procedure, which is one of the most accurate and valuable methods for measuring total suction in the mid- to high- total suction range.

The effect of the percentage of gypsum on the total suction estimation for a gypsum-sand mixture has been investigated rarely in the past. In this paper, the total suction is determined for soil specimens containing different percentages of gypsum additive mixed with Silber sand. The soil specimens were prepared at different initial conditions of density and degree of saturation in order to investigate a wide range of physical soil properties. The test results showed that an incremental increase in the percentage of gypsum and degree of saturation of a soil mixture caused a reduction in the measured value of total suction, while an increase in the initial dry density caused the measured total suction to remain almost unchanged. No constant trend is observed with respect to the effect of the curing time on the measured total suction.

Key words: Total Suction, Chilled-Mirror Hygrometer, Gypsified Soil, Silber Sand, Curing Time, Relative Humidity, Temperature.

> قياس الامتصاص الكلي لتربة جبسية صناعية غير مشبعة باستخدام مقياس الرطوبة

> > الخلاصة

الامتصاص الكلي هو مجموع الامتصاص الشعري والامتصاص التتاضحي للطاقة الحرة للتربة هناك طرق مختلفة استخدمت لقياس الامتصاص الكلي للتربة . واحدة من هذه الطرق ه ي تقنية مرأة مقياس الرطوبة .جهاز مرأة مقياس الرطوبة يعمل بطريقة ذدى النقطة ، الذي تعتبر واحدة

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من اكثر الطرق دقة وقيمة لقياس الامتصاص الكلي في حدوده الوسطى والعليا . تأثير نسد بة الج بس على تخمين الامتصاص الكلي للمزيج الجبس – الرمل تم التحري عنها بصد ورة ندادرة في الماضد ي في هذا البحث تم ايجاد قيمة الامتصاص الكلي لعينات تربة تحدّ وي على نسد ب مختلفة من مضد اف الجبس الى رمل السيلبر . تم تحضير عينات التربة في حالات مختلفة من قيم الكثافةة ودرجة التشر بع لغرض تحري مدى واسع من الخصد ائص الفيزيائية للتربة . نذ ائج الفد وص اوضد حت ب ان الزيدة داري مقاض في انخفؤدي الله يزيج التربيع لمة التشاف ودرجبس المضبة الجي نسة ف التدريجي الامتصاص الكلي المقاس ، بينما الزيادة في مقدار الكثافة الجافة الاولية تؤدي الى عدم التعبير ر في ىة علت المعالجأثير وقبة لتت بالنسط ثابتم ملاحظ ة اي نمم تذلك لي . كاص الكلة الامتصقيم مقدار الامتصاص الكلي

INTRODUCTION

he state variable of greatest relevance to unsaturated soil mechanics is soil suction. Soil suction is a general term which may be used when referring to

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matric suction, osmotic suction or total suction. The application of unsaturated soil mechanics in engineering practice is closely related to the ability to measure soil suction. Soil suction (i.e. matric suction and total suction) has proved to be a difficult variable to measure. Soil suction can be quantified in terms of the relative humidity immediately adjacent to the water surface and is called "total suction". (Fredlund et al., 2012)[16].

Total, matric and osmotic suctions can be defined as follows (Aitchison, 1964[1]; Fredlund & Rahardjo, 1993[15] and Fredlund et al., 2012[16]):

Matric suction (or the capillary component of free energy): is the equivalent suction derived from the measurement of the partial pressure of water vapour in equilibrium with soil-water relative to the partial pressure of water vapour in equilibrium with a solution identical in composition with the soil-water. Osmotic suction (or the solute component of free energy): is the equivalent suction derived from the measurement of the partial pressure of water vapour in equilibrium with a solution identical in composition with the soil-water relative to the partial pressure of water vapour in equilibrium with free pure water. Total suction (or the free energy of soil-water): is the equivalent suction derived from the measurement of the partial pressure of water vapour in equilibrium with the soil-water relative to the partial pressure of water vapour in equilibrium with free pure water. The measurement of soil suction is crucial in understanding the engineering behaviour of unsaturated soils. In arid or semi-arid regions with low rainfall intensity the wetting process of subsurface layers is normally due to a slow rising of the ground water by capillary forces. In another sense, in these regions the soils may be so dry that the total suction may reach several hundreds of MPa (Schanz et al., 2010[28]).

