SUCTION MEASUREMENT OF BENTONITE USING FILTER PAPER TECHNIQUE

A Thesis Submitted in Partial Fulfilment of the Requirements for the Degree of

Master of Technology

In

Civil Engineering

(Geotechnical Engineering)

Siddharthan.S

DEPARTMENT OF CIVIL ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

MAY 2015

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CERTIFICATE

This is to certify that the project entitled "***Suction Measurement of Bentonite using Filter Paper Technique***" submitted by Mr. Siddharthan.S (Roll No. 213CE1037) in partial fulfilment of the requirements for the award of Master of Technology Degree in Civil Engineering at NIT Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in this report has not been submitted to any other university/institute for the award of any degree or diploma.

Place: Rourkela
Date:

Prof. Ramakrishna Bag
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Acknowledgement

I am deeply indebted to **Dr. Ramakrishna Bag**, Professor of Geotechnical Engineering specialization, my supervisor, for the motivation, guidance and patience throughout the research work.

I would like to thank Prof. **S. K. Sahu**, Head of Civil Engineering Department, National Institute of Technology, Rourkela, for his valuable suggestions during other review meeting and necessary facilities for the research work. I am also thankful to all the faculty members of the Civil Engineering Department, who have directly or indirectly helped me during the project work. I would like to extend my gratefulness to the Geotechnical Laboratory in charge and labours for their help.

I sincerely thank to our Director Prof. **S. K. Sarangi** and all the authorities of the institute for providing nice academic environment and other facilities in the NIT campus.

Finally, I would like to thank my family and friends especially Gurumoorthi and Siva Sankari for their unwavering support, help and invariable source of motivation.

SIDDHARTHAN.S

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Abstract

Bentonite is used as buffer material in nuclear waste disposal for which its characteristics should be studied. Soil - water characteristics curve (SWCC) is important to understand the unsaturated soil behaviour. The SWCC provides an understanding between the mass of water in a soil and the energy state of the water phase. Curve between soil suction and water content or degree of saturation gives soil-water characteristic curve. In order determine the relationship between suction and water content of unsaturated soil, filter paper technique can be used. The method is simple, economical and provides result with reasonable accuracy. It is an indirect suction measurement technique. Contact and Non – contact methods of filter paper technique determines the total and matric suction values, respectively. In the current study, bentonite was obtained from Bikaner, Rajasthan. Suction versus water content SWCC was established for the specimens of different dry densities and results are calculated using ASTM correlations. Experimentally obtained suction versus water content relationships were compared with that of the results obtained from Fredlund and Xing (1994) and van Genutchen (1980) models. The study revealed that with the increase in water content, suction value decreases but shows no pattern with dry density. Theoretical models were used to find suction values and compared with experimental results whereas van Genutchen model gives more accurate results than that of Fredlund& Xing model.

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CHAPTER 1

INTRODUCTION

Bentonite has many applications and the significant ones are for drilling mud, sand binder (foundry-sand bond, iron ore pelletizer), purifier, absorbent and as a barrier to groundwater. Most importantly, it has been used in nuclear waste management. Radioactive wastes which are divided as high-level and low-level radioactive wastes are created in nuclear power station. To keep them disengaged from human life, it has to be stored somewhere not near to the area where human exists with a system of safety barriers. SKB – Swedish Nuclear Fuel and Waste Management Co, found a safe way for the disposal. It starts with the interim storage where used nuclear fuel kept in water basins which cool the fuel and protect the environment from radiation. Then that fuel will be encapsulated in copper containers which are also known as the canisters. The radioactive and hazardous waste containing canisters are carefully placed several hundred metres below the normal ground level in intact with the host rock, encased by the safety barriers. The copper canisters will be placed in the disposal holes and embedded in a bentonite-clay buffer. Broadly the barrier materials are classified as the buffer material and the backfilling material. In short, the material that remains intact with the waste container is named as the buffer, and the one which is used to fasten the galleries over the waste containers are the backfilling material.

Fully impermeable copper canister contains the used fuel. The bentonite buffer protects the canister against rock movements and corrosive attack of any other substance present around the disposed area. If leakage occurs in a canister, the bentonite buffer and undamaged parts of the canister should prevent water or any other materials from penetrating into the canister. The buffer will also prevent the leakage of radioactive substances from the canister. The thickness of the bentonite buffer will be around 20 to 30 cm.

Coming to bentonite structure, in an ideal crystal under equilibrium conditions, the positive and negative charges would cancel out each other. But, isomorphous substitution and interrupting the regularity of structures can give rise to a net negative charge of clay particles. In order to counter that negative charge, the clay particles evoke positively charged ions from salts in their pore water that are known to be exchangeable ions. The three oxygen atoms positioned at the base of each tetrahedron are commonly shared by adjacent tetrahedron. Each silicon atom with a positive valence of 4 is associated with four oxygen atoms with a total negative valence of 8. However, each oxygen atom at the base of the tetrahedron is linked to two silicon atoms thereby counterbalancing one negative valence charge which is on the top layer of each tetrahedron. When the sheets of silica are piled up over the octahedral sheets, the oxygen atoms replace the hydroxyls to fulfil their valence bonds. Structure of montmorillonite has been shown in Figure 1.

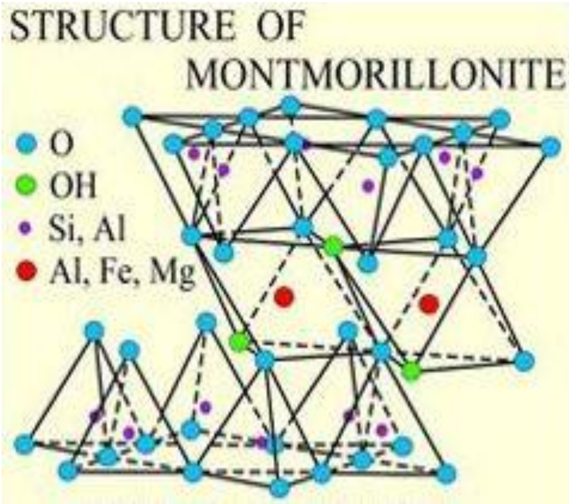

Fig.1. Structure of Smectite clay

1.1 Soil Suction

In unsaturated soil, pore pressures at depths below the water table are derived from weight of the water lying above the given elevation. The pore pressure by nature has a positive value that can be measured using a piezometer using porous filter thereby making intimate contact with water in the soil. Soil above the water table would be fully dry if the water voids of soil were subjected only to gravitational force.

The forces acting between the soil particles and water are molecular and physico-chemical forces and capillary forces resulting in moving the water into void spaces or to be held there without drainage. The cohesive force that the soil exerts on the water is termed as soil suction.

1.2 Components

- Matric suction
- Osmotic suction

Matric suction:

It is the difference between the pore-air pressure and pore-water pressure. It does not cause liquid or vapour flow.

Osmotic Suction:

It is related to the salt concentration of the free pore-water of a soil. Difference in salt concentrations at different points in a soil can cause movement of salts due to concentration gradient.

