

## Relationship between Methylene Blue Value, Initial Soil Suction and Swell Percent of Expansive Soils

Erdal ÇOKÇA

*Department of Civil Engineering, Middle East Technical University,  
06531 Ankara-TURKEY  
e-mail: ecokca@metu.edu.tr*

Received 08.04.2001

### Abstract

The purpose of this study was to investigate the relationship between methylene blue value, initial soil suction and the swell percentage of expansive soil samples. In this study, commercially processed kaolinite and bentonite mineral clays were mixed in preselected proportions to obtain soil samples possessing a wide range of plasticities and different swell percentages. The methylene blue adsorption tests, suction measurements using a thermocouple psychrometer technique and standard free swell tests in oedometer cells (ASTM D4546-90) were conducted on samples of kaolinite-bentonite mixtures. The results indicated a linear relationship between methylene blue value and swell percent and also between initial soil suction (Log) and swell percent. The experimental relationship which directly relates the methylene blue value and initial soil suction to the swell percent is established, the reliability of the swell percent prediction method based on methylene blue value and initial soil suction (Log<sub>10</sub>) data is evaluated, and the coefficient of correlation (R<sup>2</sup>) of this relationship is 0.992.

**Key words:** Expansive soil, Methylene blue value, Psychrometer, Swell percent, Soil suction

### Introduction

Mineralogical composition and clay fraction content are main factors governing the swell percent of expansive clayey soils. However, mineralogical composition determination needs relatively sophisticated test equipment and elaborate test procedures which may not be generally available for practical purposes. Therefore, indirect simple methods were developed to avoid these difficulties.

Methylene blue adsorption (MBA) is a measure of the clay particle surface area, which is a function of clay type, and an indicator of the percentage for water adsorption by the clay, and hence, its percentage for swell when wetted. Surface area measurements are a direct reflection of clay mineralogy, but are an indirect reflection of expansivity (Fityus *et al.*, 2000; Fityus and Smith, 2000). Some authors believe that the cation exchange capacities of clay minerals can be measured by the adsorption of methylene blue

from aqueous solutions; Nevins and Wientritt (1967) found a close relationship between MBA and ammonium acetate analysis and cation exchange capacities. Hang and Brindley (1970) studied the cation exchange capacity and surface areas by MBA for pure smectite clays and found a linear relationship relative to these parameters. All of these studies indicate when smectite is prominent in a specimen as well as give insight into surface areas and cation exchange capacities of this group of minerals. MBA proved to be a valuable tool in evaluating smectite in soils. The MBA test can indicate smectite rapidly and without considerable expense.

Although not technically correct, soil suction can be described in layman's terms as a measure of a soil's affinity for water. In general, the drier the soil, the greater the soil suction is (Wray, 1984). The term "total suction"  $\tau$  represents the collective contributions of all mechanisms of soil-water retention and

can be defined as the free energy of soil water with respect to that of a pool of pure water located outside the soil at the same elevation. In other words, the free energy difference is the work done to draw the pure water into the soil by countering the friction resisting the flow of water and expanding the lattice of soil. Two components of total suction, namely, “matrix”  $\tau_m$  and “osmotic”  $\tau_s$  suctions, are recognised. Matrix suction reflects the forces emanating from the solid matrix of the soil for the retention of water. It includes the effect of the particle surface (adsorptive) forces and the capillary tensile forces related to spatial pore-size distribution in the soil. Osmotic suction represents the effect of dissolved substances in retaining soil water and results from the difference in solute concentrations at different points in the soil water (Edil and Motan, 1984). In soils containing very low salt concentrations in pore water, psychrometric readings are essentially indicative of the matrix suction (Edil and Motan, 1984). Volume changes in expansive soils are generally caused by moisture variations that cause changes in the matrix suction stress state variable  $\tau_m = (u_a - u_w)$ .

Recently psychrometers, which measure total suction, have received increased usage in measuring the suction of soil in the laboratory.

The purpose of this study was to investigate the relationship between the methylene blue value (MBV), initial soil suction and the swell percent of expansive soil samples. For this reason, all the other factors were eliminated or kept constant throughout the test program.

