INTERACTION OF WATER WITH FILTER PAPER CELLULOSE/FIBER STRUCTURE IN SOIL SUCTION MEASUREMENTS

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Abstract: The filter paper technique of soil suction measurement is evaluated in this research. The main objective of this study is to evaluate the interaction between water and filter paper at molecular level through an extensive literature review and laboratory testing. Wetting and drying calibration data were obtained using tests based on a no-contact moisture transfer method between filter papers and salt solutions of different concentrations and de-ionized water. The Whatman No.42 and Schleicher and Schuell No. 589^2 filter paper types were studied. Sodium Chloride (NaCl) solutions of different concentrations were used to generate the suction needed for calibration. A series of experiments were conducted with differing equilibration periods. Wetting calibration curve equations were proposed based on the equations of Fredlund and Xing (1994) for both filter papers. An abrupt change in suction is observed in the wetting calibration curves, which is a likely indication of change in moisture sorption mechanism with the break point lying close to the Fiber Saturation Point of cotton linters measured using DSC and NMR in previous studies. The drying calibration data did not show a clear trend. Thus, no calibration curves have been fitted for the drying calibration data of both papers. Observation of the trend of the values for the drying calibration over the different equilibration periods clearly showed the data shifted inward showing less water content with longer equilibration period. This implies that the drying filter papers required longer equilibration periods and reaffirms that hysteresis decreases with increase in equilibration period. Critical values obtained in the calibration data have been associated with the type of water in the filter paper. Literature review and the data obtained in this experiment indicate that the filter paper method is dictated by the water-filter paper interaction at the molecular level in addition to the thermodynamics that dictates the moisture transfer and equilibrium conditions. Further study on this area might prove useful in the clear understanding of the filter paper method.

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CHAPTER I

INTRODUCTION

1.1 Soil Suction

Soil suction is negative pore water pressure or negative stress in the pore water in unsaturated soils. The term is commonly referred to as the free energy state of soil water (Edlefsen and Anderson, 1943) and measures the free energy of the pore water in a soil (ASTM D 5298-10, 2010). In practical terms, it is a measure of the affinity of soil to retain water (ASTM D 5298-10, 2010).

Soil suction has two components: matric suction and osmotic suction. Matric suction arises from capillarity, surface adsorptive forces on soil particles and the texture of soil particles. Osmotic suction comes from dissolved salts in the soil pore water. The knowledge of the value of soil suction is critical in the proper designing and construction of engineering infrastructure. There are a number of methods available for measuring soil suction directly and indirectly. The filter paper technique is among the simplest of the indirect methods that can be used to measure both total and matric suctions with a suction range of 10 kPa to 100,000 kPa (ASTM D 5298-10, 2010).

1.2 Total Suction and Relative Humidity

Likos and Lu (2003) described total soil suction in thermodynamic terms in terms of the free energy state of the soil pore water, which may be measured in terms of its partial vapor pressure (u_v) or relative humidity (RH) at local equilibrium. The relationship between total suction ψ_t (kPa) and relative humidity of water vapor is described by Kelvin's equation (Sposito 1981):

$$\psi_t = -\frac{RT}{V_{\omega v}\ \omega_v}\ln\left(\frac{u_v}{u_{vo}}\right) = -\frac{RT}{V_{\omega v}\ \omega_v}\ln(\mathrm{RH}) \qquad (1)$$

where u_v = the partial pressure of water (i.e., soil pore-water) vapor (kPa), u_{v0} = the saturation pressure of pure water vapor (kPa) at the same temperature, R = the universal gas constant (8.31432 J mol^{-1} K^{-1}), T = absolute temperature (K), V_{wo} = the specific volume of water (m^3/kg), and ω_v = the molecular mass of water vapor (18.016 kg/kmol).

1.3 Filter Paper Method

In the filter paper method, the suction of the soil is determined indirectly from pre-established calibration curves; wetting or drying calibration curve as appropriate (Bulut and Wray, 2005). In this method, the filter paper comes to equilibrium with the soil either through vapor (total suction measurement using non-contact filter paper technique) or liquid (matric suction measurement using contact filter paper technique) flow. At equilibrium, the filter paper and the soil will have the same suction value. After equilibrium is established between the filter paper and the soil, the water content of the filter paper is measured. For the measured value of water content of the filter paper, the corresponding suction value is read from a calibration curve for the type of filter paper used. The suctions inferred from filter paper measurements, therefore, depend on a calibration between the water content of the filter paper and suction in water (Bulut et al., 2001; Bicalho et al., 2010). Consequently, the accuracy of suction measurement using the filter paper method highly depends on the integrity of the calibration curve used.

The calibration procedure for filter paper involves measuring the water content of filter papers which have been equilibrated with salt solutions of known osmotic suction. The filter papers are suspended above the salt solutions and allowed to equilibrate for a ‘sufficient’ period of time after which the water content of the filter papers is measured. This should be performed for a number of known osmotic suctions to cover the range of suction interest when establishing the calibration curves – total suction versus water content curves.

The osmotic suction of different concentrations of salt solutions is calculated using the following equation (Bulut et al., 2001):

$$h_\pi = -\nu RTm\varphi \tag{2}$$

where h_π = osmotic suction; ν = number of ions from one molecule of salt (i.e., 2 for NaCl); R= universal gas constant (8.3143J deg/K mole); T= absolute temperature (K); m= molality and φ = osmotic coefficient.

Equation 2 is the combination of Kelvin’s equation which gives a relationship between total suction and relative humidity and the relationship between osmotic coefficients and osmotic suction (Lang 1967):

$$\Phi = -\frac{1}{vmV}\ln(\frac{P}{P_o}) \tag{3}$$

where Φ = osmotic coefficient; v = number of ions from one molecule of salt (i.e., v =2 for NaCl); m= molality; V= molar volume of free pure water; P= the partial pressure of pore water vapor and P_o = the saturation pressure of water vapor over a flat surface of pure water at the same temperature. The ratio P/P_o is the relative humidity.

Calibration curves should have a high accuracy which in turn depends on the careful setting up of the experiment, maintaining isothermal conditions, minimizing the exposure of the filter papers to the environment other than the temperature-controlled environment and accuracy in measuring the weights of the wet and dry filter papers which are used in the calculation of water contents.

1.4 Research Objectives

The primary objectives of this research are two-fold. The first objective is to relate the behavior of water sorption/desorption (wetting/drying) of the filter paper with the cellulose fiber-water interaction that takes place at the molecular level which required extensive literature review as discussed in the following chapter.

The second is to construct calibration curves for the Whatman No. 42 and Schleicher and Schuell No. 589[2] filter papers that can be used for the measurement of soil suction. A series of experiments were conducted to achieve this goal.

The secondary objectives are:

- To determine if equilibration periods used by previous researchers were sufficient to bring the filter paper and salt solutions to suction equilibrium;
- To observe the impact of longer equilibration periods on the calibration curves such as its impact on the nature of the calibration curves, and on the observed hysteresis between wetting and drying curves; and
- To evaluate the different regions of the calibration curve from the point of view of water and paper cellulose/fiber interaction.

CHAPTER II

REVIEW OF LITERATURE

2.1 Importance of Soil Suction

Soil suction is negative pore water pressure or negative stress in the pore water in unsaturated soils. It measures the free energy of the water in the soil (ASTM D 5298-10, 2010).

Unsaturated soil mechanics is a branch of the general field of soil mechanics that deals with unsaturated soils. Unlike saturated soils, unsaturated soils have more than two phases (soil, water and air) and the pore-water pressure is negative relative to the pore-air pressure (Fredlund and Rahardjo, 1993). Soils near the ground surface above the ground water table will be subjected to negative pore-water pressures and probable desaturation (Fredlund and Rahardjo, 1993). Also, processes such as excavation and recompaction result in unsaturated soils which cannot be treated using the principles of classical (saturated) soil mechanics owing to their different behavior (Fredlund and Rahardjo,1993). It is the negative pore-water pressure that makes the behavior of unsaturated soils different from that of saturated soils which have a positive pore-water pressure. It is this phenomenon exhibited by unsaturated soils that is called soil suction.

Soil suction measures the affinity of soil to attract and retain water and consequently the knowledge of the value of soil suction is vital as it has a strong influence on the mechanical behavior of unsaturated soils (Haghighi et al., 2012). Soil suction controls such crucial properties as volume change, deformation and strength in unsaturated soils (ASTM D 5298-10, 2010).

2.2 Soil Suction Measuring Devices

There are a number of devices that measure soil suction directly or indirectly. Almost all the methods measure only one component of the total suction, i.e, either the matric suction or osmotic suction. The exceptions to this are the Psychrometers which measure total suction and the filter paper method which can measure both total and matric suctions with good laboratory procedures. Table 1 below provides a list of devices/methods used for measuring soil suction with the type of suction and range of suction they measure.

Table 1 Soil Suction Measurement Methods (Pan et al., 2010)

	Technique/ Method	**Suction component measured**	**Range (kPa)**
Direct Measurement Technique	Tensiometer	Matric	0 – 1500
	Axis- transition technique	Matric	0 - 1500
	Suction probe	Matric	0 - 1500
Indirect Measurement Technique	Thermocouple Psychrometers	Total	100 – 10000
	Filter paper	Total and Matric	Entire range
	Electrical conductivity sensors	Matric	50 - 1500
	Thermal conductivity sensors	Matric	0 - 1500
	Relative humidity sensor	Total	100-8000
	Chilled mirror hygrometer	Total	150 - 30000
	Pore fluid squeezer	Osmotic	Entire range

The advantages of the filter paper method over most of the other methods are its simplicity, low cost, the wide range of suction values that it can measure and the fact that it does not require any special equipment. However, extreme care should be exercised in the test procedure and in the use of appropriate calibration curve if accurate suction values are to be obtained (Haghighi et al., 2012). The primary disadvantage of the filter paper method is the fact that its accuracy depends on the accuracy of the calibration curves (Likos and Lu, 2002).

2.3 The Filter Paper Method

It is believed that the filter paper was first introduced by Gardner in 1937 for measuring soil suction indirectly (Bicalho et al., 2010). The method was originally developed in the soil science discipline. According to Marinho and Oliveira (2006), Shull (1916) was the first researcher to use absorption processes as a soil suction measuring tool by using specially selected seeds as an absorbent material with the intention of measuring the force with which different sized soil particles hold moisture at various degrees of dryness. Before Gardner (1937), Hansen (1926) made use of filter papers to measure the soil suction but the filter papers were saturated with sugar solution with predetermined vapor pressure. Thus, Gardner (1937) is believed to be the first researcher to use filter paper as an absorbent material without priorly saturating it with any solution (Marinho and Oliveira, 2006). Fawcett and Collis-George (1967) believed that the use of filter paper rendered the measurement more reliable as the industrial process involved in the production of the paper is essentially the same which, in turn, makes the basic characteristics of the filter paper uniform irrespective of the batch used.

It is stated in the ASTM D 5298 (2010) that the filter paper method measurement range is from 10 to 100,000kPa. Fredlund and Rahardjo (1993) claimed the method measures the ‘entire range’. However, Agus and Schanz (2006) suggested that the technique should be limited to suction values higher than 200 kPa with equilibration temperature fluctuation limited to $\pm 0.1^{o}$C to keep the error in suction measurement to 30%.