Gypseous soils are soils that contain gypsum. Gypsum is a mineral salt containing hydrated calcium sulphate ($CaSO_4.2H_2O$) in a ratio of 20.9% calcium sulphate to water, plus 46.6% sulphur trioxide (SO₃) and 32.5% calcium oxide (CaO). It has a low specific gravity of 2.32 and this has a significant influence on the physical and mechanical properties of any soil containing large amounts of this material (Nashat, 1990[25]). Gypseous soils are considered one of the problematic (collapsible) types of soils and are fotly in arid deserts in an unsaturated

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state. In their natural condition they display low dry density and moisture content due to the presence of cementation bonds and a meta-stable open gypsum structure.

Many geotechnical reports and other studies estimate that gypseous soils cover large areas and are widely distributed throughout the world (see Figure (1)). The major areas of gypseous soil are in the Middle East (Iraq and Saudi Arabia), the southern parts of the former USSR (Siberia, Georgia and Transcaucasia), the northwest of Africa (Libya, Algeria), the south-western Europe (Spain) and in the drier parts of the USA (from California to Texas) (see Buringh, 1960[9];Van Alphan and Romero, 1971[30]; Barazanji, 1986[7];Nafie, 1989[24]; Nashat, 1990[25]; Al-Mufty, 1997[2]; Al-Obaidi, 2003[4]; Saleam, 2006[26]).

Previous work and the academic literature offer only rear information concerning the effect of the percentage of gypsum on the total suction estimation of a gypsum-sand mixture. The present paper focus on measurements and study the behaviour of total suction as it corresponds to changes in the initial condition of gypsum-sand mixtures using chilled-mirror psychomotor.



Figure (1) World distribution of gypseous soils (after FAO, 1990[17]).

TECHNIQUES, MATERIALS AND PROCEDURES The Chilled Mirror Hygrometer

The chilled-mirror hygrometer uses a dew-point measurement to determine the total suction in a soil. The measurement is performed under isothermal conditions in a sealed container.

The chilled-mirror technique allows the measurement of total suction in the mid-to high-suction range from 3000 kPa to 300,000 kPa (Gee et al., 1992[18]).

The chilled-mirror Water PotentiaMeter , WP4, from Decagon has been examined by several geotechnical researchers (Leong et al.,2003b[21]; Schanz et al., 2004[27]; Agus and Schanz,2005[5]; Thakur et al.,2005[31]; Campbell et al.,2007[10]; Cardoso et al.,2007[11]; Schanz et al.,2010[28]). The testing procedure has been set forth in the American Society Testing and Materials (2008) standard D 6836-02. The device is reliable and easy to use in total-suction measurement as high as 300,000 kPa. It is particularly useful for studying

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unsaturated soil behaviour in the residual soil suction range (Fredlund et al., 2012[16]). The chilled-mirror hygrometer used in this study was a water activity meter (Type 3TE, Decagon Devices, Inc., Pullman,WA). The device has been shown in Figure (2). 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 into the device by placing it in a small container (see Figure (2a)), which in turn is placed on a tray in a drawer, and the drawer pushed into the device. Vapour from the specimen fills the headspace above the container. Air in the chamber is circulated by the fan, which spreads the vapour evenly during the measurement. The condensation of water vapour is detected by the mirror and reflected to the photodetector cell. The temperature sensor measures the Temperature at which the condensation occurs. Using the temperature, the water activity or relative humidity is automatically calculated and shown on the display.



(a) (B) Figure (2) Chilled-mirror hygrometer device, a. photo, b. schematic diagram.