## Methylene Blue Adsorption Test

### Adsorption of methylene blue by clay minerals

Methylene blue is a large polar organic molecule which is adsorbed onto the negatively charged surfaces of clay minerals. The amount of methylene blue adsorbed by a given mass of clay depends on the relative concentration of negatively charged sites on the clay particle surfaces, and on the surface area of the clay per unit mass.

Because methylene blue molecules are preferentially adsorbed onto the negatively charged site which might otherwise attract cations, titration with methylene blue can also be considered to give a relative measure of the cation exchange capacity of a clay soil (Cokca and Birand, 1993).

### Methylene blue adsorption test method (see Figure 1)

The basic relationship in MBA analyses of clays in materials is that a ground powder is titrated with methylene blue dye, and a spot tested on filter paper until the spot of material absorbs no more dye as evidenced by a lighter blue ring around the darker blue ring of the spot tested.

After investigation, the following procedure was found to be the most convenient means of performing the test. About 30 g of soil (dry equivalent) is mixed (speed of mixer is 700 rpm) with 200 mL of distilled water. Once the mixture is homogeneous, a known amount of methylene blue at a concentration of 10 g/L is added to the soil suspension from a burette. The soil water-methylene blue mixture is then agitated (at 400 rpm throughout the test). After each addition of methylene blue to the soil suspension, a drop of the slurry is placed on a filter paper with a glass rod for visual examination.

Initially, a faint blue spot of solids is observed surrounded by a transparent ring of clear liquid that soon evaporates. As titration proceeds, the colour of the inner spot becomes progressively darker blue as the clay adsorbs more dye, but the boundary of the blue spot remains clearly marked and distinct. Eventually, the end point is reached when the outline boundary of the inner blue spot breaks down into a diffuse light blue-green halo, radiating outwards.

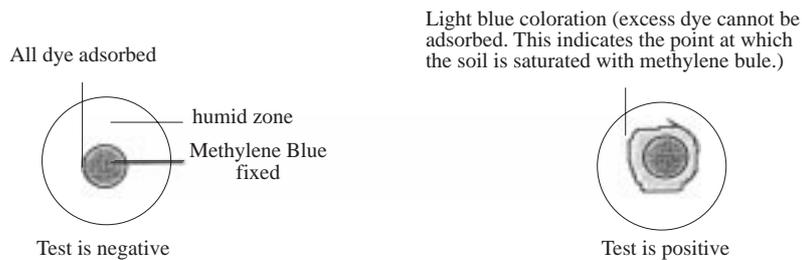
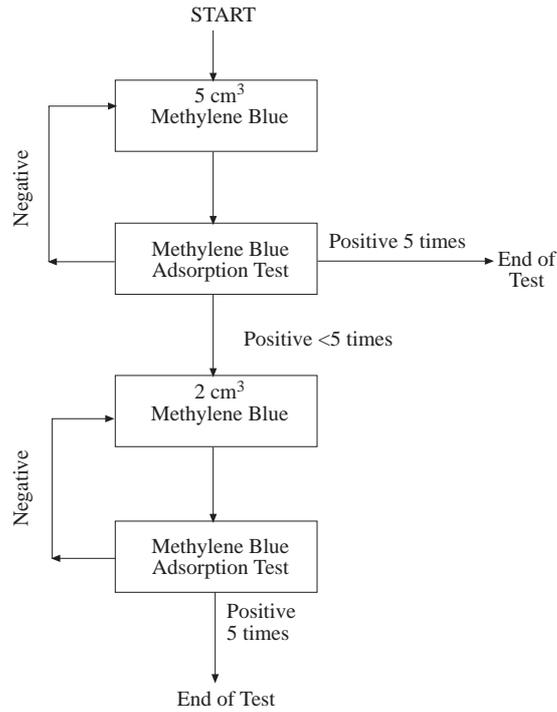
At the first appearance of the blue-green halo, titration is stopped and the drop test on filter paper repeated five times. If the halo persists, the end point has been reached; if not, titration is continued. Note the burette reading at this point and deduct the initial reading from it to give the volume of the methylene blue solution injected into the soil solution,  $V_{cc}$  (when learning the test, it is worthwhile to carry the additions of methylene blue solution several steps beyond the end point to compare MBA before and after the end point).