The fact that the filter paper method should be conducted with utmost care cannot be too emphasized. The accuracy of the result obtained when using the filter paper method, especially the non-contact vapor transfer calibration method highly depends on strict adherence to tested protocols such as used for this research. Harrison and Blight (1994) observed that while both the contact and non-contact methods show scattered results, the data from the non-contact method were found to be more scattered and contradictory. Houston et al. (1994) also stated that inaccurate results can be obtained if appropriate precautions are not taken. ASTM also notes that the quality of the result from the standard put forward in D 5298 depends on the capability of the personnel conducting the experiment, and the suitability of the equipment and facilities used. Fredlund and Rahardjo (1993) also emphasized that the filter paper technique is “highly user-dependent” and that extreme care must be taken as the measurement involves small masses associated with the filter papers.

Furthermore, extra care should be exercised to avoid errors from temperature fluctuations, relative humidity error and insufficient equilibration time when using the non-contact filter paper technique or the vapor moisture transfer method (which is the method used to calibrate filter papers in this research) (Bicalho et al., 2010).

2.3.1 How the Filter Paper Method Works

Most soil suction measuring devices make use of an exchange of water between the soil and the device (Marinho and Oliveira, 2006) and the filter paper method is no exception. Filter paper, being a porous material, absorbs water when it is placed with a soil specimen. The filter paper method works on the assumption that the filter paper will come to equilibrium with a soil having a specific suction. The equilibrium state, which is attained after sufficient equilibration time is allowed, results in the same suction in both the filter paper and the soil specimen. The moisture exchange can take place in either the liquid or vapor form. When a dry filter paper is placed in direct contact with a soil specimen, water flows from the soil to the paper until equilibrium is reached, whereas if the paper is suspended over a soil specimen with no direct contact with the specimen, the moisture exchange takes place in the form of water vapor (Fredlund and Rahardjo, 1993). After equilibrium is established, the water content of the filter paper is measured from which value will be inferred the suction of the soil specimen by making use of a pre-existing calibration curve constructed for the specific type of filter paper.

2.3.2 Filter Paper Calibration

The filter paper method measures soil suction indirectly, and thus the accuracy of the method depends on the moisture-suction relationship of the filter paper which makes the calibration procedure for the filter paper very important (Leong et al, 2002).

The calibration curve for the filter paper is the soil water retention curve for the filter paper (Marinho and Oliveira, 2006). The calibration procedure involves allowing the paper to equilibrate with a known suction source; measuring the weight of the paper after equilibration; oven-drying the filter paper and taking the oven-dry weight of the filter paper to calculate the water content of the filter paper (Marinho and Oliveira, 2006). The measured water content is related to the suction generated and a water content – suction relationship curve is plotted. For a calibration curve to be constructed several measurements need to be made to cover all the range of suction of interest.

ASTM D5298 – 10 states that a typical calibration curve consists of two parts. A break point occurs between the two parts at a water content value equal to 45.3% for Whatman No. 42 (Fawcett and Collis-George, 1967; Greacen, et al., 1987) and 54% for Schleicher and Schuell No. 589[2] (McQueen and Miller, 1968; Greacen, et al., 1987).

Leong et al. (2002) also stated that the calibration curve is usually represented by two equations representing “different sensitivities of the filter paper response in the higher and lower suction ranges” thereby implying a two-segmented curve. However, a single equation can also be used over the entire suction range. Leong et al. (2002) used the soil – water characteristics equations of Genuchten (1980) and Fredlund and Xing (1994) to fit their experimental data with a single equation.

Many researchers agree that calibration of filter papers should be performed under conditions similar to experimental conditions (Ridley 1995, Sibley & Williams 1990, Swarbrick 1995). Marinho and Oliveira (2006) clearly stated that all the experimental conditions used during calibration must be adopted during the soil suction measurement, including the equilibration time. When using published total suction calibration curves, Bicalho et al. (2010) also recommended caution since such curves will be valid only for the equilibration time used during calibration.

2.3.2.1 Quality/Consistency of Filter Paper

There seems to be a divided belief in the literature on the use of the same calibration curve for filter papers from different batches or boxes (Leong et al., 2002, Hamblin, 1981, Marinho and Oliveira, 2006, Likos and Lu,2002, Deka et al., 1995). However, it is also believed that the better control in the filter paper production process would make it more uniform in quality and therefore make the filter paper a repeatable sensor (Leong et al, 2002).

Hamblin (1981) calibrated two different batches of Whatman No.42 filter paper and reported very good agreement between the data from the two different batches and also that the data were almost identical with those of Fawcett and Collis-George (1967) for the same type of filter papers. Leong et al. (2002) mentioned that the calibration data of Chandler and Gutierrez (1986) and Swarbick (1995) were in similar agreement with those of Fawcett and Collis-George (1967). Leong et al. (2002) also calibrated Whatman No. 42 and Schleicher and Schuell No. 589[2] filter papers and compared their calibration data with data from the literature and found that the data were close to each other although the data were obtained by different researchers, at different times, with different batches of filter paper. This suggests that the filter papers are consistent. Marinho and Oliveira (2006) also affirmed that the filter paper is an industrial material, produced under rigorous quality control, and thus its calibration curve should not change from batch to batch.

On the other hand, Likos and Lu (2002) investigated seven different batches of Whatman No. 42 filter papers to examine the variation in the calibration characteristics of different batches of paper. They concluded, on the basis of their experiments, that the variation observed between the calibration curves for the various papers were significant among different batches and strongly recommended independent calibration on a batch-to-batch basis. Likos and Lu (2002) also cautioned against the “unverified acceptance” of the ASTM calibration curve as the calibration data from their experiments for Whatman No. 42 did not exactly coincide with that suggested by ASTM D5298.

Deka et al. (1995) also investigated four batches of Whatman No. 42 filter papers and concluded the differences in the calibration curves between different batches of filter paper were significant and thus it was necessary to calibrate each batch.

Different researchers have come up with different calibration curves for the same type of paper. However, Leong et al (2002) attributed the differences in the filter paper calibration curves in the literature to the suction source used in the calibration and equilibration time. The filter paper calibration process consists of generating known values of suction. There are several methods for generating suctions and different researchers who calibrated Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers generated suctions in different ways. For example, Leong et al (2002) calibrated Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers using pressure plate and the salt solution methods and compared them with the calibration data of the same type of filter papers calibrated using soil samples of known matric suction. They found that the latter data showed greater scatter than the former and attributed the scatter to the uncertainty in the suction of soil samples used in the calibration and stated that discrepancy is usually caused by the uncertainty in the suction of the soil. However, calibrating filter papers using soil samples of 'known' suction is seldom done.

This observation calls for the careful consideration of the calibration conditions in question when one tries to compare different calibration curves for the same type of filter papers.

2.3.2.2 Single Calibration Curve for both Total and Matric Suction Measurement

The ASTM D 5298-10 (2010) provides a single calibration curve for the measurement of both matric and total suction. Houston (1994) found different total and matric suction calibration curves for Fisher filter papers. Bulut et al (2001) also found different curves for Schleicher and Schuell No. 589^2 filter papers. The same observation was made by Leong et al. (2002) who developed total and matirc suction calibration curves for Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers and found that the calibration data showed different responses. However, Bulut et al. (2001) and Marinho and Oliveira (2006) showed one calibration curve can be used for both the total and matric suction measurement using the filter paper method. It is the type of contact between the filter paper and the soil water that defines whether total or matric suction is being measured; when the filter paper is in close contact with the soil water, matric suction is measured whereas when the filter paper is not in contact with the soil water, the moisture transfer will take place in the form of vapor flow and thus total suction is measured (Bulut et al. 2001, ASTM D 5298-10, 2010). Based on this concept, Marinho and Oliveira (2006) stated that "it is not possible to have two different calibration curves for the filter paper according to the type of contact" whether matric or total suction is measured. Marinho and Oliveira (2006) attributed the differences observed by the other researchers to a misunderstanding of the suction generated and not to calibration curves.

2.3.2.3 Equilibration Time

Another important point in the calibration process is the equilibration time. It is imperative that sufficient time be allowed for the filter paper and the salt solution to come to suction equilibrium. Different researchers have reported and recommended different equilibration times. The ASTM D 5298 recommends a minimum equilibration time of seven days. Munoz-Castelblanco et al. (2012) measured suction for soil samples using filter papers and as part of their study monitored equilibration by taking measurements at different equilibration periods that range from 1 to 12 days and concluded that seven days were sufficient for a filter paper and soil specimen to reach equilibrium. The equilibration time of the filter paper depends on the suction source, contact condition, and suction level (Leong et al., 2002) which explains the different equilibration times suggested by different researchers for the contact and non-contact methods. The minimum equilibration period used is minutes used by Hamblin (1981) for the contact method while the maximum is 25 – 30 days by Harrison and Blight (1998) for drying calibration curve using the contact method. Other factors that affect the equilibration time are temperature fluctuations during equilibration, thickness and also structure of the filter paper (Marinho and Oliveira, 2006).

The equilibration time for the calibration of filter papers also depends on the suction level of the solution used for calibration when using the non-contact vapor transfer technique (Marinho, 1994a). Marinho (1994a) concluded that longer time is needed for equilibration for lower suction solutions. Accordingly, he recommended a longer equilibration period of 30days or more for suctions less than 250 kPa when using the vapor equilibration technique. Bicalho et al. (2010) also recommended that sufficient equilibration period be allowed in the vapor moisture transfer method as vapor equilibration is slow in the wet range since a large amount of water will be transferred before equilibrium is attained.

According to Marinho (1994a), equilibration time is also related with the gap between the filter paper and the suction source. Marinho (1994a) showed that the gap between the filter paper and the suction source affects the moisture absorption at a given time and thus the equilibration time should be associated with this distance at which the filter paper is placed from the soil/salt solution. Marinho and Oliveira (2006) recommended the distance between the filter paper and the liquid surface be at most 1cm.

Marinho and Oliveira (2006) also stated that when the time allowed is less than the equilibration time needed for the whole setup to come to equilibrium, the filter paper water content will depend on its distance from the source of suction.

2.3.2.4 Hysteresis

Owing to hysteresis, a single calibration curve cannot be used for both wetting and drying filter paper calibrations. The calibration curve for initially dry filter paper is not the same as that for the paper initially soaked in water.

Experiments showed that the drying and wetting of filter paper exhibits hysteresis (Leong et al., 2003; Bulut and Wray, 2005) and the calibration curves also show this phenomenon. The moisture absorption by a filter paper, or any porous medium, at a given relative vapor pressure (suction) is greater when it is brought to suction equilibrium from the wet state than when the paper is brought to equilibrium from the dry state. This discrepancy is known as hysteresis (Christensen and Giertz, 1965).

Regarding the hysteresis exhibited by the sorption/adsorption isotherms of cellulose materials, Christensen and Giertz (1965) reported that the ratio of adsorption water content value to desorption water content value is essentially constant (about 0.85) at all relative vapor pressures, except at the ends of the loops and shows a slight change between different cellulose materials. Houston et al. (1994) reported no significant hysteresis between wetting and drying calibration curves for matric suctions in the range between 8 and 2,500 kPa.

Leong et al (2002) stated inadequate equilibration time results in larger hysteresis. They also made the observation that hysteresis was small (between 1 and 5% in the filter paper water content) whereas the largest differences were observed for suctions less than 100 kPa.

Leong et al (2002) investigated the drying and wetting curves for two types of papers: Whatman No. 42 and Schleicher and Schuell No. 589^2 and observed hysteresis. They observed that the equilibration times for drying were longer than for wetting confirming that inadequate equilibration time will result in larger hysteresis in the wetting and drying responses of the filter paper. On the basis of their results, Leong et al (2002) noted that hysteresis appeared minor with sufficient equilibration time. They also noted that hysteresis was less noted for the Whatman No. 42 filter paper. From experiments, Leong et al (2002) also believed that Whatman No. 42 filter paper showed a better data consistency than Schleicher and Schuell No. 589^2 filter papers.