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 vapour. It can be written as follows (Fredlund et al., 2012[16]):

$$= - - \ln \left(\frac{1}{2} \right) \qquad \dots \quad (1)$$

Where ψ = soil suction or total suction (kPa), R= universal (molar) gas constant (i.e., 8.31432 J/(mol K)), T_K = absolute temperature (i.e., $T_K = (273.15+T \text{ (K)})$, T= temperature, ${}^{0}\text{C}, v_{w0}$ =specific volume of water or the inverse of the density of water (i.e., $1/\rho_w$ (m³/kg)), ρ_w = density of water (i.e. 998 (kg/m³) at temperature T=20 ${}^{0}\text{C}$), ω = molecular mass of water vapour (i.e., 18.016 kg/kmol), \overline{u}_v = partial pressure of pore water vapour (kPa), and \overline{u}_{v0} =saturation pressure of water vapour over a flat surface of pure water at the same temperature (kPa). The term = is called the relative humidity (RH) and is entered as a fraction (i.e. water activity) in equation (1). Figure (3) shows a plot of total suction against (RH) for three different temperatures computed using equation (1). According to Figure (3), the total

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suction is ($\psi = 0 \text{ kPa}$) when (RH =100 %) and increases when (RH is less than 100 %).

The device was firstly calibrated to check its accuracy for total suction measurements using a salt standard solution of (0.5 M KCl, 6.0 M NaCl, 8.57 M LiCl, and 13.41 M LiCl) giving water activity (a_w) or relative humidity (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 suction measured. The reading obtained for the standard salt solutions were found to vary with a range of (\pm 0.3%) RH with a response time of no longer than 3 minutes. The experimental tests were done in constant humidity and temperature (20 °C) laboratory, Chair of Foundation Engineering, Soil and Rock Mechanics, Ruhr-Universität Bochum, Germany.



Figure (3) Thermodynamic equilibrium between relative humidity and total suction, (after Fredlund et al., 2012[16]).

Materials used

The soil samples used in this study consist generally of two main materials; the first one is sand (S) namely (Silber sand) from Germany, and the second material

used in this study is pure Gypsum (G) (i.e. Calcium sulfate, $CaSo_4.2H_2O$). The source of gypsum materials is Alfa Aesar GmbH&Co.KG A Johanson Matthey Company, Germany. The chemical analysis of the Silber sand and gypsum is shown in Table (1).

Silber	SiO ₂	Fe ₂ O ₃	Al_2O_3	TiO ₂	Loss on	
sand ⁽¹⁾					Ignition	
	99%	0.05%	0.3%	0.08%	0.2%	
Gypsum ⁽²⁾	Assay	Iron	Lead	Fluoride	Loss on	Solubility in
					drying	water at 100 °C
	100.26%	<100ppm	<2ppm	<30ppm	20.55 %	2.22 gm/l

Table (1) Chemical analysis of Silber sand and gypsum.

(1) Source: Quarzwerke GmbH in Frechen.<u>www.Quazwerke.com</u>

(2) Source: Alfa Aesar GmbH&Co.KG A Johanson Matthey Company.<u>www.alfa.com</u>

To investigate the total suction of wide range gypsum content in soil samples, the pure gypsum was added in different percentage to Silber sand (at dry state and

by weight percent). The gypsum-sand mixture is (0, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 %) gypsum additives.

To investigate the effect of different initial conditions of soil specimen on the value of total suction, the gypsum-sand mixture of (70%G-30%S) was selected.

The physical properties of the Silber sand and gypsum-sand mixture of (70%G-30%S) are summarized in Table (2), the grain size distribution of Silber sand and (70%G-30%S) are shown in Figure (4).The effect of dry density, degree of saturation and curing time on the behavior of total suction of (70%G-30%S) soil mixture was experimentally measured.

Properties	Silber sand	(70%G-30%S)					
Specific gravity	2.65	2.40					
Compaction(proctor) test							
Max.dry density, (g/cm ³)	1.58	1.69					
Opt. moisture content, (%)	17.8	12.85					
Classification: (DIN18123-1990)							
d ₁₀ , d ₅₀ , d ₆₀ ,(mm)	0.14, 0.23, 0.25	0.025, 0.028, 0.03					
C _U	1.8	1.2					
Relative density: (DIN18126-1996)							
$\rho d_{min.}, \rho d_{max.}, (g/cm^3)$	1.399, 1.709	-					
e _{max.} , e _{min.}	0.901, 0.556	-					

Table (1) Physical test results of Silber sand and (70%G-30%S).