For 100 g of fine soil, the MBV of the soil is given by the following equation:

$$\text{MBV (g/100g)} = \frac{[V_{cc}(\text{mL}) \cdot 10(\text{g}) \cdot 100(\text{g})]}{[1000(\text{mL}) \cdot f'(\text{g})]} \quad (1)$$

where

$V_{cc}$  = volume of the methylene blue solution injected into the soil solution, mL



**Figure 1.** Schematic representation of methylene blue adsorption test.

$f'$  = dry weight of the specimen used, g, i.e., the specimen weight corrected for moisture loss at 105°C for 24 h.

**Soil Suction Test**

**Evaluation of thermocouple psychrometers**

Here soil suction is evaluated from measurements of relative humidity in soils determined with thermocouple psychrometers (according to Edil *et al.* (1981) their use is restricted to soil suctions greater than 100 kPa). Details of the procedure for measuring soil suction using psychrometers is presented in the literature (Snethen, 1980), but for the sake of completeness a summary is also given in this paper.

The thermocouple used in geotechnical engineering is a miniature thermocouple enclosed in protec-

tive casing made of ceramic or steel mesh, and it measures the relative humidity in the soil by a technique called Peltier cooling. By causing a small direct current of about 4 to 8 milliamperes to flow through the thermocouple junction for about 15 seconds in the correct direction, this junction will cool and water will condense on it when the dew point temperature is reached (Snethen 1980). The condensation of this water inhibits further cooling of the junction, and the voltage developed between the thermocouple and reference junction is measured by microvoltmeter.

Correction for the readings taken at temperatures other than 25°C was made by using the following equation:

$$E_{25} = E_T / (0.325 + 0.027 * T) \tag{2}$$

where

$E_{25}$  = the equivalent output at the calibration temperature of 25°C,

$E_T$  = the psychrometer voltage output, at T °C (in microvolts)

T = temperature in °C.

Before using the psychrometers in soils, the voltage outputs of the psychrometers are calibrated by tests with salt solutions.

### Suction tests using the thermocouple psychrometer method

**Equipment** The psychrometric technique has been used successfully for laboratory measurements. Thermocouple probes and readout apparatus are commercially available. In this study, the apparatus manufactured by Wescor Inc. (Wescor type HR-33T Dew point Microvoltmeter) of the United States (it is accurate in the range of pF 3.2-4.8) was used. A glass jar as a sample container, rubber stoppers, plastic mud, a thick cardboard box filled with plastic foam, a medicine dropper and NaCl solutions with different molalities of 0.3, 0.55 and 0.7 were used.

**Calibration** NaCl solutions with molalities of 0.3, 0.55 and 0.7 and having water potentials of -13.68, -25.1 and -32.10 bars respectively were used to calibrate the psychrometers. The psychrometer measurements on sodium chloride solutions reflect the osmotic (and the total) suction equivalent of the solution. Four jars were filled with NaCl solutions of the same molality and then the jars were closed with the rubber stoppers with psychrometer wires passing through. Finally, plastic mud was used to seal the openings between the psychrometer wire and rubber stopper in order to prevent air entrance. These jars were placed in a thick cardboard box filled with plastic foam in order to minimize the temperature variation of the sample container. After two or three hours, the psychrometer voltage output readings stabilized. Thus the vapor equilibrium was reached. After reaching equilibrium, the psychrometer voltage output and temperature readings were taken. The psychrometer voltage outputs  $E_T$  (in microvolts) were converted to the equivalent output at the calibration temperature of 25°C,  $E_{25}$ , using Equation (2).