2.4 Interaction between Filter Paper and Water

The study of the interaction between filter paper and water calls for a study into the constituent elements of the paper and the ways in which the water interacts with these elements. The following sub-sections present a review of the nature of paper and its major constituent cellulose; water classification; the interaction of cellulose with water and how this interaction affects or is related to the moisture absorption/desorption process the filter paper is subjected to during the calibration and/or soil suction measurement process.

2.4.1 Paper/ Filter Paper

The primary constituent of paper is cellulose fiber which comes from the cell wall of plants, mainly trees (Walsh, 2006). The cell wall of a tree, in turn, consists of approximately 40% to 50% cellulose, 15% to 35% lignin and 20% to 30% hemicelluloses (Dimmel, 2001). A significant part of the hemicelluloses, however, are dissolved during kraft pulping (Biermann, 1996). Kraft pulping also results in delignification of fibers reducing the lignin content to 3 to 5.2% for bleachable pulp grades which is further reduced by bleaching (Biermann, 1996). Although the pulping process gives filter papers higher porosity and makes it devoid of pore structures, the overall cell structure (cell wall and lumen) and cellulose matrix remain intact (Felby et al., 2008).

The filter papers used in the measurement of soil suction and for calibration purposes should be ash-free quantitative Type II filter papers, the most common of which are Whatman No. 42 and Schleicher and Schuell No. 589[2]. Whatman No. 42 cellulose filters are manufactured from high quality cotton linters which have been treated to achieve a minimum alpha cellulose content of 98%. Schleicher and Schuell No. 589[2] filter papers also contain between 97-98% alpha cellulose. Cotton linters are the short fibers that remain stuck to the cotton seed after the cotton fluff (seed fiber) has been removed (KLUG Conservation information document taken from www.klug-conservation.com).

2.4.2 Cellulose

Natural cellulose is arranged as parallelly oriented cellulose chains commonly called microfibrils (Brown et al., 2002). Microfibrils are found throughout the fiber combined with hemicelluloses and lignin (Biermann, 1996, Mark, 1967). Figure 1 shows all the sub- parts of the cellular structure of a tree. Figure 2 and Figure 3 show the cell wall structure and the cellulose molecule at a larger scale.

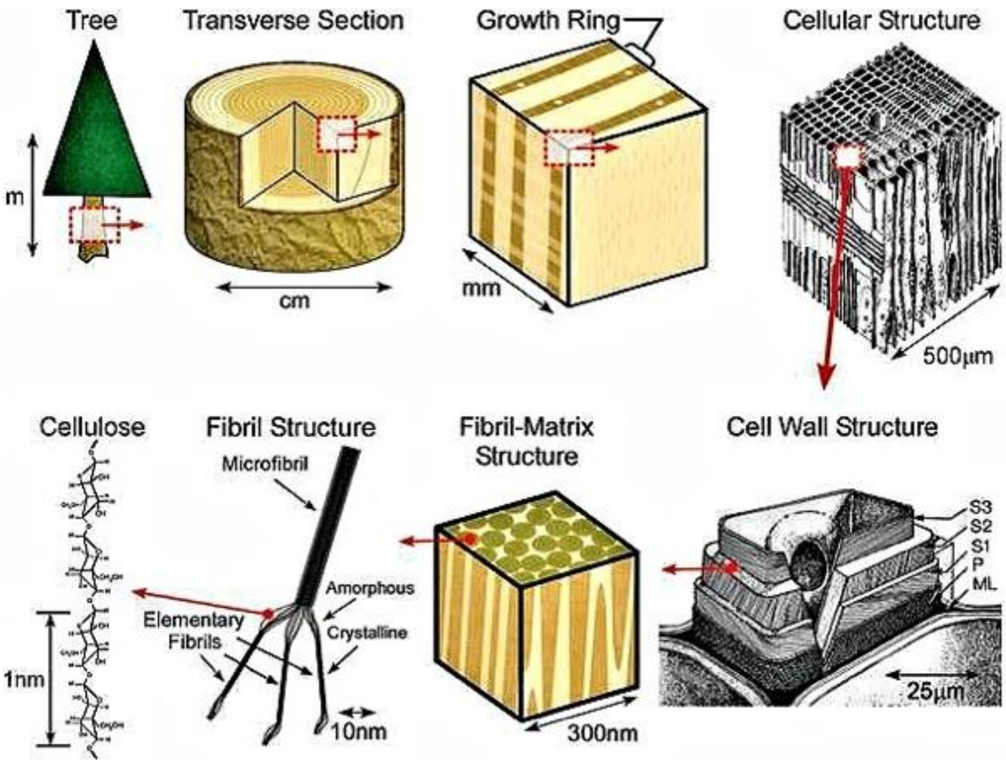


Figure 1: From Tree to Cellulose (Source: a.purposefulprocess.org)

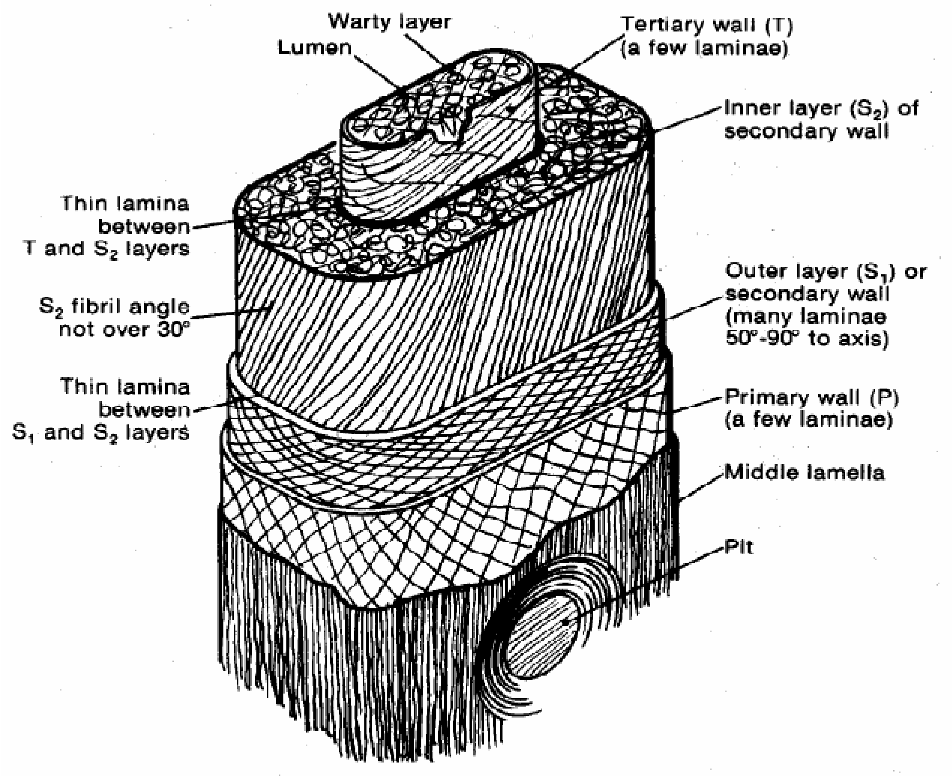


Figure 2: Cell wall structure (Walsh, 2006)

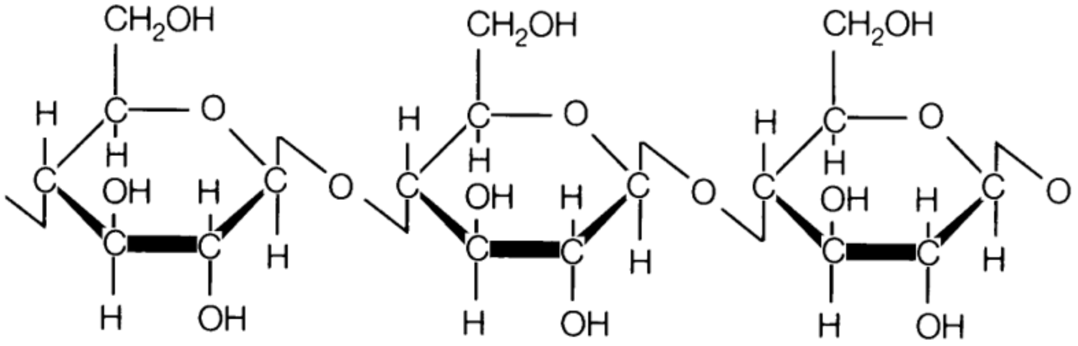


Figure 3: Cellulose molecule (www.thebiochemsynapse.wordpress.com)

The microfibril molecules are hydrogen-bonded to one another and form crystalline and amorphous regions rendering cellulose semi-crystalline (Grinsted and Wilson, 1979). The strength and rigidity of cellulose is attributable to the crystalline region which has a perfect bonding (Walsh, 2006). The highly ordered crystalline structure of cellulose has extensive hydrogen bonding between the molecular chains which renders the cellulose insoluble in water (Walsh, 2006; Child and Jones, 1973). The flexibility, on the other hand, of cellulose comes from the amorphous regions where the bonds are not aligned thus creating movement within the molecule (Nissan, 1961). Amorphous cellulose has unstructured regions which are accessible to water, and thus are critical in the cellulose – water interaction (Clark, 1985). The maximum water uptake of cellulose is determined by its degree of crystallinity (Olsson and Salmén, 2004). The greater the degree of crystallinity, the lower will be the moisture uptake at a given relative humidity (Waterhouse, 1985).

2.4.3 Water Classification

Owing to the physical structure and chemical composition of the plant cell wall matrix which subject water to a number of interactions, water in a cellulose system has different states and locations. A common classification of the water identified in the literature (Walsh, 2006, Felby et al., 2008) on the basis of its association with the fiber is as follows.

- Free water (unbound water): water with properties similar to bulk free water surrounding, but not associated directly with the cellulose fiber
- Bound freezing water
- Bound non-freezing water

The following two types are also considered as additional types of water although they are not as frequently encountered as the above three:

- Water of constitution
- Trapped water

Bound water (freezing and non-freezing) is the water associated with cellulose surfaces.

On the surface of the cellulose microfibrils, the water is packed in ordered layers or clusters reflecting the crystalline structure of the cellulose. This packed water is denoted non-freezing water (Felby et al., 2008). According to Overloop and Vangerven (1993), the first layer of two to three molecules in direct contact with cellulose constitutes this non-freezable water.

At the cell wall level, absorbed water is located in a porous structure with confined spaces where the water is bound. Bonding occurs either by capillary forces, by hydrogen bonds to hydroxyl groups on hemicelluloses and lignin or by hydrogen bonds to hydration layers already adsorbed. This type is classified as freezing bound water (Felby et al., 2008; Ping, 2001). This water has a depressed freezing point compared to bulk water (free liquid water) owing to its location in small pores within the cell wall where the interaction with cellulose destructures the water (Perkins and Batchelor, 2012; Nelson, 1977). The classification primary and secondary for non-freezing and freezing water, respectively is also used.

Christensen and Giertz (1965) also included another type of water that can be considered as taking part in the cellulose-water interaction: water of constitution. They stated, however, that the line of demarcation, between this water of constitution and the bound water is not very sharp. This water may be defined as the condition corresponding to zero relative vapor pressure, which, experimentally can be approached only by heating at temperatures above 100^{o}C which could decompose the cellulose material (Christensen and Giertz, 1965). The water of constitution is defined by most English language dictionaries as the water so combined into a molecule that it cannot be removed without disrupting the entire molecule. According to Christensen and Giertz (1965) and Walsh (2006), the bound water is the second most strongly held water, next to the water of constitution.