Figure (4) Grain size distribution according to DIN18123 (1990)[14].

Specimen preparation

The experimental work commenced by carefully mixing the gypsum material with Silber sand at air drying state (i.e. hygroscopic water content) to reach a target gypsum content of (0, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 %) gypsum additives.

To investigate the effect of gypsum content and initial dry density on the value of total suction, all soil mixture specimens were statically compacted in the device container to a target dry density of (1.3 g/cm^3) for gypsum content effect and of $(1.0, 1.3, 1.45 \text{ and } 1.6 \text{ g/cm}^3)$ for initial dry density effect. Then the soil specimens were covered by the containers fastened cap and nylon folia and left for 7 days at air drying (i.e. hygroscopic water content) then tested.

The average hygroscopic water content for all mixture specimens was about (0.25%). 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 (Schanz et al., 2010[28]). In this study, hygroscopic water content and normal water content was determined using (D2216-10) ASTM standard [6]. The heating temperature for water content determination was kept to (45 °C) to avoid lose of hydration water molecules of gypsum particles by high temperature (ASTM 2216-10[6]; Al-Mufty and Nashat, 2000[3]). However for the accuracy of water content calculations, the soil specimens were left for one week in the oven until no change in dry weight was observed. After oven drying, the total suction of the soil specimens were measured again to investigate the effect of hygroscopic water content and drying method (i.e. air drying and oven drying) on the total suction measurement even with small amount of difference in water content between the two methods.

To study the effect of degree of saturation on the total suction, soil mixture of (70%G-30%S) was prepared at (1.3 g/cm^3) dry density by mixing the specimen with deaired-distilled water to reach a target degree of saturation of (0, 5, 10, 20, 40, 60, and 100 %), then the soil specimens were covered by the containers fastened cap and nylon folia and left for 7 days in constant humidity and temperature atmosphere to be sure for good moisture distribution and to allow for building up the cementing bonds between soil particles. The actual degree of saturation of soil specimens was immediately determined after the suction measurements and it was found as (0, 4, 8, 18, 40, 55 and 100 %).

Moreover, several specimens of (70%G-30%S) soil mixture were prepared at (1.3 g/cm³) dry density and (3%) degree of saturation to investigate the effect of curing time on the soil suction. Total suction measurements were performed after the specimens reached ages of (0 hour, 0.5 hour, 1.0 hour, 3 hours, and 24 hours (1 day), 48 hours (2days), 72 hours (3days), 168 hours (7days), 240 hours (10days) and 336 hours (14days)).

Total suction measurement

For reliable and accurate total suction measurements, the user of chilled mirror hygrometer device must read and follow the operator's manual carefully to avoid any errors could be happened. The total suction measurements must be calibrated firstly by using special standard salt solutions that have a specific molality and water activity that is constant and accurately measurable (as mentioned above). In order to provide accurate readings, chilled mirror device should ideally be allowed a warm-up period of (15-60) minutes after turning it on. This allows the air inside the device to equilibrate to the temperature of its surroundings.

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The soil sample used must be homogenous and don't fill the sample cup more than half full, because of the overfilled cup will contaminate the sensors in the sensor chamber, see Figure (2).

When the prepared soil specimens reach to desired test age, the specimen 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 equal to zero. The measurement is started by locking the drawer in its position. Water activity (a_w) or relative humidity and temperature are shown in the display for no longer than 3 minutes. Chilled mirror reading cycle was repeated until two of consecutive reading have the same water activity (a_w) value or within (0.001) difference between each other. The variation in time, water activity (a_w) and temperature were recorded in order to investigate the change in total suction measurements until reach to equilibrium. Another point to be noted that the total time needed to reach to the final reading may vary depending on temperature differences between the chamber and your sample, and other properties of soil sample.