CHAPTER 2

LITERATURE REVIEW

2.1 Suction

2.1.1Soil Suction Concept

Total suction is the combination of matric suction and osmotic suction. Matric suction emerges due to texture, capillarity and adsorptive forces of soil whereas osmotic suction comes due to dissolved salts in the soil. The relation is

$$h_t = h_m + h_p$$

where h_t is total suction (kPa) h_m is matric suction (kPa) and h_p is osmotic suction (kPa). Kelvin's equation which is from ideal gas law is used to calculate total suction

$$h_t = \left(\frac{RT}{V}\right) * ln\frac{P}{P_o}$$

where R - universal gas constant, T - absolute temperature, V -molecular volume of water and P / P_o - relative humidity.

Matric suction is calculated using pressure membrane devices. Across the porous plate, difference between applied air pressure and water pressure gives the matric suction value.

$$h_m = -(u_a - u_w)$$

where u_a - applied air pressure and u_w - water pressure at atmospheric condition.

Osmotic suction is calculated using the relationship between osmotic suction and osmotic coefficients. Osmotic coefficients are promptly available in literature for different salt solutions. It can also be calculated from the following relation

$$\emptyset = \left(\frac{\rho_w}{vmw}\right) * ln\frac{P}{P_o}$$

where Φ is osmotic coefficient, v is the number of ions from one molecule of salt, m is molality is molecular mass of water and ρ_w is density of water.

2.1.2 Soil-water characteristic curve (SWCC)

The SWCC provides an understanding between the mass of water in a soil and the energy state of the water phase. Curve between soil suction and water content or degree of saturation gives soil-water characteristic curve.

2.2 Measurement of Suction:

Measurement of suction can be done by both direct and indirect methods which are given below. By measuring the negative pore water pressure, suction can be obtained. Tensiometer and Axis translation technique are used to measure the suction **directly**. Using electrical resistance sensor and thermal conductivity suction sensors, matric suction can be measured **indirectly**. Filter paper technique, Psychrometer technique, capacitance sensor, chilled-mirror hygrometer are some of the methods to measure total suction **indirectly**.

2.3 Comparison of other methods:

Psychrometer can measure the suction accurately only in range of 100 to 8000 kPa. In case of thermal conductivity sensors, the range is 10 to 1500 kPa.

Haghighi et al. (2012) and Yahia-Aissa et al. (2000) using different techniques, reported comparable results. In their study, the methods used are Insertion Tensiometer for matric suction by direct measurement and Pressure Membrane Extractor for matric suction measurement by axis translation technique and a Dew-point Potentiometer (WP4) for total suction measurements using relative humidity concept.

The outcomes obtained are distinctive in matric suction measurement using Tensiometer and Extractor techniques. For silty soil samples of 30% water content, matric suction values determined using Extractor and Tensiometer techniques are 1430 kPa and 150 kPa respectively. Value got from Tensiometer should have been below atmospheric pressure but it is not. Value measured exceeded the maximum value for that tensiometer due to the formation of bubbles and provided inaccurate measurements. So, it is not reliable to use Tensiometer technique for suction measurement in this range.

Only if an appropriate filter paper calibration curve is used and enough time given to reach equilibrium and also without any temperature change, matric suction obtained by these techniques would be similar.

Fattah et al. (2012) collected different samples from Baghdad city – al-Rasafa region and measured soil suction. Those specimens found to have different properties as prepared at varying degrees of saturation. For each specimen, total and matric suction were measured by the filter paper method at varying degrees of saturation going from 50% to 100% and the soil properties and suction are correlated. It was thus concluded that the suction increases with decrease in degree of saturation. And also same with soil shear strength. Suction value decreases with the increase in clay content and it decreases abruptly when clay content exceeds 80 %.

Guan & Fredlund (1997) conducted laboratory tests for measuring matric suction of fine silt and Regina clay using filter paper, null-pressure plate, high suction probe, and thermal conductivity sensor. The results obtained using those methods was in reasonable agreement with the measured suction using the suction probe at relatively high degree of saturation.

Petry & Bryant (2002) showed that total suction values obtained from WP4 chilled mirror device are generally somewhat higher than filter paper method values and those differences are due to the difference in equilibration time in both methods.

Navaneethan et al. (2005) performed suction measurements on four different clays using pressure plate, triaxial cell (measurement of positive pore water pressure after undrained loading) and filter paper techniques. It concluded that the results obtained from filter paper method are highly dependent on the calibration curve used.

Patrick et al. (2007) showed differences and scatter between the total suction results from filter paper and chilled-mirror device. They reported that the possible sources of these discrepancies are (i) errors in chilled-mirror technique measurements because of incompletion in equilibration time in the sealed test chamber of the device and (ii) errors in estimated filter paper values due to natural variations of the zero-water content intercept in the log total suction versus water content relationship.

2.4 Errors in measurement:

Agus and Schanz (2005) reported that while measuring matric suction, sufficient accuracy can be achieved but with total suction, following errors will be there.

1. In built error in the accuracy of equipment during manufacture. This type of error is difficult to avoid due to its presence in all sorts of sensors.

2. Temperature gradient between soil and vapour space causes error and this is more common in filter paper technique and psychrometer.
3. Condensation of water vapour cause fluctuations in temperature and relative humidity and again error occurs.
4. Error due to improper manual handling of sensors and variation in the procedures followed by the operator and occurs in case of filter paper technique.

2.5 Theoretical models to predict soil-water characteristic curve

Other than experiments, there are theoretical models to predict the soil-water characteristic curve. Those models have 2 or 3 parameters which govern the characteristic curve. Some of the models are explained below.

2.5.1 Van Genutchen model

van Genutchen made numerical model to find the hydraulic conductivity of unsaturated soils by using soil – water retention curve for which an equation has been formed. That equation was modified from Maulem's and Burdin's model. Modified equation is

$$\boldsymbol{\theta = \theta r + \frac{\theta s - \theta r}{1 + (\alpha h)^{(n)^{m}}}}$$

where **m** = 1-1/n and

α and n are independent parameters

ϴs – saturated water content

ϴr - residual water content

Of these four parameters, saturated water content can be obtained easily and residual water content can also be obtained by getting the water content in dry sample. Other parameters have to be arranged so that we get the characteristic curve and these parameters will be different for different soil. Graph is shown in figure 2 with parameter values of **α=0.005 and n=2.**