Trapped water is sometimes included as a fifth category. Trapped water is described as the water that is not bound to the fibers but is difficult to evaporate (Park et al., 2007).

The fibers in a paper sheet form a porous network; the porosity comes from the spaces between the fibers. This porous system contains some of the free water (so called trapped water). The fibers are made of stacks of microfibrils which are, as stated in Section 2.4.2 parallelly oriented cellulose chains. There are, consequently, pores of nanoscale dimension within the fibers. The location of both freezing and non-freezing bound water types are within the fiber porous network and the surface of the fibers, in close proximity with the microfibrils (Perkins and Batchelor, 2012).

2.4.4 Cellulose – Water Interaction

Water can be directly attached to cellulose through the hydroxyl regions in cellulose which interact easily with water by exchanging of hydrogen atoms in the cellulose structure with those in the water. Water can also be attached to cellulose indirectly thorough modified water structure by attaching itself to hydroxyl groups of water molecules already directly attached to the cellulose. (Walsh, 2006; Child and Jones, 1973; Felby et al., 2008)

Some of the hydrogen atoms present in the cellulose structure are exchangeable with those atoms present in the water. The hydrogen atoms of the cellulose structure bonded to the carbon are considered non-exchangeable. About half of the hydrogen atoms attached to the oxygen in the cellulose structure are in an amorphous structure and readily exchangeable with water. These hydroxyls interact easily with water due to the dipole nature of water (Walsh, 2006).

2.4.4.1 Water Sorption Theories

Christensen and Giertz (1965) presented three main theories of water “binding”. The following sections describe each of these theories.

i) Surface adsorption theory – states that cellulose fibers possess a large amount of internal surfaces which could be the surfaces of microfibrils or surfaces around accessible voids and capillarities. These surfaces contain active hydroxyl or other polar groups to which the water molecules can be attached strongly through hydrogen bonds. When water is initially taken up, it is assumed to form a monomolecular layer over these available surfaces. This monolayer adsorption takes place approximately up to 20% relative humidity. Multi-layer absorption is assumed to take place at higher relative humidity values (Christensen and Giertz, 1965).

ii) Capillary condensation theory – capillary condensation is the “process by which multilayer adsorption from the vapor phase into a porous medium proceeds to the point at which pore spaces become filled with condensed liquid from the vapor" (Schramm, 1993). In capillary condensation, vapor condensation occurs below the saturation vapor pressure of the pure liquid (Hunter, 2001). The theory is based on the premise that vapor pressure of water in a capillary depends on the radius of the capillary, decreasing with decreasing radius. Small capillaries will cause water vapor to condense at a relative vapor pressure of approximately 0.7 (relative humidity of 70%). The amount of water condensed increases with the lumens filling close to relative humidity value of 100% (Christensen and Giertz, 1965).

iii) <u>Swelling theory</u> – according to this theory, water is absorbed in the disordered regions (consisting mainly of amorphous regions) of the cellulose where there are hydroxyl groups that can form hydrogen bonds with water molecules (Christensen and Giertz, 1965). Amorphous regions of the fiber are believed to control swelling/ water- accessibility to a great extent (Stamm, 1964).

2.4.4.2 Sorption/ Water Up-take Models

Perkins and Batchelor (2011) studied water and cellulose interaction in paper cellulose fibers by making use of Nuclear Magnetic Resonance (NMR) pulsed field gradient. A number of researches on the interaction of cellulose and water made use of the Nuclear Magnetic Resonance (NMR) spectroscopy technique.

Measurements of proton NMR relaxation can describe the interactions of absorbed water with cellulose because the rate of relaxation of the water protons is quite sensitive to the nature of their environment and to the degree of mobility the molecules have within the structure. At very low water content, the molecules are tightly bound, and the water molecules behave more like solid and their relaxation is quite slow. With the addition of more water molecules to the environment, they are less tightly bound, their relaxation is faster than that of protons in water molecules that are very close to the interface and the average relaxation time gets diminished (Platt et al., 1987).

By measuring relaxation times T1 (spin-lattice relaxation time) and T2 (spin-spin relaxation time), this method gives an indication of the mobility of water molecules interacting with cellulose substrates. The T2, however, seems to be the more commonly used measure of relaxation time. The T2 relaxation time of hydrogen nuclei depends on the extent of freedom the hydrogen nuclei has to move, i.e. which molecular environment it is surrounded by and the physical state of that solid or liquid environment (Felby et al., 2008).

According to Perkins and Batchelor (2011), the mobility of water is dominated by its direct interaction with cellulose. As part of their research on water interaction in paper cellulose fibers, they proposed the following water layer hypothesis to describe the change in diffusion behavior with moisture content increase. When the moisture content is very low, all the water within the fibers is expected to be in close adjacency to the surface of the cellulose microfibril and thus the diffusion behavior is strongly affected by this interaction between the cellulose and water. As more moisture is absorbed, the water layers thicken, the closest layer to the cellulose covers the outer layer from the effect of the cellulose and as a result the outer layer begins to show a more independent or mobile phase unaffected by the cellulose surface. The closest layer will now be interacting with the outer layer, but still has some interaction with the cellulose. According to Perkins and Batchelor (2011), different diffusion coefficients are observed for the close and outer layers. At very high moisture content where bulk water starts to be present, the close layer and outer layer are observed as one component and the bulk, or free water, is observed as second component.

Using NMR technique, Hartley et al. (1992) proposed a “tentative hydration model” for wood which explains the peculiar form of the T2 with moisture content in qualitative terms as follows.

When water first attaches itself to the wood, hydration sites are occupied by single water molecules. As more water is added to the wood, the monomolecular hydration continues, but some of the water molecules will be attached to sites already occupied, forming water polymers (dimers and perhaps trimmers). With the formation of such cluster, the bond to the cellulose substrate is likely to be weakened with the cluster becoming more mobile. As the clusters become larger, the water molecules experience an environment close to that of bulk water. Consequently, the mobility of the water molecules increases with the result that the water proton T2 becomes longer (Hartley et al., 1992).

Child (1972) studied the state of sorbed water on celluloses derived from cotton and wood pulp sources using pulsed NMR techniques and proposed a sorption mechanism which involves chain attenuation (motion attenuation) at a certain stage in the sorption process which is caused by water bridges formed between cellulose chains. According to this theory, initial sorption takes place at hydroxyl groups replacing relatively weak inter-chain hydrogen bonds with stronger water-chain hydrogen bonds, but as more water enters the system, and the chains move further apart, water bridges may be formed. According to Child (1972) primary and secondary bridges with one or two water molecules form a link between the chains and tend to weaken the chain motion. As more water enters the system, the bridges will be broken and chain motion will increase.

2.4.4.3 Moisture Transitions during Sorption

Studies have also been conducted to determine moisture transitions in cellulosic materials. Specific moisture distributions could be identified in cellulose at any moisture content by making use of NMR spectroscopy (Froix and Nelson, 1975). This is essential for cellulosic products as specific physical property changes are related to the incorporation and interaction of particular types and amounts of water within the cellulose (Froix and Nelson, 1975). One moisture transition identified in cellulose materials is fiber saturation point. Tiemann (1906) defined fiber saturation point (FSP) as the moisture content below which wood would begin shrinking. Christensen and Giertz (1965) defined FSP as the amount of water required to completely fill the cell wall without filling the lumen. They also defined the FSP thermodynamically as a point that occurs when the heat of sorption becomes zero – any more moisture absorption beyond this point will be without heat effects and is called free water. However, Walsh (2006) argued the result of the measurement of FSP is not "bound water" as this measurement gives the total amount of water within the pores of the fiber and thus it is very likely that some and probably most of the water measured is not chemically bound to the fiber through hydrogen bonds.

Nelson (1977) used Differential Scanning Calorimetry (DSC) to determine moisture transitions in cellulosic materials. Differential scanning calorimetry is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature (source : http://en.wikipedia.org/wiki/Differential_scanning_calorimetry). DSC is commonly used to thermodynamically determine the water types in the fiber (Perkins and Batchelor, 2011). Total bound water contents were determined for cotton linters, which have been used as a model system in this study. Three distinct moisture transitions have been identified for the cotton linters as follows: free water incorporation point of 0.05g/g (WC=5%), total bound water content of 0.18g/g (WC=18%) and fiber saturation point (FSP) of 0.5g/g (WC=50%). Nelson (1977) claimed that the 0.5g/g FSP value agrees well with a published FSP of a cotton linters sample using the solute exclusion technique. Nelson (1977) defined FSP slightly differently as the transition between “free” water – water contained within the cell wall and freezes at a lower temperature than normal water – and “bulk” water which exists outside the cell wall.

Also, the free water incorporation point and total bound water values determined by Nelson (1977) were compared to those found in a pulsed NMR study where the free water incorporation point was found to be 0.046g/g and total bound water was 0.19g/g, which is an adequate agreement (Nelson, 1977). Nelson (1977) also determined the bound water content of various paper samples to be between 0.21 and 0.27 g/g; the total bound water for Whatman No.1 filter paper was determined to be 0.265 g H_2O/g dry sample.

Froix and Nelson (1975) studied the cellulose-water system by measuring NMR relaxation times for cotton linters samples. A *point of plasticization* associated with the transition between primary and secondary bound water was identified to be 0.09 g/g. This point, in turn, is associated with swelling of the cellulose structure which permits increased mobility of both the cellulose chains and bound water. A moisture content of 0.2 g/g has also been identified as another moisture transition, namely, total bound water. Froix and Nelson (1975) also showed that although the water initially absorbed is bound water, free water exists even at low moisture contents with the amount of free water hiking above the point of plasticization.

Ping (2001) also made use of Differential Scanning Calorimetry (DSC) and Fourier Transform Infrared Spectroscopy (FTIR) to show the types of water absorbed by different hydrophilic polymers while wetting. FTIR (Griffiths and de Hasseth, 2007) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. A sorption model similar to Hartley et al. (1992) was proposed by Ping (2001). This study concluded that below a certain value of water content, the absorbed water is not freezable (first hydration layer attached directly to the active site of the polymer) and with increase in water content, the second layer of water molecules is freezable and is attached to the first hydration layer and behave differently from bulk water.

Felby et al. (2008) considered the fiber saturation point of cellulose to be between 0.33 – 0.43g/g moisture while 1.85 to 2.33g/g moisture was considered as full saturation.

The following list summarizes the different moisture transitions identified by the different researchers as discussed in the preceding paragraphs:

Nelson (1977) determined the following transitions for cotton linters:

- Free water incorporation point of 0.05g/g (WC=5%);
- Total bound water content of 0.18g/g (WC=18%); and
- Fiber saturation point (FSP) of 0.5g/g (WC=50%).

Nelson (1977) also determined the bound water for various paper samples and found them to be between 0.21 to 0.27 g/g. The total bound water of Whatman No.1 filter paper was determined to be 0.265 g H_2O/g dry sample

Froix and Nelson (1975) determined the following moisture transitions for cotton linters:

- Point of plasticization moisture of 0.09g/g (WC=9%); and
- Total bound water content of 0.2g/g (WC=20%).

Felby et al. (2008) stated the following transitions for cellulose fibers:

- Fiber saturation point between 0.33 – 0.43g/g (WC=33 - 43%); and
- Full saturation point between of 1.85 – 2.33g/g (WC=185 - 233%).