The psychrometer output  $E_{25}$  is then plotted against water potentials (i.e. suction), and this procedure is repeated for the remaining two molalities to obtain the calibration line for the specific psychrometer (Figure 2). A typical example of a calibration

line for the thermocouple psychrometers used to collect the soil suction data in this study is

$$\tau = 2.3636 * E_{25} - 1.7188 \quad (3)$$

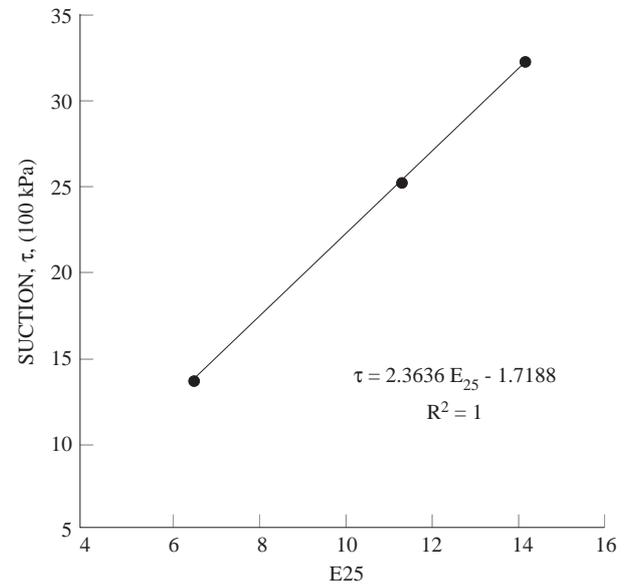


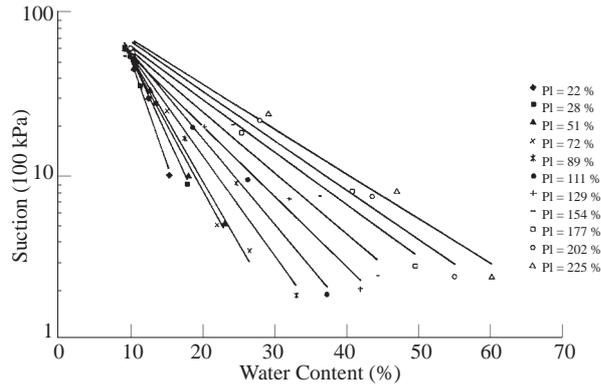
Figure 2. Calibration line for No. 36670 psychrometer.

**Test Procedure** From each clay mixture four disc-shaped specimens having a diameter of 36 mm and height of 15 mm were prepared at a dry density of 1.64 Mg/m<sup>3</sup> and a water content of 10%, using static compaction. After preparing four compacted samples, one of them was left at the initial water content level. The remaining three samples were wetted with varying amounts of water for varying lengths of time to establish a range of water content conditions. After this gradual wetting process, all four specimens were left to swell in the desiccator for 2 to 3 days to attain equilibrium water contents.

Then all four specimens were waxed. A hole was drilled on the waxed, covered sample so that the ceramic shield of psychrometer could be placed in the hole to measure the final equilibrium soil suction values. The sides of the psychrometer were isolated by plastic modelling clay to prevent any water content change. These four samples were placed in jars and the jars were closed with the rubber stoppers which had been affixed to the psychrometer wire. The jars were placed in a cardboard box filled with plastic foam packing material for better heat insulation and left to reach equilibrium. Usually it took 24 hours to reach equilibrium. Psychrometer voltage outputs and temperature readings are taken.

This procedure was repeated at least three times a day until the successive readings stabilise, meaning that vapor pressure equilibrium is reached.

**Data Reduction and Interpretation** The psychrometer output (microvolt) at the measured temperature is converted to an equivalent output using Equation (2). The psychrometer output is then converted to soil suction using the calibration line for the specific psychrometer. The data is then plotted versus water content on a semi-log plot to establish the log soil suction versus water content relationship (Figure 3), which is linear and has the form



**Figure 3.** Relationship between the suction and water content.

$$\text{Log}\tau = A - B * w \quad (4)$$

where  $\tau$  = soil suction without surcharge pressure

A = y-intercept of soil suction versus water content curve, i.e. the soil suction value (logarithmic scale) at zero moisture content.