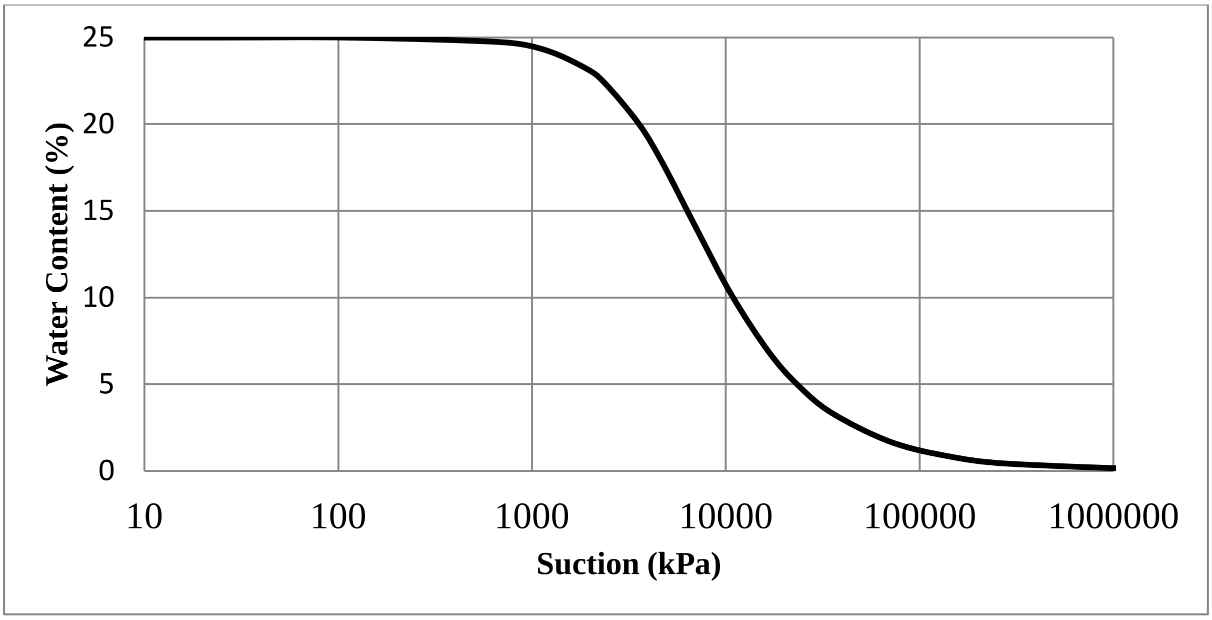

Fig.2. Variation of suction with water content by van Genutchen model

2.5.2 Fredlund and Xing model

A general equation for the all the soil water characteristic curve is proposed by Fredlund and Xing (1994). The equation is based on the assumption that the shape of soil – water characteristic curve depend on the pore size distribution of the soil. The equation provides a good fit for sand, silt and clay soils over the entire suction range from 0 to 10^6kPa. A mathematical equation is described and a best fit procedure is outlined to obtain the parameters for the equation. And is equation is

$$\boldsymbol{\theta} = \boldsymbol{\theta s} * \{\frac{\mathbf{1}}{[\boldsymbol{ln}(\boldsymbol{e} + \left(\frac{\Psi}{\alpha}\right)^{\boldsymbol{n}}]}\}^{\boldsymbol{m}}$$

where the parameters are same as The SWCC provides an understanding between the mass of water in a soil and the energy state of the water phase. Curve between soil suction and water content or degree of saturation gives soil-water characteristic curve. van Genutchen model except fixing the parameter values will be different. Graph has been drawn with a set of parameter values which will vary for other types of soil and it is shown in figure 3.

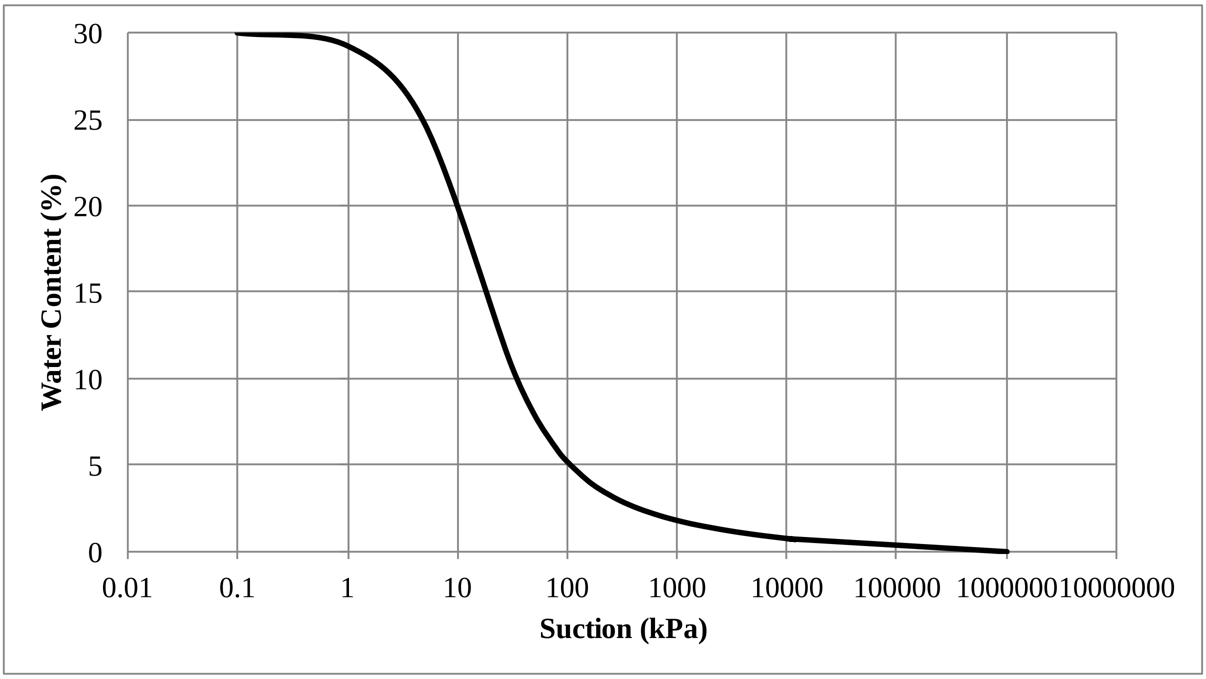

Fig.3. Variation of suction with water content by Fredlund and Xing model

2.6 Objective of the study

Review of past studies has revealed that much research had been done all over the world on the bentonite and correlations were made between suction with soil properties like shear strength, consistency limits and degree of saturation but not on the Indian bentonite. So, the main objective of this study is to find out the suction parameters of Indian Bentonite.

CHAPTER 3

MATERIAL CHARACTERISTICS AND METHODS

3.1 General

The aim of this study is to investigate the suction parameter of bentonite and suction with water content relationship. This chapter explains the methodology and materials used to achieve the objectives. Bentonite was collected from Bikaner, Rajasthan. Procedure for sample preparation, sampling and testing techniques are reported.

3.2 Method

3.2.1 Atterberg Limits

Atterberg limit tests were conducted to study the plasticity property of the soils. The liquid limit and plastic limit are the water contents at which the soils exhibit both liquid and plastic property, respectively. The liquid and plastic limits tests were conducted according to IS: 2720-Part 5 and 6 (1985) and the results are presented in the table1.The liquid limit was determined using the fall-cone method and the plastic limit was determined by rolling 3 mm diameter threads of the soils until they began to crumble. The difference between these liquid limit and plastic limit is known as the plasticity index, which is generally used to characterize the plastic nature of soils.