2.4.4.4 Desorption /Drying

Desorption/drying is the reverse of the sorption (moisture uptake) process. The strong interaction among cellulose fibers and interaction between the sorbed water and cellulose fibers directly affects the thermodynamics of water removal from cellulose fiber (Sen et al., 2012). According to Weise et al. (1996), during drying, the different water types (non-freezing bound, freezing bound and free/bulk water) are removed in sequence according to the strength of their interaction with the fibers, with some expected overlap at the boundary between two types. Park et al. (2007) determined using DSC that unbound water is removed first in wood-based fibers, followed by freezing-bound water and then nonfreezing-bound water. However, Almeida and Hernandez (2006) indicated that during desorption, a region exists where the loss of bound water takes place in the presence of unbound (free) water, confirming the existence of water type overlap in the drying process. According to Almeida and Hernandez (2006), the range of water content of this overlap region depends on the size distribution of fiber capillaries which, in turn depends on wood species. For the three types of woods they stated, the water content values at which the loss of bound water started in the presence of liquid water range from 40 to 56%, while the amount of free water in the wood range from 6 to 18%.

Ping et al. (2001) stated in their paper that the structure of polymer materials are altered after water sorption and consequently the mechanical and physical properties of hydrophilic polymers can show a significant change with the relative humidity in the surrounding area. Water absorption could result in the destruction of small or less perfect polymer crystallites (Gref et al., 1992). Absorbed water acts as a plasticizer in the polymer; the material glass transition temperature, as a result, decreases dramatically with the increase in water content in the polymer (Rault et al., 1994).

On the other hand, absorbed water does not act thermodynamically the same way as free water, except probably for the "free water" (unbound water that acts like bulk water) which is not in direct interaction with the cellulose sorbent. Three types of water have been identified to be present in a cellulose-water system as stated in a previous section: non-freezing bound water; freezing bound water and free water. The non-freezing bound water does not freeze (even when the sample is cooled down to $-100^{o}C$); the freezing bound water crystallizes at temperatures lower than the normal freezing temperature of water while free water freezes at $0^{o}C$ – the normal freezing temperature of water (Ping et al., 2001). This deviatory behavior of water absorbed by cellulose has been explained by the "effect of capillary condensation, the confinement of water clusters by polymer chains" (Cuperus et al., 1992, Arndt and Zander, 1990) or the strong interactions of the water molecules with the polymers or with the first hydration layer (Filho and Bueno, 1992, Scherer, et al., 1985).

2.4.4.5 Hysteresis Cause

It has been mentioned in Section 2.3.2.3 that hysteresis is observed between the wetting and drying of cellulose materials. In simple terms, hysteresis is the gap observed between the water content value when moisture is being added and when it is being removed at the same relative humidity value. The exact causes of hysteresis might not yet be known certainly. However, according to Howson (1954) the following is the mechanism. When dry cellulose is exposed to a source of moisture, molecules of water are adsorbed which will change the nature of the dry fiber causing swelling of the (hemicellulose) network. This swelling breaks secondary bonds between the molecules of the network and causes an increase in the number of hydroxyl groups acting as strong sorption sites with increasing relative humidity. If following this moisture uptake, drying/desorption is allowed to occur, the network tends to contract. This compression is delayed, however, by the persistence of bound water on the new sorption sites formed upon absorption of water molecules. This is what leads to a higher moisture content at a given relative vapor pressure upon drying which follows water uptake.

Thus, hysteresis can be thought of as a delay in the loss of water that has been absorbed by cellulose fibers resulting in higher moisture content at a given relative humidity.

2.4.4.6 Sorption Isotherms

The relationship between water content and relative humidity of a material at equilibrium can be depicted by a curve, commonly called moisture sorption isotherm. For each humidity value, a sorption isotherm indicates the corresponding water content value at a given, constant temperature (Bell and Labuza, 2000).

The general form of sorption isotherms is the same for all kinds of cellulose materials and is sigmoid in shape. This sigmoid shape, however, is not unique to cellulose materials (Christensen and Giertz, 1965).

Caulfield (1977) suggested that the fiber-water interaction structured the water around the fiber. When moisture is taken up by cellulose fibers, only the first layer is strongly attracted by the surface while the water layers absorbed subsequently are attached to the first layer (bound to the cellulose surface) and thus do not interact with the cellulose surface directly (Caulfield, 1977). The attraction in subsequent layers becomes weakened with more layers until the layers are “destructured” by the surrounding water (Caulfield, 1977). Goring (1977), on the other hand, has a different view of the water-fiber interaction model postulated by Caulfield (1977).

According to Goring (1977), the cellulose fibers disrupt the water structure. The water molecules surrounding the cellulose surface consist of a disordered layer which is evidenced by the lack of “hydrogen bonded ring structures” found in the unperturbed layer (Goring, 1977). This should imply that the first layer adsorbed to the cellulose surface is different from the subsequent layers. Hernádi (1984) concurred on the issue and explained the “S” shaped adsorption isotherms of cellulose using this difference between the layers of water absorbed. According to Hernádi (1984), the following isotherm given in Figure 4 is a generally accepted one for cellulose fibers.

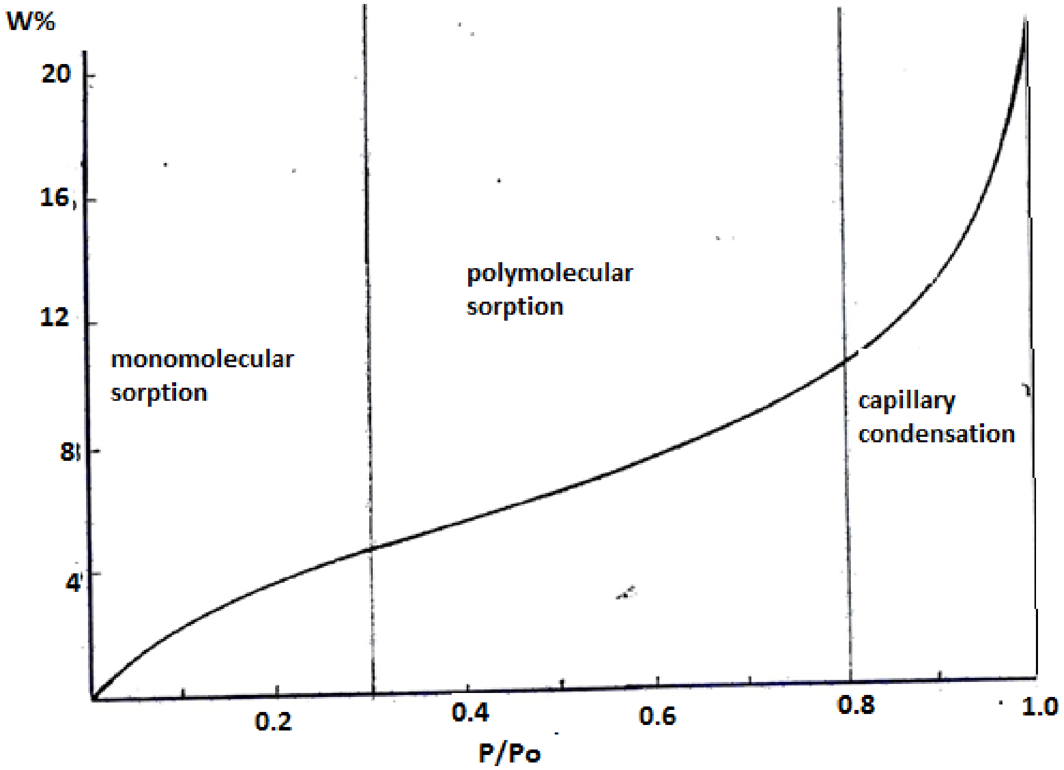


Figure 4: Typical Cellulose Fiber Adsorption Curve (Hernádi, 1984)

As can be seen from Figure 4, Hernádi (1984) explained that if the relative partial water vapor pressure is between 0 – 0.3 (or 0% - 30% relative humidity), a monomolecular absorption takes place which is accompanied by strong heat generation. Between 0.3 – 0.8 of relative vapor pressure, additional water molecules are attached on the first water layer leading to a polymoelcular sorption and the sorption is accompanied by heat generation. However, the heat generation in this relative humidity range is less than in the previous case (when the relative humidity is between 0% and 30%). It is known that water “imbibition” (absorption) by cellulose is an exothermic process (Christensen and Giertz, 1965). Between 0.8 – 1.0 of relative partial water vapor pressure (or 80% - 100%

relative humidity), however, the absorption occurs due to capillary condensation and no heat is generated (Hernádi, 1984).

Christensen and Giertz (1965) illustrated the effect of relative humidity on the moisture uptake by cellulose fibers by using the isotherms in Figure 5.

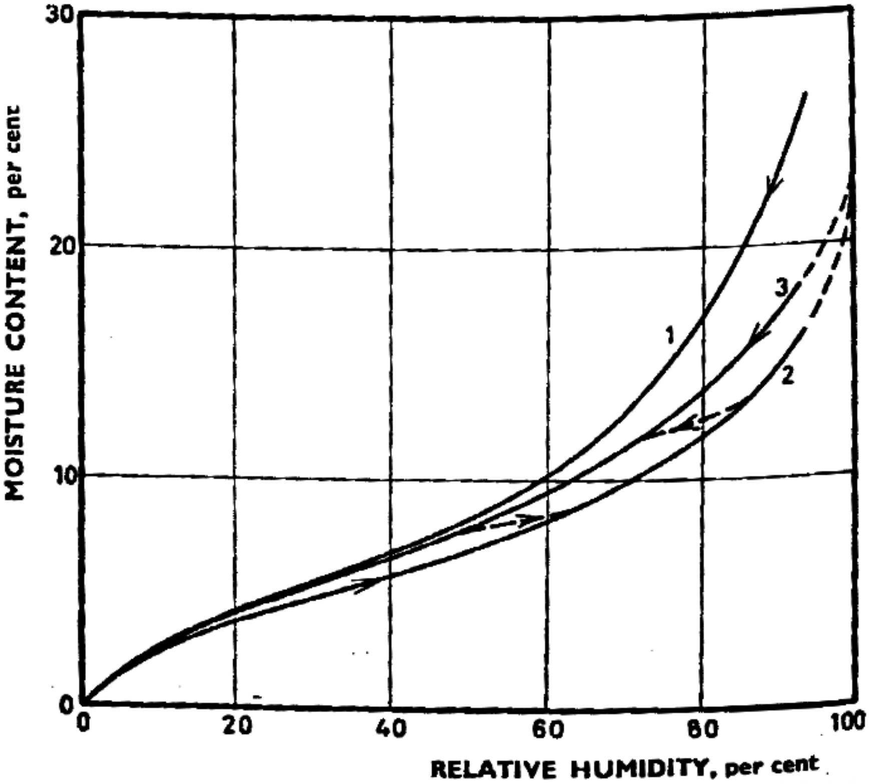


Figure 5: Desorption and Adsorption/Absorption Isotherms of a Bleached Sulphite Pulp (Seborg et al., 1938)

Also, on the basis of the experimental data and data from the literature on adsorption of standard cotton fibers, Hernádi (1984) plotted a curve and concluded that the relationship between water take-up and partial vapor pressure can be expressed by bi-linear lines (two straight lines). Figure 6 shows the relationship between bonded water and partial vapor pressure presented by Hernádi (1984). Hernádi (1984) explained the break points by a change in sorption mechanism, i.e, the first portion of the straight line represented the build-up of monomolecular layer while in the second part polymolecular adsorption takes place as water molecules are attached on the already adsorbed monomolecular layers. The break point in this curve occurs between 5 – 10% water content. This value roughly agrees with the free water incorporation point of about 5% determined by Nelson (1977) for cotton linters.