B = slope of soil suction versus water content curve

w = water content

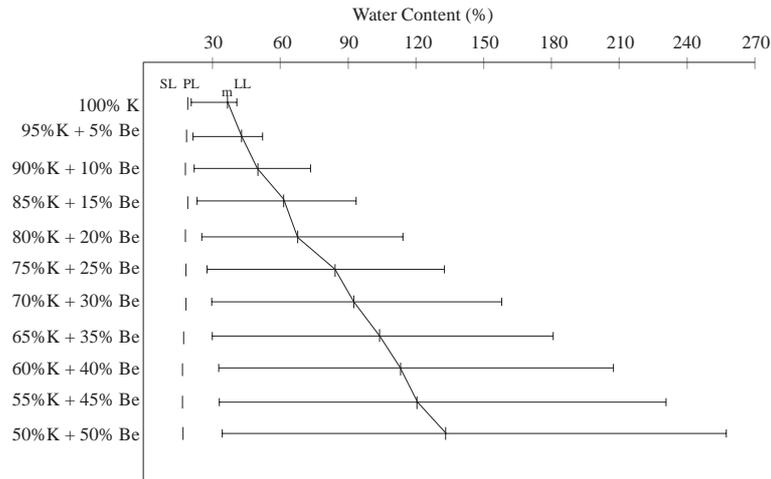
**Experimental Works**

**Material**

The chemical compositions of kaolinite and bentonite are given in Table 1. Commercially processed kaolinite and bentonite mineral clays were mixed in pre-selected proportions to obtain clays possessing a wide range of plasticities. The kaolinite and bentonite mixtures are shown in Table 2. The hydrometer tests were performed to find the proportion of clay in the soil mixtures. Particle size distribution curves show that as the bentonite content of the samples increase from 0 to 50%, the percentage finer than 0.002 mm

**Table 1.** Results of chemical analysis.

Chemical Composition (%)	Kaolinite	Bentonite
MgO	0.04	0.45
Al <sub>2</sub> O <sub>3</sub>	33.01	18.54
SiO <sub>2</sub>	49.65	59.75
CaO	0.35	3.07
Fe <sub>2</sub> O <sub>3</sub>	1.84	3.63
SO <sub>3</sub>	0.18	0.13
K <sub>2</sub> O	1.90	0.99
Na <sub>2</sub> O	0.11	2.25
TiO <sub>2</sub>	1.48	0.65
Loss on Ignition	11.10	9.87



**Figure 4.** Final water contents and consistency limits.

**Table 2.** Soil properties, methylene blue values, A, B values, initial soil suction, measured and estimated swell percent.

Sample* (%)	G <sub>s</sub>	Clay Content (%)	LL %	PL %	PI %	Soil Class**	MBV g/100g	A	B	Initial Soil Suction (kPa)	Measured Swell % (oedometer)	Predicted Swell %, (Eqn 6)
100% K	2.62	47.5	42	20	22	CL	2.93	2.909	0.1214	3746	23.26	16.79
95% K+ 5% Be	2.61	48.0	48	20	28	CL	4.13	2.640	0.0933	4109	26.15	28.08
90% K+ 10% Be	2.59	48.8	71	20	51	CH	5.20	2.473	0.0781	4111	40.41	45.49
85% K+ 15% Be	2.58	49.9	93	21	72	CH	6.40	2.479	0.0756	4440	59.00	58.17
80% K+ 20% Be	2.56	51.0	112	23	89	CH	7.47	2.332	0.0605	4640	71.09	71.68
75% K+ 25% Be	2.55	52.0	135	24	111	CH	8.67	2.265	0.0519	4944	85.04	85.57
70% K+ 30% Be	2.54	53.2	155	26	129	CH	9.73	2.159	0.0427	4890	103.63	103.85
65% K+ 35% Be	2.53	54.0	180	26	154	CH	10.93	2.132	0.0371	5295	118.24	116.29
60% K+ 40% Be	2.52	54.8	205	28	177	CH	12.53	2.101	0.0317	5653	132.74	136.54
55% K+ 45% Be	2.51	56.0	230	28	202	CH	13.60	2.112	0.0299	6069	143.18	147.63
50% K+ 50% Be	2.50	57.5	255	30	225	CH	14.67	2.099	0.0270	6339	168.57	161.19

\*K: Kaolinite, Be: Bentonite

\*\*Unified Soil Classification System

in the samples increase from 47.5% to 57.5% (Table 2). Soil properties are given in Table 2. The initial water content of the specimens is 10% ( $w_i = 10\%$ ). The final water contents of the specimens (at the end of swell percent test) and the consistency limits are shown in Figure 4.