Table 1. Atterberg limits of Bentonite

Limits and Indices	Values
Liquid Limit	140 %
Plastic Limit	61 %
Shrinkage Limit	40 %
Plasticity Index	79 %

3.2.2 Specific Gravity

Specific gravity is the ratio of density or specific weight of the particles to the density or unit weight of water. The specific gravity of bentonite was determined using pycnometer method as per IS: 2720-Part 3 (1980) and it is found to be **2.67**.

3.2.3 Particle Size Distribution

Particle size distribution of bentonite was determined using hydrometer method in accordance with IS: 2720- part 4 (1975).Bentonite sieved through 75µm sieve size was collected carefully and used for particle size analysis and the analysis was performed using Hydrometer method. Value of D10 (Diameter of particle corresponding to the 10% finer), D30 (Diameter of particle corresponding to the 30% finer) and D60 (Diameter of particle corresponding to the 60% finer) are to be obtained. But since particles are very fine, it is not possible to get those values and the distribution is shown in figure 4. Around 69% of particles are clay sized.

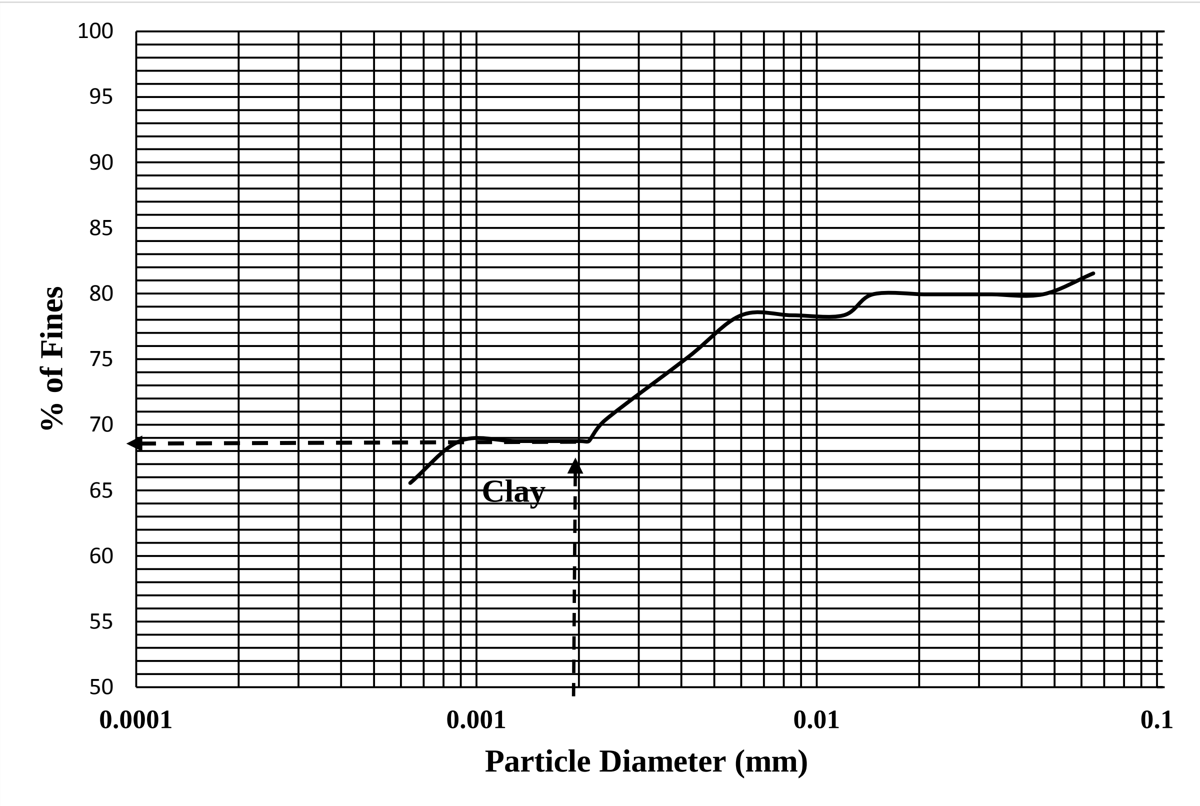

Fig.4. Particle Size Distribution of Bentonite

3.2.4 XRD Analysis

The mineral composition of bentonite was determined by X-ray diffraction method. According to Bragg's law, the XRD identifies the minerals based on the relationship between the angle of incidence of the X-rays, θ, to the *c*-axis spacing, *d*. A Philips automated powder diffractometer was used for XRD analysis in this study.

1.5 g of fie grained sample is kept in oven drying for 2 hours and allowed to cool in room temperature. Then, sample is filled in the sample holder of diffractometer and the XRD pattern is obtained by scanning over angle range of 20° to 100°, 2θ at 0.25°/min. In the step mode, a 0.05° - 2θ step for 2 s is given. Results are analysed using Xpert High Score software and mineral composition has been found. Quartz, Montmorillonite, Muscovite and Calcite are the minerals found. Result is shown in Figure 5.