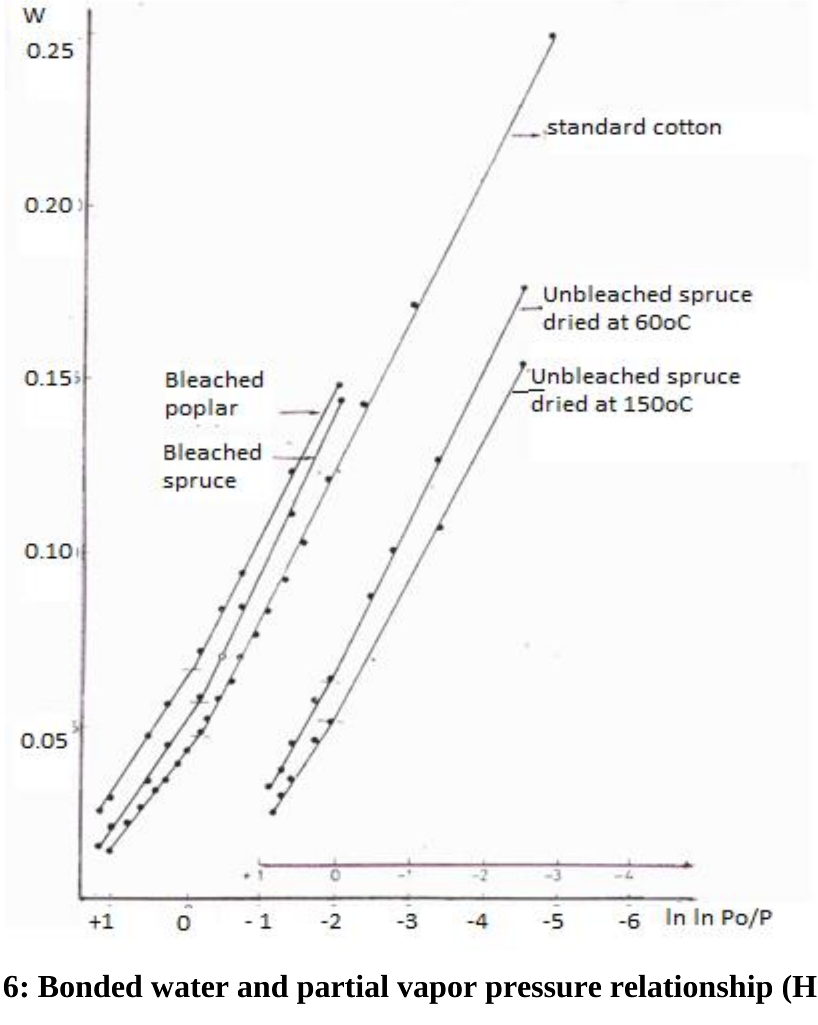
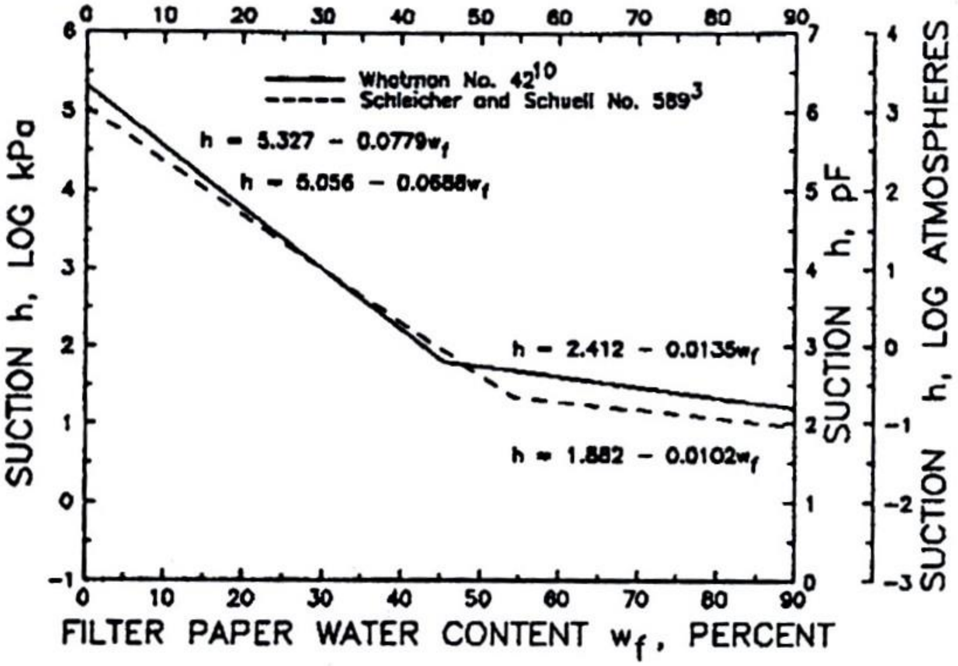


Figure 6: Bonded water and partial vapor pressure relationship (Hernádi, 1984)

ASTM D5298 – 10 (2010) also describes a typical calibration curve (suction versus filter paper water content) as two-segmented. Figure 7 shows wetting calibration curves for Whatman No. 42 and Schleicher and Schuell 589^2 filter papers given by the ASTM D5298 – 10 (2010).



NOTE 1—Coefficient of determination r >0.99.

Figure 7: Calibration of filter papers (ASTM D5298 – 10 (2010))

The ASTM D5298 – 10 (2010) calibration curve depicted in Figure 7 is a combination of both wetting and drying curves (Bicalho et al., 2010). The upper segment of the curve is constructed from an initially dry filter paper in a wetting experiment while the lower segment from an initially wet filter paper in a drying experiment. The calibration data for the wetting segment of the curve was obtained using non-contact vapor method using salt solutions whereas the lower drying segment using pressure plate or membrane device (contact method) (McQueen and Miller, 1968).

ASTM D5298 – 10 (2010) states that the upper segment of the calibration curves shown in Figure 7 represents moisture retained as water films adsorbed to particle surfaces, while the lower segment represents moisture retained by capillary or surface tension forces between particles.

CHAPTER III

EXPERIMENTAL PROGRAM

Second part of this research study has been the experiments undertaken to obtain the data needed to construct calibration curves for two types of filter papers. Consequently, the experimental test results are used to evaluate the interaction between the water and the cellulose structure in the filter paper. This section outlines the details of the materials and equipment used and the experimental protocols adopted in this study.

3.1 Materials and Apparatus

3.1.1 Filter Papers

The filter papers used in this study are Whatman No. 42 and Schleicher and Schuell No. 589^2 quantitative filter papers. Both papers are in ASTM D5298 -10 (2010) among the filter papers listed for soil suction measurements. Manufacturer's website indicates that Whatman No. 42 cellulose filters are manufactured from high quality cotton linters which have been treated to achieve a minimum alpha cellulose content of 98%. Schleicher and Schuell No. 589^2 filter papers also contain between 97-98% alpha cellulose and their ash content is less than 0.007%. The filter papers used in the first two sets of experiments were used from the same boxes (Cat. No. 1442055, H11324793 for Whatman and Ref.

No. 10300107, Lot ES 0166-1 for Schleicher and Schuell No. 589^2) while those used in the third and fourth sets were used from the same boxes of (Cat. No. 1442055, H11324793 for Whatman and Ref. No. 10300107, Lot ES 0166-1 for Schleicher and Schuell No. 589^2).

3.1.2 Salt Solutions

Sodium chloride (NaCl) was used to prepare solutions of different salt concentrations. Concentrations between 0 (de-ionized water) and 2.2 molality were prepared to generate osmotic suctions of known values. Table 2 shows the solutions used with their concentrations.

Table 2 Salt Solutions, Concentrations and Equivalent Osmotic Suction Values

Solution (Molality)	Osmotic Suction (kPa)	NaCl (grams per liter of de-ionized water)
0.000	0.000	0.00000
0.002	9.757	0.11688
0.005	24.195	0.29221
0.020	95.024	1.16885
0.200	916.076	11.66849
1.000	4646.912	58.44247
2.200	10887.347	128.57343

3.1.3 Glass Jars and Plastic Cups

Glass jars of approximately equal size were used in which were put salt solutions and filter papers in a non-contact manner. The diameter of the glass jars is used is 90 mm and their height is 95 mm. Plastic cups were used as a support to hold the filter papers and separate the papers from the solution in the jars since vapor transfer method (non-contact method) is employed to equilibrate the filter papers and salt solutions. Figure 8 shows a schematic diagram of a glass jar with a plastic cup in it holding a filter paper.

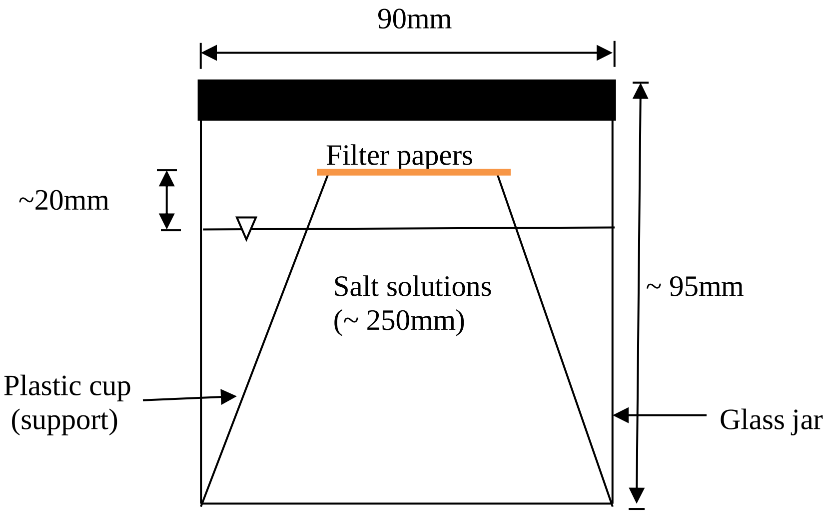


Figure 8: Schematic diagram of testing configuration

3.1.4 Temperature-Controlled Chamber

A constant-temperature chamber was used to keep the jars in isothermal conditions throughout the equilibration period. Figure 9 shows a schematic diagram of the chamber used in this study. The temperature inside the chamber was maintained constant at 25 ±0.1^{o}C throughout the equilibration period. The chamber was also kept in a constant-temperature room 10ft by 10ft, where the temperature is maintained constant with a fluctuation of ±1^{o}C. The chamber is seated on a wooden support 15 cm high above the ground. The chamber is designed in such a way that everything is submerged under water (i.e., water circulates all around the inside container (which is also sealed) holding the sealed glass jars)