### Methylene blue tests

Methylene blue tests were performed as described before. Temperature changes cause structural changes in the clay and methylene blue capacity; therefore, all the tests were performed under the same temperature conditions. Before performing the methylene blue test, soil specimens were dried in the oven for 24 hours (oven temperature was around 100°C) and methylene blue tests were performed at room temperature (i.e. 20-24°C).

Hills and Pettifer (1985) state that the particle size of the test sample affects the methylene blue test results; to get rid of this effect and to maintain uniformity of the test results, all the samples were sieved through a No. 40 sieve (400  $\mu\text{m}$ ) before performing the methylene blue test.

After using Equation 1 the methylene blue values of the samples were found. As the bentonite

percentage increases, the methylene blue value increases (Table 2).

### Soil suction tests

A linear relationship is assumed between log soil suction and water content (Figure 3), which has the form:  $\text{Log}_{10}\tau = A - B \cdot w$

Table 2 summarises the intercept A, which is the soil suction value (logarithmic scale) at zero moisture content, slope B of log soil suction versus water content behaviour. The intercept A and slope B of log soil suction versus water content behaviour decrease with increasing PI. The steep slope (large B) for a soil sample is indicative of a relatively low swell capability (e.g. sample with PI = 22%), while the flat slope (small B) for a soil sample is indicative of a relatively large swell capability (e.g. sample with PI = 225%).

Soil suction tests using thermocouple psychrometers were performed to obtain the soil suction versus water content relationship. The soil suction value corresponding to initial water content ( $w_i = 10\%$ ) was read from the log soil suction versus water content relationship.

**Oedometer method**

Standard free swell tests (ASTM D4546-90) were conducted on statically compacted samples of the kaolinite-bentonite mixtures (initial water content of 10% and dry density value of 1.64 Mg/m<sup>3</sup>) in oedometer cells. The swell percent of each specimen, possessing plasticity indices comparable to those obtained in the suction measurement tests, was directly measured. The sample was confined in the consolidation ring, which was placed in the oedometer under a small surcharge of about 1.94 kPa, and water was added to the sample and it was allowed to swell freely. Because of the high swell potential of bentonite, a new consolidation ring and free swell test apparatus were made for this test method. The height of the ring was 6.0 cm. All the laboratory free swell tests were laterally confined. As the sample swells the deflections of the dial gauge is recorded. At some point the sample has no further tendency to swell and the maximum deflection recorded is used for the calculation of the free swell. The percent of free swell can be expressed as

$$\text{Free swell (= swell percent)} = \Delta H / H * 100 \quad (5)$$

where  $\Delta H$  = change in initial height (H) of the specimen, and H = initial height of the specimen

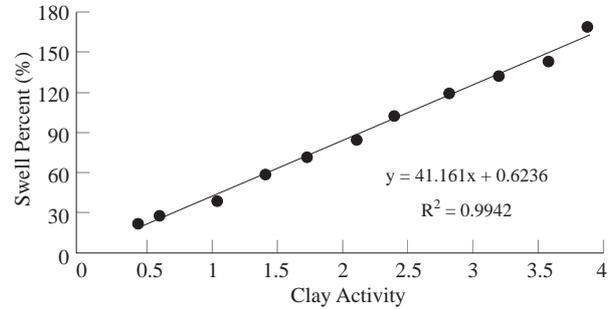
The time - swell percent relationships for varying percentages of bentonite generally follow a standard ‘S’ shape. Swell percent increases as plasticity index increases. It was observed that for the mixtures, the increases in swell with log time is slow initially, increase steeply, and then reaches an asymptotic value. The time required to reach an asymptotic value varies considerably, depending upon the percentage of bentonite clay. For the lowest percentage of bentonite, swell stops in 2000 minutes and to stop the swell for the highest percentage of bentonite more than 35,000 minutes is needed. As the plasticity index increases, the slope of the portion of the curve for the primary swell increases.