RESULTS AND DISCUSSIONS

Gypsum content effect

Figure (5) shows the relationship between the total suction values obtained from the water activity (or relative humidity) measurements using chilled-mirror hygrometer and the percentage of gypsum (CaSo₄.2H₂O) added as a dry weight percent of Silber sand. Generally, the test results showed that the increment of gypsum percentages in the soil mixture causes a slightly decrease in the value of measured total suction. This trend was clearly observed in oven drying specimens. This behavior can be attributed to an increase in water activity (a_w) occurred simultaneously with an increase in gypsum percentage. According to the chemical composition of gypsum (CaSo₄.2H₂O), the increase in gypsum percentage causes an increase in water molecules per unit mass of soil specimen. Moreover, because of uniformly distributed gypsum in gypsum-sand mixture and due to it's composes of very fine particles with large specific surface area, the slightly increased relative humidity leads to a decrease in the measured total suction.

For air dried (i.e. with hygroscopic moisture content) specimens, the results showed that the curve of total suction-gypsum percentage consists of three parts. The first part represents a slight decrease in measured total suction with increasing gypsum percentages till 40%. The second part shows that the suction value tends to increase till 60% gypsum percentage, after that sharply drops in total suction as evidently present in the third part of the curve in Figure (5) till it reach's to the lowest value at 100% gypsum percentage. Generally, the behaviour of total suction decreases with the increase of gypsum percentage in soil mixture can be attributed to the same reasons of the oven drying specimens above. The no discernible trend obtained from air drying specimens can be attributed to even the perceived difference in the moisture content of gypsum-sand mixture, which can affect the value of total suction measurements.

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Figure (5) Effect of gypsum contain on the total suction of gypsum-silber sand mixture.

Initial dry density effect

Figure (6) presents the relationship between the measured total suction and the initial dry density of (70%G-30%S) mixture. The results showed that for the soil specimens of the same water content but increasing compaction effort (i.e. dry density), the total suction almost remained unchanged. Similar observations were also made by (Gens et al., 1995[19]; Li, 1995[23]; Yahia-Aissa et al., 2000[32]; Leong et al., 2003[21]; Delage and Cui, 2008[13] and Schanz et al., 2010[28]).



Figure (6) Effect of initial dry density on the total suction of (70%G-30%S) mixture.

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Degree of saturation effect

The effect of degree of saturation on the value of total suction measurements is explained in Figure (7) and Table (3). The results showed that there is a sharply decrease in the measured total suction with correspond to the increase of degree of saturation of (70%G-30%S) mixture. According to Equation (1) and Figure (3) above, the relative humidity is the ratio of partial pressure to the saturation pressure of water vapour over a flat surface of pure water at the same temperature. Therefore the increase in the degree of saturation leads to simultaneously increase in relative humidity and of course the reduction in measured total suction will occur. An analysis was performed to investigate possible error due to variation of temperature during total suction measurements.

As shown in Figure (8), the curves presents that the relative humidity(RH) is a function of time, where (RH) values tend to increase with time and reach a constant value after several readings (Schanz et al., 2010[28]). The temperature in the chamber during measurements were almost constant for the same specimen and also for specimens with different degree of saturation except the specimen of (Sr=4%). These results confirm with the results obtained by (Leong et al., 2003[21]; Agus and Schanz 2005[5] and Schanz et al., 2010[28]).



Figure (7) Degree of saturation effect on the total suction of (70%G-30%S) mixture.

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Figure (8) Relative humidity and temperature versus time for (70%G-30%S) mixture.

Curing time effect

Figure (9) and Table (3) show total suction as a function of time for (70%G-30%S) mixture at initial dry density of (1.3 g/cm^3) and moisture content of (1.0 %). No constant trend is obtained with respect to the effect of curing time on the measured total suction. Such behavior was also observed by Schanz et al., 2010[28] when they used the same device with bentonites soil, see Figure (10).



Figure (9) Effect of curing time on the total suction of (70%G-30%S) mixture.