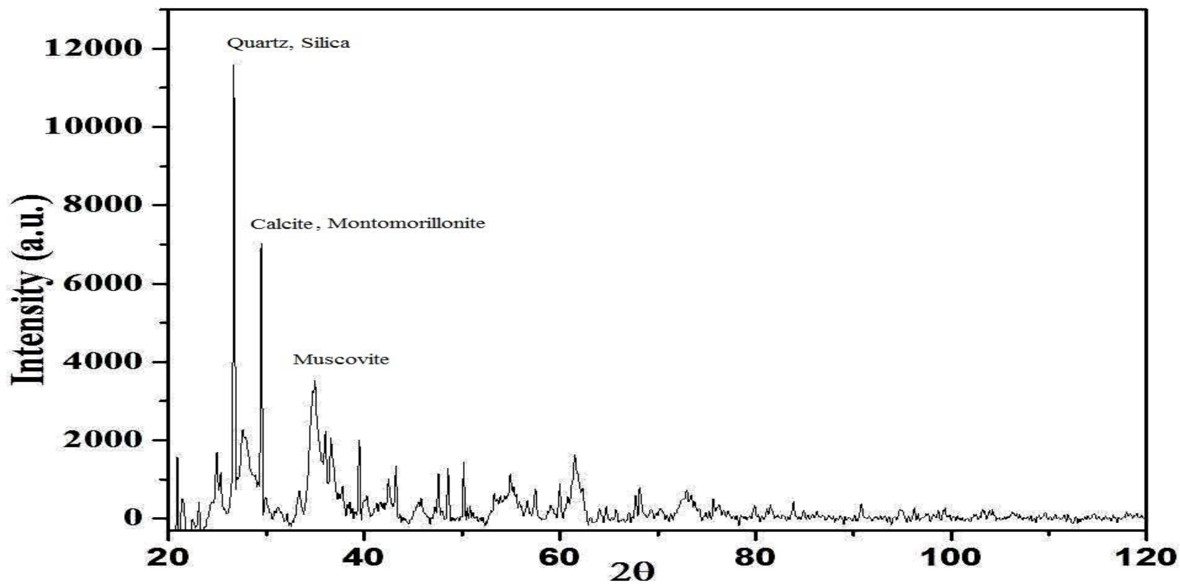

Fig.5. XRD analysis of Bentonite

3.2.5 BET Analysis

Specific surface area being the significant physical parameter has a great impact on the quality and utility of solid phase samples. Gas adsorption analysis is the widely used method for measurement of specific surface area. This method involves exposure of gas to the solid sample under various environmental conditions thereby measuring volume of sample. The Brunauer, Emmett and Teller (BET) technique is commonly employed tool to determine the surface area of powder sample. Here, nitrogen gas is used as probe that is exposed on the solid material under standard conditions. Thus, surface area of the sample can be measured from the monolayer adsorbed using the prior knowledge of cross sectional area of probe being used. Activation of sample must be done to ensure that no air or gas has been adsorbed on the solid particle before evaluation which may affect the sample quality. Activation can be done by heating the sample under vacuum conditions. Specific Surface Area has been determined by BET analyser and value is found to be **79.23 m²/g**.

3.2.6Cation Exchange Capacity (CEC)

Cation Exchange Capacity (CEC) is defined as the amount of exchangeable ions that a soil can hold at a given pH value. The Cation Exchange Capacity (CEC) of the soil sample and soil lime mixture was found as per ASTM D7503 – 10 method. The Nitrogen concentration was determined by spectrophotometer as per modified Parsons et al. (1984). The nitrogen concentration was determined by the graph obtained by the absorbance of the standard solution of known concentration. After Nitrogen Concentration is known, the CEC of the sample is calculated by the equation

$$CEC\ \left(\frac{cmol}{kg}\right) = \frac{N \times 1 \times 0.25}{140 \times mass\ of\ soil\ taken} \times 1000$$

where N = nitrogen concentration in mg/L.

And the CEC value of bentonite is calculated as **54.71 meq/100 g.**

3.2.7 Filter paper technique:

Filter paper method is nowadays being used for suction measurement due to its advantages like method is simple, economical and reasonable accuracy (Bicalho et al. 2007and Oliveira &Marinho 2006). Other methods are having one or two of these advantages but not all. In this method, filter paper has to attain equilibrium with the specimen by either through liquid or vapour. Water content of filter paper is determined after it reached its equilibrium time normally 15 days. From calibration curve given by ASTM, suction value is found. This method can be used to measure suctions from 10 to 30000 kPa.

The experiments should be performed with great care. There are two procedures for measuring suction by this technique. **Non-contact** filter paper procedure is to measure total suction. Filter paper is suspended above the specimen and allowed for vapour transfer between filter paper and soil until vapour pressure equilibrium is achieved. **Contact** filter paper procedure is used to measure either the total or matric suction depending on the degree of contact between the soil and the filter paper. In this technique, water content of filter paper increases due to a flow of water in liquid form until equilibrium is established. Thus, it is necessary to form an adequate contact between the filter paper and the soil.

According to the American society for Testing and Materials (ASTM), the filter paper used must be ash-free quantitative Type II filter paper, for example, Whatman No. 42, Fisher brand 9-790A or Schleicher and Schuell No. 589 White Ribbon. Diameter depends on the specimen size. To prevent organism growth or biological decomposition on the filter paper, it can be treated by dipping each paper in a 2 % concentration of formaldehyde. Biological degradation is necessary when filter papers are subjected to a moist, humid environment for greater than 14 days.

Calibration curve recommended by ASTM has been used to get the matric and total suction values. ASTM D 5298 gives the standard test method procedure for filter paper method. Calibration curve contains both wetting and drying curve but because of hysteresis, wetting curve can be used. Or else, correlations (equation from calibration curve) given by ASTM can also be used for calculation of suction values. Calibration equation is given below.

$$\boldsymbol{log\varphi = 5.327 - 0.0779w_{fp}}$$

where ψ – suction value

and w_{fp} – water content in filter paper

Specimens kept in container with filter paper inside desiccator is shown in fig 6 and 7.

Fig.6. Filter paper placed in - between two halves of sample

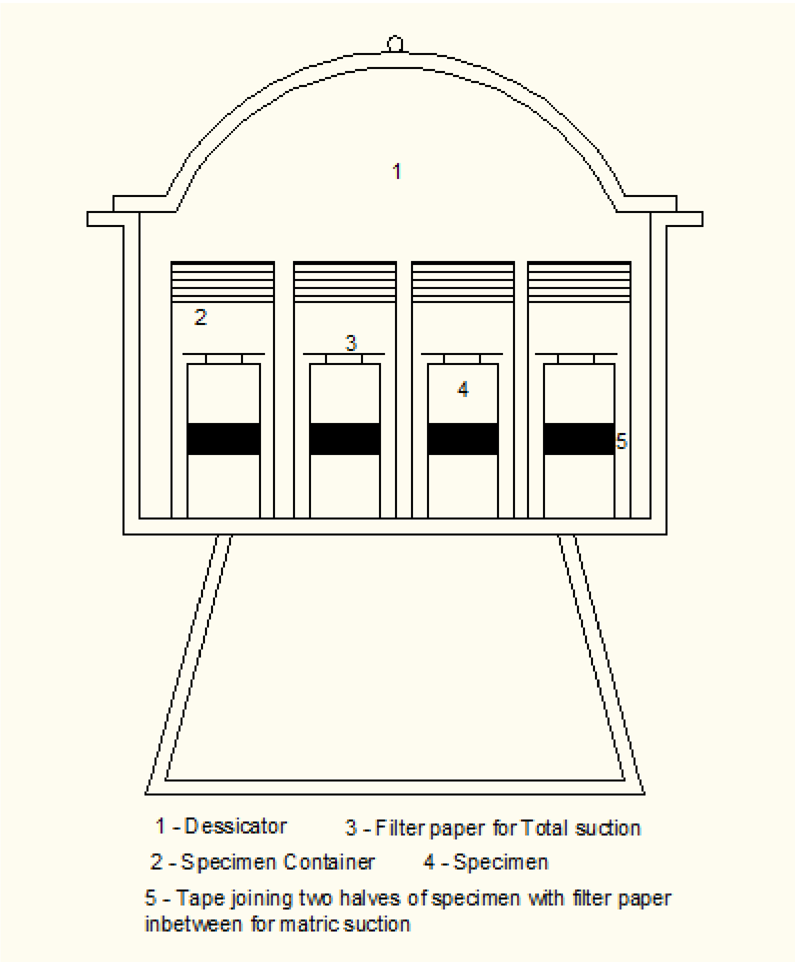

Fig.7. Sketch of Desiccator with specimens

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

Experiments have been conducted to find the suction values both total and matric suction using filter paper technique and results are discussed in this chapter. Results are calculated by using the given ASTM correlations.