The results of swell percent tests performed under various bentonite contents are shown in Table 2.

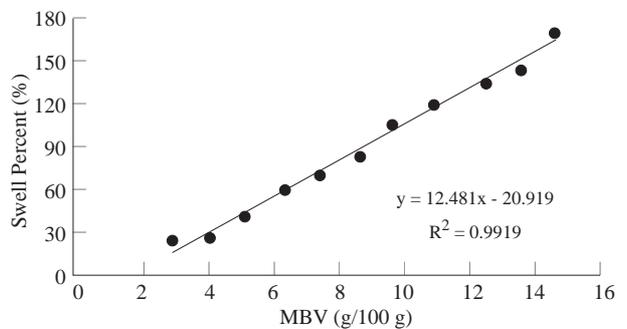
**Test Results**

The study confirmed the influence of mineralogical composition as a controlling factor governing the swell behaviour of expansive clayey soil. As shown in Figure 5, as clay activity increases swell percent

increases. The relationship between the methylene blue value and swell percent is shown in Figure 6; it can be seen from this figure that as the methylene blue value increases, the swell percent also increases. The results indicated a linear relationship between MBV and swell percent (Figure 6).

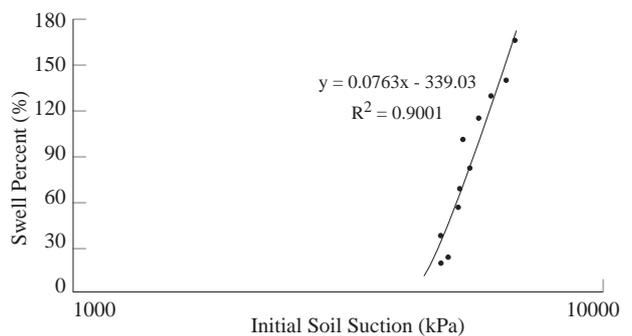


**Figure 5.** Relationship between the clay activity and swell percent.



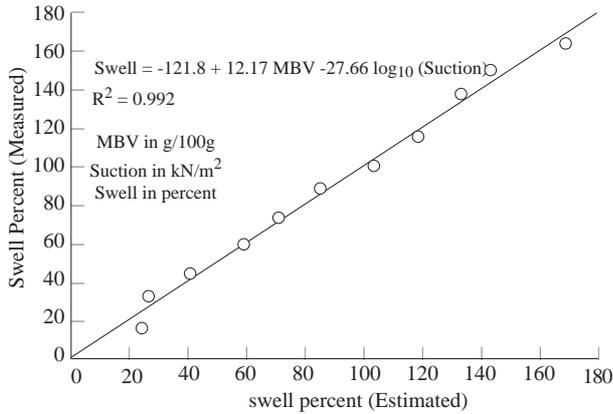
**Figure 6.** Relationship between the methylene blue value and swell percent.

The results indicated a strong relationship between initial soil suction (Log<sub>10</sub>) and swell percent (Figure 7).



**Figure 7.** Relationship between the initial soil suction and swell percent.

The regression analysis made on the MBV-initial soil suction-swell percent (oedometer test) data has revealed the following correlation (Figure 8):



**Figure 8.** Relationship between the estimated and measured swell percent.

$$\text{Swell (\%)} = -121.807 + (12.1696 * \text{MBV}) + [27.6579 * \text{Log}_{10} (\text{initial soil suction})] \quad (6)$$

As seen in Figure 8, there is a strong relationship between MBV, initial soil suction and the swell percent. As the initial soil suction and MBV increases, swell percent also increases for the given initial dry density and water content.

### Conclusions

In this study, the relationship between MBV, initial soil suction and the swell percent of expansive soil samples was investigated.