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Degree of	0	0.7	4	8	18	40	55	100	
(%)									
Total	219804	198016	4111	3279	1629	1493	1220	0	
suction,									
(kPa)									
Curing	0	0.5	1	3	24	48	72	168	336
time,									
(hrs.)									
Total	3557	3280	3557	6781	6216	3973	4391	5793	4252
suction,									
(kPa)									

Table (3) Summary of total suction according to degree of saturation and curing time of (70%G-30%S) mixture.

Agus and Schanz, 2005[5] and Schanz et al., 2010[28] reported that there is an error in the total measurements may be occurred due to two factors: the excessive condensation of water vapour onto the measuring system and the existence of isothermal nonequilibrium between the vapour space, the soil, and the sensor. The total suction measurement error due to temperature variations can be computed using the first derivative of equation (1) with respect to temperature (Eq.2)

$$\frac{d(\ln t)}{d(RH)} = \frac{1}{T} - \frac{1}{\ln (RH)} \dots$$
(2)

 P_o varies with T and the variation of P_o with T can be found, for instance, in Lide and Frederikse, 1994[22]. 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 equation (1) with respect to relative humidity (Eq.3) (Schanz et al., 2010[28]):

$$\frac{1}{d(RH)} = \frac{1}{(\frac{1}{\rho})RH} \qquad \dots (3)$$

Since d(RH) is essentially the inaccuracy of the device (i.e., $\pm 0.3\%\psi\psi$; Decagon Device Inc., 2002[12]), M_w is the molecular weight of water (i.e., 18.016 kg/kmol), the change in total suction (ds_t) encountered at 20 °C(or 293K) is about ± 405 kPa. Combining the finding obtained by Schanz et al., 2010, (as shown in Figure 10) and this study, the measurements error can be attributed to the inaccuracy of the device .The error plotted as a band of total suction versus time curve are presented in Figure (9 and 10).

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Figure (10) Total suction as a function of time for the Calcigel soil (after Schanz et al., 2010[28]).

Moreover, the non uniform trend of measured total suction as a function of curing time may be also because of the continuous chemical reaction between the calcium sulfate and pore water molecules (i.e. gypsum-anhydrite formation cycle). In addition to that, activity of the water and temperature are control on the form of calcium sulfate (Berner, 1971[8] and Scheirber 1986[29]). This reaction need sufficient time to reach to equilibrium under constant temperature and humidity because instability of gypsum. Horta, 1980 [20] showed the equilibrium curves for gypsum-anhydrite as a function of temperature and water vapor partial pressure for a total pressure of (1 atm.). Gypsum stability corresponds to high water vapour pressure and low temperatures. Anhydrite is stable at the opposite conditions, see Figure (11).



Figure (11) Equilibrium of gypsum-anhydrite as a function of water vapour partial pressure of a total pressure of 1 bar. (1) ,(2) and (3) gypsum-anhydrite rom different sources and for different relative humidity ($\epsilon = 100, 75, 50, \text{ and } 30 \%$) respectively, (as cited in Al-Mufty, 1997[2]).

CONCLUSIONS

This paper investigates the total suction measurements of artificially gypsified soil using chilled- mirror hygrometer technique. The experimental study was carried out on gypsum-silber sand mixtures at different initial states of gypsum percentages, initial dry density, degree of saturation and curing time. The discussion of the results obtained can be summarized as follow:

- 1. The chilled- mirror hygrometer technique is useful to predict the total suction value of gypseous soils, considering the device inaccuracy especially at low suction range.
- 2. The test results showed that the increment of gypsum percentages in the soil mixture causes a slightly decrease in the value of measured total suction.
- 3. For the soil specimens of the same water content but increasing compaction effort, the total suction almost remained unchanged.
- 4. The increase in the degree of saturation leads to simultaneously increase in relative humidity, and of course a reduction in measured total suction will occur.
- 5. No constant trend is obtained with respect to the effect of curing time on the measured total suction. Chemical reaction between the calcium sulfate and pore water molecules besides the inaccuracy of the device may cause unstable measurements of the total suction.

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