4.2 Soil Water Characteristic Curve

A soil – water characteristic curve can be used to estimate various parameters used to describe unsaturated soil behaviour. Behaviour of unsaturated soil is highly dependent on the magnitude of soil suction, which is influenced by water content for a given soil. The soil-water characteristic curve (SWCC) represents the ability of a soil to retain water at over a range of suctions. The SWCC defines the relationship between the amount of water in the soil pores, which is generally quantified in terms of gravimetric water content (w) or volumetric water content (θ) or degree of saturation, (S_r) and soil suction.

In this study, SWCC is obtained for the samples of different density. Densities of 1.1, 1.2, 1.3, 1.4, 1.5 and 1.6 g/cc are prepared and kept with filter paper in containers inside desiccator for 15 days to attain equilibrium. Details of test specimen are given in table 2. And water content is obtained from the filter paper for both total and matric suction. Graph has been plotted between suction values and water content which is shown in figure 8 and 9.

Table 2. Test Specimen - Details

Sample	Density	Water content
1	1.1	11, 13, 15, 17, 19, 20
2	1.2	11, 13, 15, 17, 19, 20
3	1.3	11, 13, 15, 17, 19, 20
4	1.4	11, 13, 15, 17, 19, 20
5	1.5	11, 13, 15, 17, 19, 20
6	1.6	11, 13, 15, 17, 19, 20

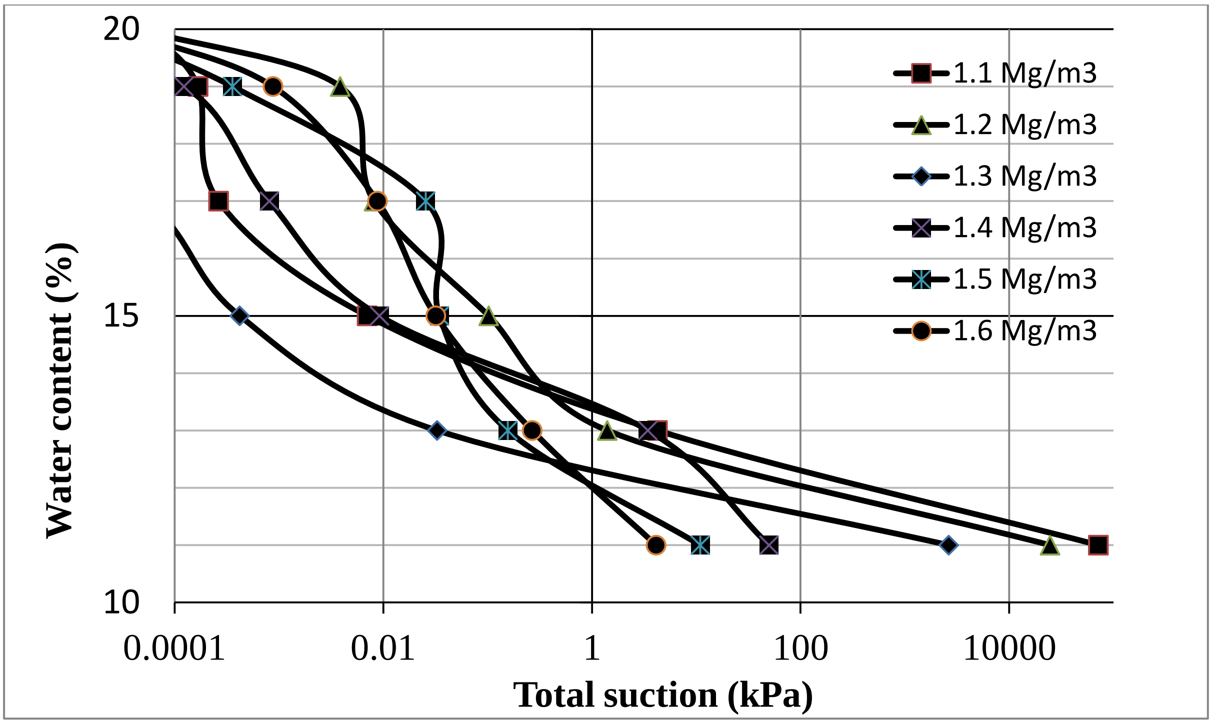

Fig.8. Variation of total suction with water content

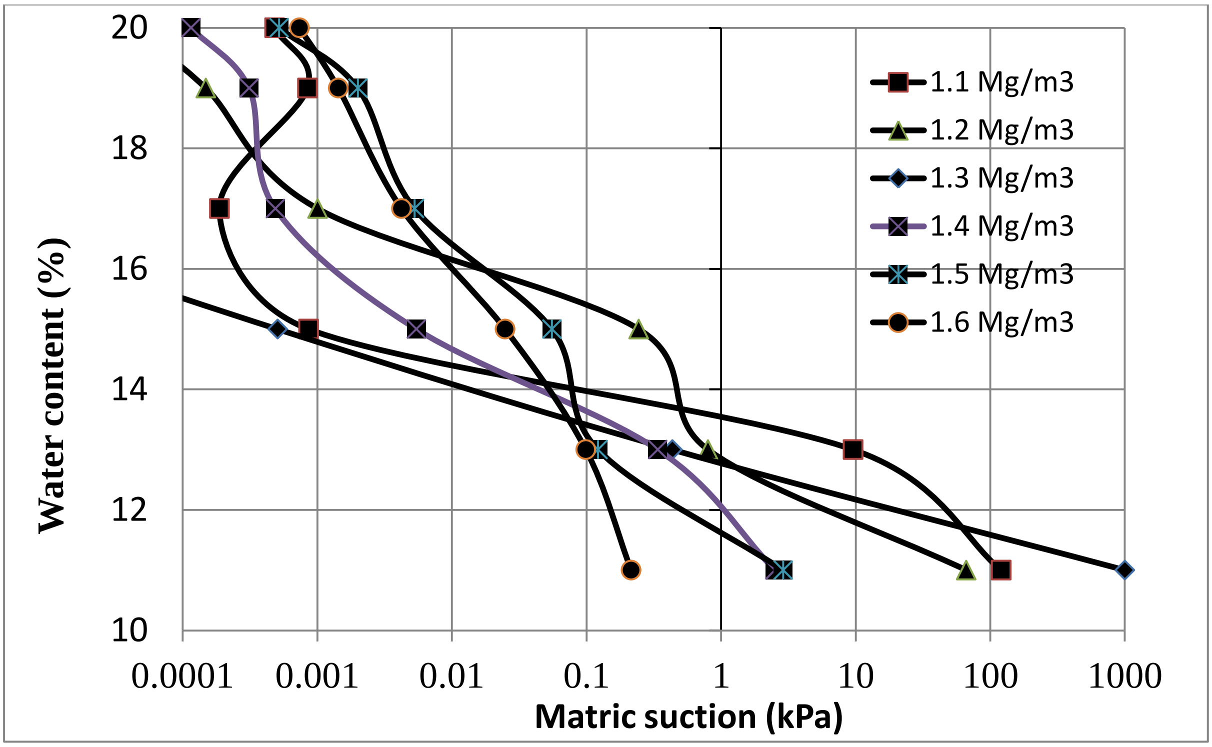

Fig.9.Variation of matric suction with water content

As we know, both total and matric suction values increases with the decrease in water content for any value of density, graph shows the same told behaviour. When it comes to density, suction value did not follow a pattern. The value of suction increases and decreases with dry density in a non-uniform manner irrespective of water content. The relation between dry density and suction is shown clearly in figure10 and 11.