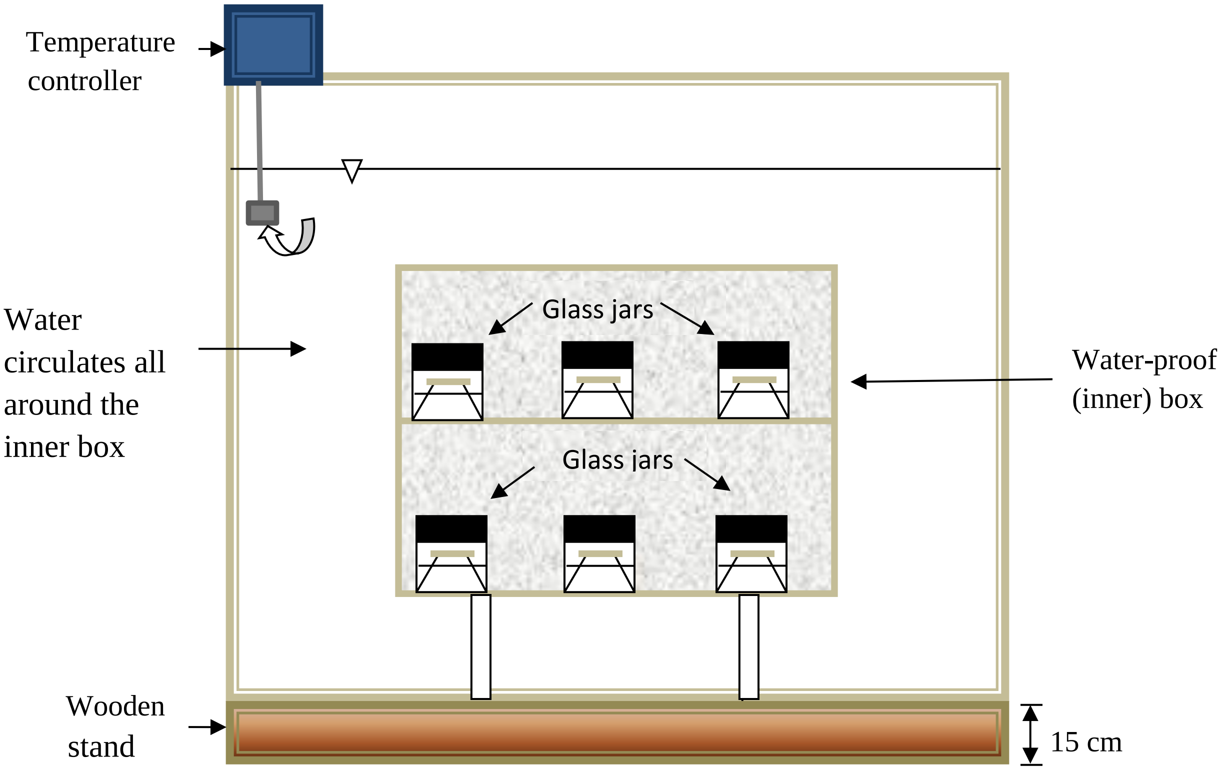


Figure 9: Temperature-controlled Chamber

3.1.5 Balance

A digital Sartorius CPA Analytical Balance CPA 225D accurate to 0.00001g was used to measure the wet and dry weight of the filter papers and the moisture tins for the purpose of calculating filter paper water contents.

3.1.6 Aluminum Containers

Aluminum moisture tins with lids were used to hold the filter papers throughout the measuring of the wet weights, oven-drying and dry-weight measurement after they were removed from the constant-temperature chamber. The outside of these moisture tins and their corresponding lids were labeled alphanumerically to avoid any mix-up as shown in Figure 10.

Figure 10 Labeled moisture tin

3.1.7 Convection Oven

A gravity convection oven was used to oven-dry the filter papers at a temperature of 105 ±5^{o}C.

3.1.8 Aluminum Block

An aluminum block was used as a heat sink to expedite the cooling of the moisture tins holding filter paper immediately after removal from the oven and before oven-dried weight measurements were taken.

3.1.9 Tweezers

Clean tweezers were used to handle the filter papers.

3.1.10 Miscellaneous Tools and Supplies

Gloves and electrical tape to seal the jars during equilibration period are needed to complete the experiment.

3.2 Experimental Methods

Four sets of experiments were run with different equilibration periods. Equilibration periods of 31, 48, 76 and 42 days were allowed for the 1st, 2nd, 3rd and 4th sets, respectively. Calibration curves should have a high accuracy which in turn depends on the careful setting up of the experiment, strict maintenance of isothermal conditions, minimizing the exposure of the filter papers to the environment other than the temperature-controlled environment and accuracy in measuring the weights of the wet and oven-dried filter papers which will be used in the calculation of water contents. The experiments in this research were conducted accordingly with utmost care and using a temperature-controlled environment/chamber with fluctuation limited to $\pm 0.1^{o}$C for the equilibration and a balance with 0.00001 accuracy for measuring weights of filter papers. Also, no single experiment was undertaken by a single person; setting up the jars and measuring filter paper weights were invariably conducted by a group of two or three persons at a time to minimize the exposure of filter papers to the lab environment – when one person opens the jars, the other puts the filter papers in the moisture tins and weighs them while the third person records the weights. The exposure time of the filter papers to the lab environment was between 4 – 7 seconds while transferring them to the moisture tin and measuring their wet weight.

3.2.1 First Set with Equilibration Period of 31 Days

In this set of experiment, 28 glass jars were used in each of which were put two same-type filter papers (i.e., either Whatman No. 42 or Schleicher and Schuell No. 589^2). Half of the jars were used for the drying filter paper calibration curve data while the remaining half for the wetting filter paper calibration data.

For the drying calibration curves data, initially wet/saturated filter papers were used. The papers were soaked in de-ionized water for a number of days to make them saturated. For this first set of experiment, half of the filter papers were saturated in de-ionized water for 4 days and the remaining half for 18 days.

For the wetting calibration curves, initially dry filter papers, i.e, filter papers from the original boxes were allowed to absorb moisture from the salt solution in the jars. The average water content of the filter papers from their boxes in the laboratory environment was measured to be around 9% and 10%, for Whatman No. 42 and Schleicher and Schuell No. 589^2 papers, respectively.

The following seven salt (NaCl) solutions were used to generate suctions: 0.0 (de-ionized water), 0.002, 0.005, 0.02, 0.2, 1.0 and 2.2 Molalities. The osmotic suction of different concentration solutions is calculated using the following equation at $T = 25^oC$, $R = 8.3143$ J deg K^{-1} mole $^{-1}$ and $v = 2$:

$$h_\pi = -vRTm\varphi \qquad (2)$$

where h_π = osmotic suction; v = number of ions from one molecule of salt (2 for NaCl); R = universal gas constant (8.3143J deg/K mole); T = absolute temperature; m = molality and φ = osmotic coefficient. Table 3 gives the relationship between osmotic suction and NaCl molality using Equation 2.

The jars were marked with black electric tapes at the same heights so that the solutions are filled to equal levels and consequently the gap between the solution and the filter papers is uniform in all the jars at an approximate distance of 20 mm. The volume of solution/ de-ionized water in each jar was approximately 250 ml. The volume of the jars is about 510 ml.

Plastic cups were put in to the jars to serve as a support so the filter papers are suspended over the solutions. The jars were filled with different concentrations of solutions prepared to the required levels which were already marked with black electric tape. Funnels were used to channel the solutions from their respective containers into the jars without contaminating the tops of the plastic cups where the filter papers are to be placed.

Table 3 Osmotic Suction and Corresponding NaCl Amount per Volume of De-ionized Water

Molality (m)	Osmotic Coefficient	Suction h (kPa)	Suction h(log kPa)	NaCl (g/liter)	NaCl (g/2 liters)	NaCl (g/3 liters)	NaCl (g/4 liters)	NaCl (g/5 liters)
0.000	1.00000	0	0	0.00000	0.00000	0.00000	0.00000	0.00000
0.001	0.98840	4.900	0.690	0.05844	0.11688	0.17533	0.23377	0.29221
0.002	0.98402	9.757	0.989	0.11688	0.23377	0.35065	0.46754	0.58442
0.005	0.97604	24.195	1.384	0.29221	0.58442	0.87664	1.16885	1.46106
0.010	0.96804	47.994	1.681	0.58442	1.16885	1.75327	2.33770	2.92212
0.020	0.95832	95.024	1.978	1.16885	2.33770	3.50655	4.67540	5.84425
0.050	0.94357	233.902	2.369	2.92212	5.84425	8.76637	11.68849	14.61062
0.100	0.93250	462.316	2.665	5.84425	11.68849	17.53274	23.37699	29.22123
0.200	0.92387	916.076	2.962	11.68849	23.37699	35.06548	46.75397	58.44247
0.300	0.92123	1370.187	3.137	17.53274	35.06548	52.59822	70.13096	87.66370
0.400	0.92106	1826.579	3.262	23.37699	46.75397	70.13096	93.50795	116.88494
0.500	0.92224	2286.149	3.359	29.22123	58.44247	87.66370	116.88494	146.10617
1.000	0.93729	4646.912	3.667	58.44247	116.88494	175.32740	233.76987	292.21234
1.200	0.94567	5626.151	3.750	70.13096	140.26192	210.39288	280.52385	350.65481
1.400	0.95491	6627.977	3.821	81.81946	163.63891	245.45837	327.27782	409.09728
1.500	0.95980	7137.769	3.854	87.66370	175.32740	262.99111	350.65481	438.31851
1.600	0.96487	7653.838	3.884	93.50795	187.01590	280.52385	374.03180	467.53974
1.800	0.97545	8704.985	3.940	105.19644	210.39288	315.58933	420.78577	525.98221
2.000	0.98657	9782.467	3.990	116.88494	233.76987	350.65481	467.53974	584.42468
2.200	0.99818	10887.350	4.037	128.57343	257.14686	385.72029	514.29372	642.86715

Since two types of filter papers, Schleicher and Schuell No. 5892 and Whatman No. 42, are used in this set, each concentration of solution is filled in 4 jars – for drying and wetting calibration curves using the two types of filter papers. Thus, a total of 28 jars were set up. Table 4 below shows the number of jars used with the concentration of solution and the filter paper type in each.

Table 4 Number of Jars and Solution and Filter Paper Arrangement (1st set)

Solution (Molality)	Suction value in kPa	Whatman No. 42		Schleicher and Schuell 589[2]	
		No. of jars*		No. of jars*	
		Drying**	Wetting	Drying**	Wetting
0.000	0.000	1	1	1	1
0.002	9.757	1	1	1	1
0.005	24.195	1	1	1	1
0.020	95.024	1	1	1	1
0.200	916.076	1	1	1	1
1.000	4646.912	1	1	1	1
2.200	10887.347	1	1	1	1

* Each jar contains two filter papers. Two were used for precautionary purpose.

** In each of the jars labeled for drying papers, two soaked filter papers were put: the bottom ones were from the group saturated for 4 days whereas the top papers came from the group saturated for 18 days.

Filter papers were carefully placed on the plastic cups in the jars using tweezers without the filter papers touching anything including the sides of the jars. The filter papers were never touched by bare hands; clean tweezers and gloves were used to avoid contamination of the papers which might affect their water absorption behavior. In line with the recommendation of ASTM D5298 – 10 (2010), the edge of the top filter papers was bent up to facilitate its removal from the jar after the equilibration period has been reached. The jars were closed with their lids and then sealed with electric tape to preclude moisture exchange between the inside and outside of the jars. Each jar was put in yet another container which in turn was sealed and placed in the temperature – controlled chamber. The chamber is filled with water and the temperature inside was maintained constant at 25^{o}C throughout the equilibration period of 31 days, with temperature fluctuations of ±0.1^{o}C. The chamber is designed in such a way that everything is submerged under water (i.e., water circulates all around the inside container (which is also sealed) holding the sealed glass jars) as shown in Figure 8. During this period, the filter paper and the aqueous solution will come to suction equilibrium.

At the end of the equilibration period, the jars were removed from the temperature-controlled chamber following which the filter papers were removed from the jars and each were put in a moisture tin with a pre-determined empty weight. One glass jar was removed from the water bath at a time. As per the suggestion by ASTM D5298 – 10 (2010), the empty weight of the moisture tins was taken immediately prior to determining the wet weight of the tins with a filter paper in them. The weights of the wet filter papers and the moisture tins were then taken and the moisture tins with the filter papers inside were placed in an oven with their lids half open to allow evaporation. After allowing the moisture in the papers to evaporate overnight in the oven, the weights of both the dry filter paper with the tins and subsequently the empty hot tins were taken from which are calculated the water contents of each of the filter papers. The results of these measurements are presented and discussed in Chapter IV.