The swell percent predicted by Equation (6) is compared with the direct measurements from the oedometer free swell test, as shown in Table 2. The regression analysis made on the MBV-initial soil suction - swell percent (oedometer test) data has revealed the following correlation:

$$\text{Swell (\%)} = -121.807 + (12.1696 * \text{MBV}) + [27.6579 * \text{Log}_{10} (\text{initial soil suction})]$$

The reliability of swell percent prediction method based on methylene blue value and initial soil suction

(Log<sub>10</sub>) data is evaluated, the coefficient of correlation (R<sup>2</sup>) of this relationship is 0.992.

### Acknowledgements

The author wishes to acknowledge the contributions by Prof. Dr. Altay Birand and Prof. Dr. Orhan Erol. The author also acknowledges the assistance of the technical staff in the Geotechnical Engineering Laboratory of the Civil Engineering Department at the Middle East Technical University.

### Nomenclature

- A y-intercept of soil suction versus water content curve, i.e. the soil suction value (logarithmic scale) at zero moisture content
- B slope of the soil suction versus water content curve
- Be bentonite
- E<sub>25</sub> the equivalent output at the calibration temperature of 25°C
- E<sub>T</sub> the psychrometer voltage output, at T °C (in microvolts)
- f' dry weight of the specimen used, g, i.e., the specimen weight corrected for the moisture loss at 105°C for 24 h
- H initial height of the specimen
- Δ H change in initial height (H) of the specimen
- K kaolinite
- LL liquid limit (%)
- m final water content (%)
- MBV methylene blue value
- MBA methylene blue adsorption
- PI plasticity index (%)
- PL plastic limit (%)
- SL shrinkage limit (%)
- T temperature in °C
- τ total suction
- τ<sub>m</sub> matrix suction
- τ<sub>s</sub> osmotic suction
- u<sub>a</sub> pore air pressure
- u<sub>w</sub> pore water pressure
- V<sub>cc</sub> volume of the methylene blue solution injected into the soil solution, mL
- w water content (%)

## References

- ASTM, Standard Test Method for One-Dimensional Swell or Settlement Percent of Cohesive Soils, Annual Book of ASTM Standards, D4546-90, 04.08, 853-859, 1993.
- Cokca, E. and Birand, A., "Determination of Cation Exchange Capacity of Clayey Soils by the Methylene Blue Test", Geotechnical Testing Journal, GTJODJ, 16, 4, 518-524, 1993.
- Edil, T.B., Motan, S.E. and Toha, F.X., "Mechanical Behaviour and Testing Methods of Unsaturated Soils", American Society of Testing and Materials, Special Technical Publication, 114-129, 1981.
- Edil, T.B. and Motan, S.E. "Laboratory Evaluation of Soil Suction Components", Geotechnical Testing Journal, ASTM, 7, 4, 173-181, 1984.
- Fityus, S., Smith, D.W. "The Influence of Fabric and Mineralogy on the Expansivity of an Australian Residual Clay", Unsaturated Soils for Asia, Rahardjo and Toll (eds), 657-662, 2000.
- Fityus, S., Smith, D.W. and Jennar, A.M., "Surface Area Using Methylene Blue Adsorption as a Measure of Soil Expansivity", Geo2000 Conference (on CD), Australia, 2000.
- Hang, P.T. and Brindley, G.W., "Methylene Blue Absorption by Clay Minerals. Determination of Surface Areas and Cation Exchange Capacities (Clay-Organic Studies XVIII)", Clays and Clay Minerals, 18, 203-212, 1970.
- Hills, J.F. and Pettifer, G.S., "The Clay Mineral Content of Various Rock Types Compared with the Methylene Blue Value", J. of Chemical Technology Biotechnol. Chem.-Tech., 35A, 168-180, 1985.
- Nevins, M.J. and Weintritt, D.J., "Determination of Cation Exchange Capacity by Methylene Blue Adsorption", Ceramic Bulletin, 46, 6, 587-592, 1967.
- Snethen, D.R. "Characterization of Expansive Soils Using Soil Suction Data", 4<sup>th</sup> International Conference on Expansive Soils, 54-75, 1980.
- Wray, W.K. "The Principle of Soil Suction and its Geotechnical Engineering Applications", Fifth International Conference on Expansive Soils, Adelaide, South Australia, 114-118, 1984.