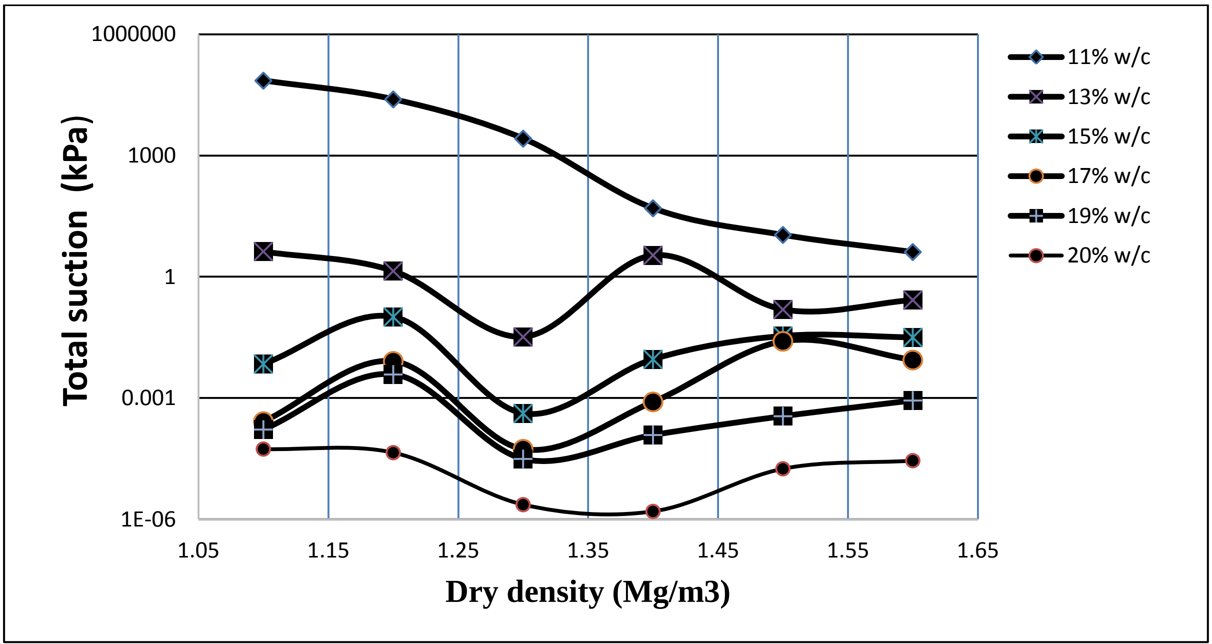

Fig.10. Variation of total suction with density

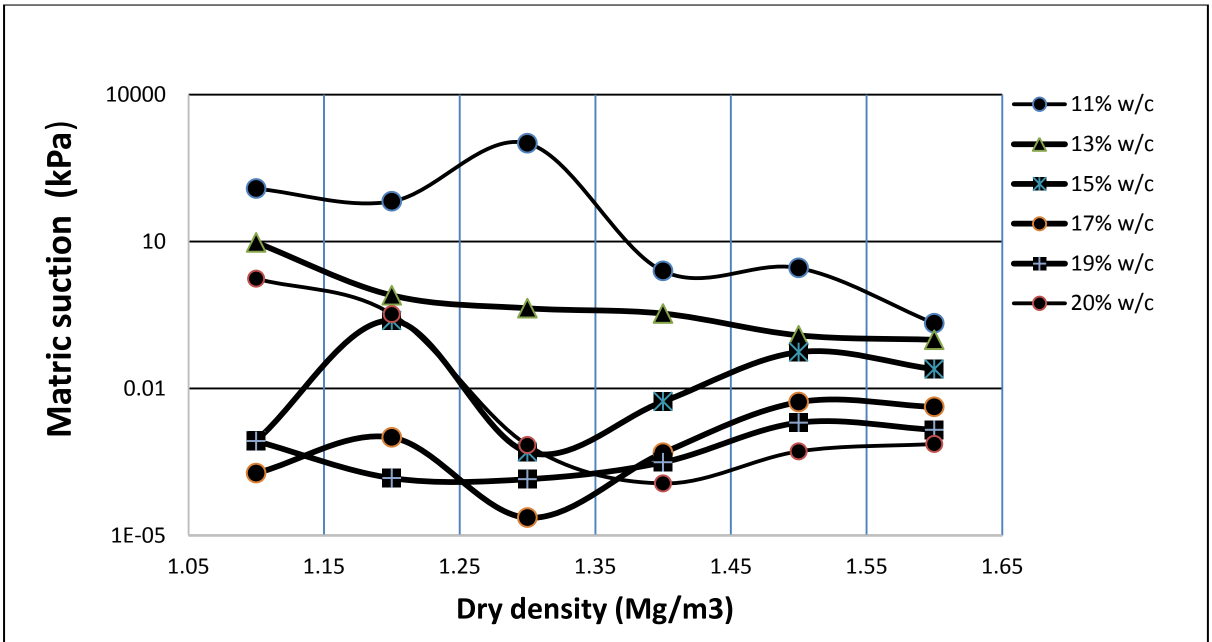

Fig.11. Variation of Matric Suction with density

Van Genutchen Model (1980)

Parameters have to be arranged so that we get the characteristic curve and these parameters will be different for different soil. Graph is shown in fig 6 for total suction and figure 7 for matric suction with parameter values of **α=1000 and m=0.3.** Saturated and residual water content are obtained from the experimental data.

Fredlund and Xing Model (1994)

Everything substituted in the given equation gives water content value for the corresponding suction value and the parameter values are residual suction as 4000 kPa, suction at inflection point as 0.01 kPa and water content as 16%.

Experimental data have been compared with both the models and the results are shown in figure12 and 13.

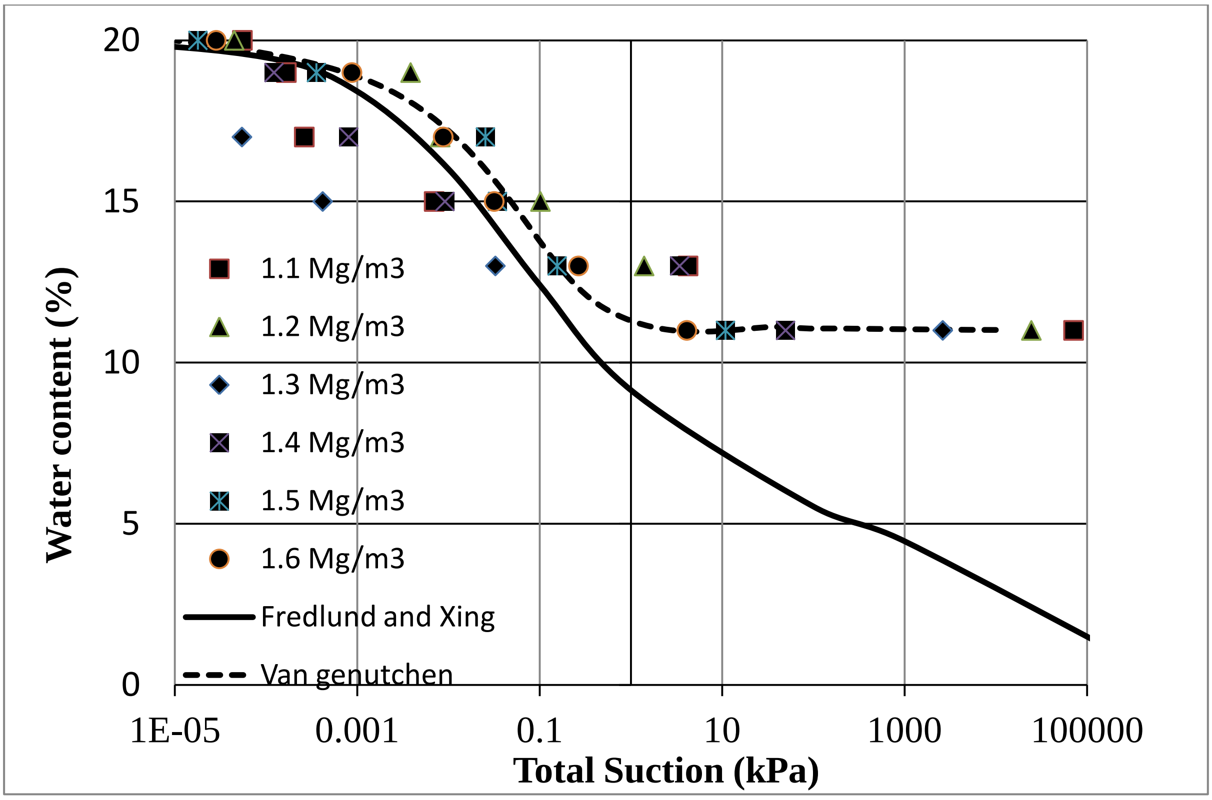

Fig.12. Variation of total suction with water content – Fredlund and Xing and van Genutchen models

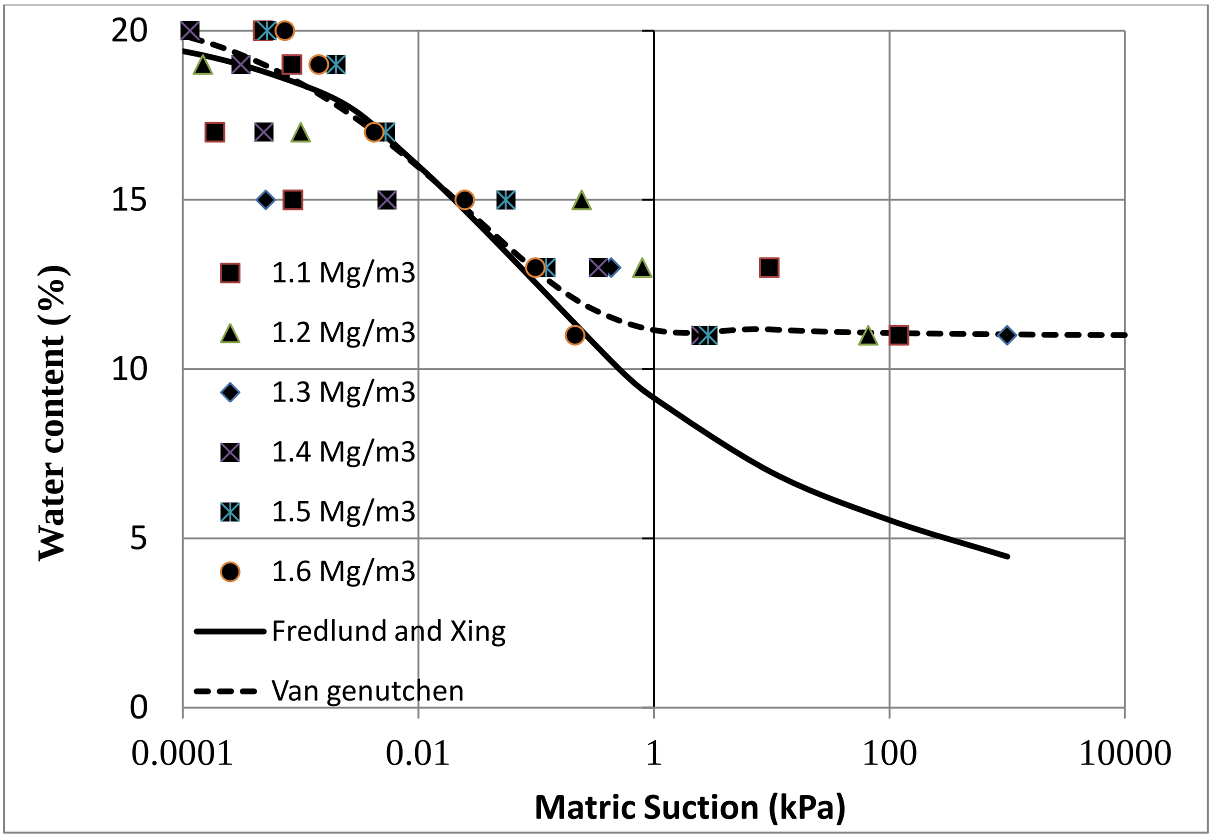

Fig.13. Variation of matric suction with water content – Fredlund and Xing and van Genutchen models

CHAPTER 5

CONCLUSIONS AND FUTURE WORK

5.1Conclusions

In the current study, suction behaviour of Indian bentonite was studied. Different densities and water content of bentonite were considered. It was found that with increase in water content, both total and matric suction values were decreased.

There was no particular pattern observed due to change in dry density at equal water content on matric and total suction variation.

Theoretical models such as Fredlund& Xing (1994) and van Genutchen (1980) were used to determine suction versus water content relationships. Comparison between experimental and theoretical results showed that van Genutchen (1980) model provide better relationship between suction and water content than that of Fredlund& Xing (1994) model.

5.2 Future Scope of work

Parameters such as swelling characteristics, compressibility, and consolidation can be determined for bentonite. Suction can also be measured by adding chemicals to the bentonite sample. Suction parameter may be determined for unsaturated soil types which are easily available than bentonite.

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