3.2.2 Second Set with Equilibration Period of 48 Days

The same procedure as for the first set was used for the second set of experiment with the following changes made to this latter set.

Thirty two jars were used in this set of experiment. Only five concentrations of NaCl solutions were used; the two highest concentrations used in the first set (1.5 and 2.2 molality) were omitted to make room for more number of tests for the lower suction measurements including zero suction. Repeated experiments (more number of jars) were set up for the lower concentration solutions in this set. Table 5 shows the number of jars used with the concentration of solution and the filter paper type in each. Equilibration period for this set was 48 days. Results of the water content measurements with the associated suction values are presented and discussed in Chapter IV.

Table 5 Number of Jars and Solution and Filter Paper Arrangement (2nd set)

Solution (Molality)	Whatman No. 42		Schleicher and Schuell No. 589^2	
	No. of jars*		No. of jars*	
	Drying	Wetting	Drying	Wetting
0.000	2	2	2	2
0.002	2	2	2	2
0.005	2	2	2	2
0.020	1	1	1	1
0.200	1	1	1	1

* Each jar contains two filter papers. Two were used for precautionary purpose.

3.2.3 Third Set with Equilibration Period of 76 Days

The same procedure as for the first set was used for the third set of experiment with the following changes made to this set.

Only four concentrations of NaCl solutions were used; the two highest concentrations used in the first set (0.2 , 1.5 and 2.2 Molality) were omitted to make room for more number of tests for the lower suction measurements including zero suction. Table 6 shows the number of jars used with the concentration of solution and filter paper type in each. Equilibration period for this set was 76 days. Results of the water content measurements with the associated suction values are presented and discussed in Chapter IV.

Table 6 Number of Jars and Solution and Filter Paper Arrangement (3rd set)

Solution (Molality)	Whatman No. 42		Schleicher and Schuell No. 589[2]	
	No. of jars*		No. of jars*	
	Drying	Wetting	Drying	Wetting
0.000	3	3	3	3
0.002	2	2	2	2
0.005	2	2	2	2
0.020	1	1	1	1

* Each jar contains two filter papers. Two were used for precautionary purpose.

3.2.4 Fourth Set with Equilibration Period of 42 Days

The same procedure as for the first set was used for this last set of experiment with the following changes made to this set.

Thirty two jars were used with double jars used for each of the measurements involving zero suction solutions, i.e, de-ionized water. Table 7 shows the number of jars used with the concentration of solution and filter paper type in each. Equilibration period was 42 days. Results of the water content measurements with the associated suction values are presented and discussed in Chapter IV.

Table 7 Number of Jars and Solution and Filter Paper Arrangement (4th set)

Solution (M olality)	Whatman No. 42		Schleicher and Schuell No. 589^2	
	No. of jars		No. of jars	
	Drying	Wetting	Drying	Wetting
0.0	2	2	2	2
0.002	1	1	1	1
0.005	1	1	1	1
0.02	1	1	1	1
0.2	1	1	1	1
1.0	1	1	1	1
2.2	1	1	1	1

* Each jar contains two filter papers. Two were used for precautionary purpose.

3.2.5 Initial Water Content of Saturated/Soaked Filter Papers

To determine the maximum water uptake by the filter papers upon soaking, and also to identify any significant difference between the saturated water content values (of the different soaking periods), the water content of saturated/soaked filter papers was measured. For this purpose, filter papers from each of the Whatman No. 42 and Schleicher and Schuell No. 589^2 types were soaked for 4 days, for 18 days and for 1 day (26 hours). The results of these measurements are presented in Section 4.3.

CHAPTER IV

TEST RESULTS

Water content values were calculated from the measured weights of the wet and oven-dried filter papers. The measured weights of each filter paper and the water content values calculated are presented in Table 9 through Table 12 in Appendix A. Summarized water content versus total suction data is also presented in Table 13 through 16 in Appendix B. The water content values were plotted against the suction values for both the Whatman No. 42 and Schleicher and Schuell No. 589[2] papers. These plots are the calibration curves for the respective papers. The "full" range of suction has been used only in the 1st and 4th sets of experiments (those with equilibration period of 31 and 42 days). Repeated measurements were taken for the lower suction solutions in the 2nd and 3rd sets of experiments. The "full" suction range included values ranging from 0 kPa (0 Molality NaCl) to nearly 11,000 kPa (2.2 molality NaCl) as shown in Table 2.

To plot the calibration curves, the water content values for filter papers from the same jar were averaged. In doing so, a few water content values from filter papers from the same jar significantly different from each other have been omitted. ASTM recommends the test results be discarded if the suction difference between two papers exceeds 0.5 log kPa.

The following are among values that have been omitted:

- Water content values of 0.044 and 0.379 from the jar containing 0.02 M solution set up for the wetting experiment of Whatman No. 42 papers in the 1st set of experiments;
- Water content values of 0.400 and 0.753 from the jar containing 0.02 M solution set up for the drying experiment of Whatman No. 42 papers in the 2nd set of experiments;
- Water content values of 1.828 and 2.154 from the jar containing 0.2 M solution set up for the drying experiment of Schliecher and Schuell No. 589^2 papers in the 4th set of experiments

4.1 Drying Calibration Data and Trends

In plotting drying calibration data, outliers were excluded. The criterion used to discard data values has been if the values were higher than the maximum initial saturated/soaked water content values measured for the respective types of filter papers as identified in Section 4.3.

4.1.1 Whatman No. 42 Filter Papers

The drying calibration "curves" for Whatman No. 42 filter papers are shown in Figures 11, 12, 13 and 14 for equilibration periods of 31, 42, 48, and 76 days, respectively. The "curves" shown are not fitted curves to the data but are "trendlines" added to better show the trend of the data.

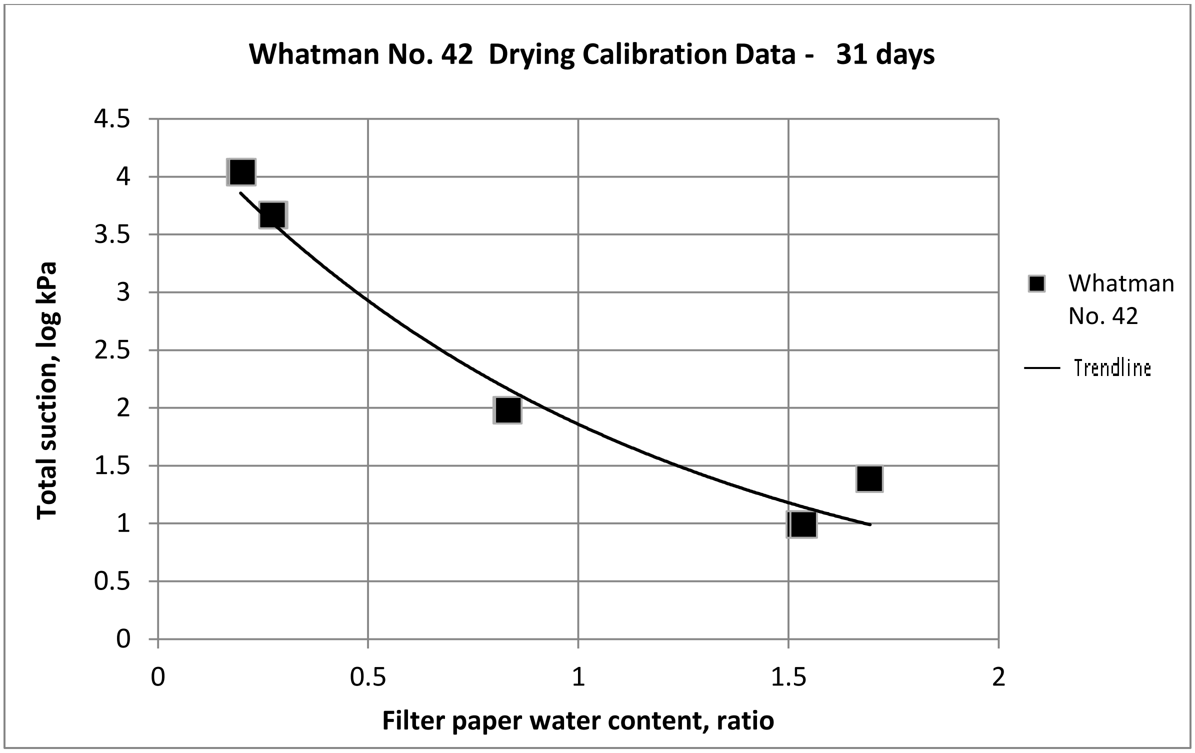


Figure 11: Drying calibration data for Whatman No. 42 filter paper (Equilibration period - 31 days)

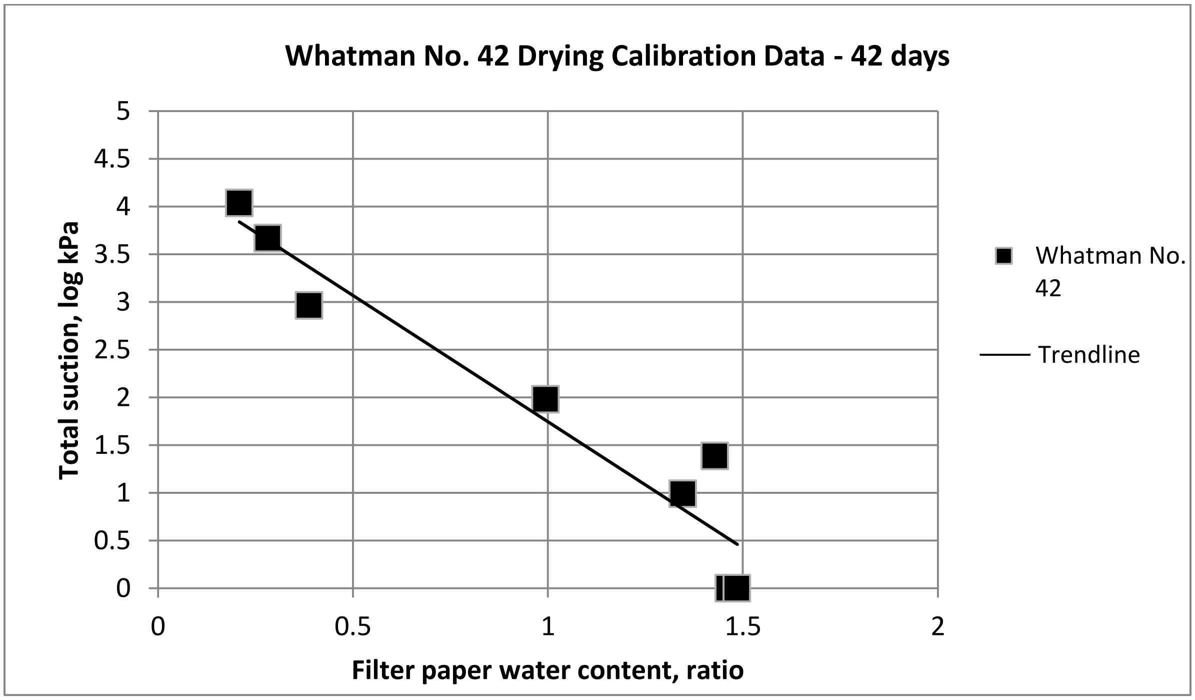


Figure 12: Drying calibration data for Whatman No. 42 filter paper (Equilibration period - 42 days)

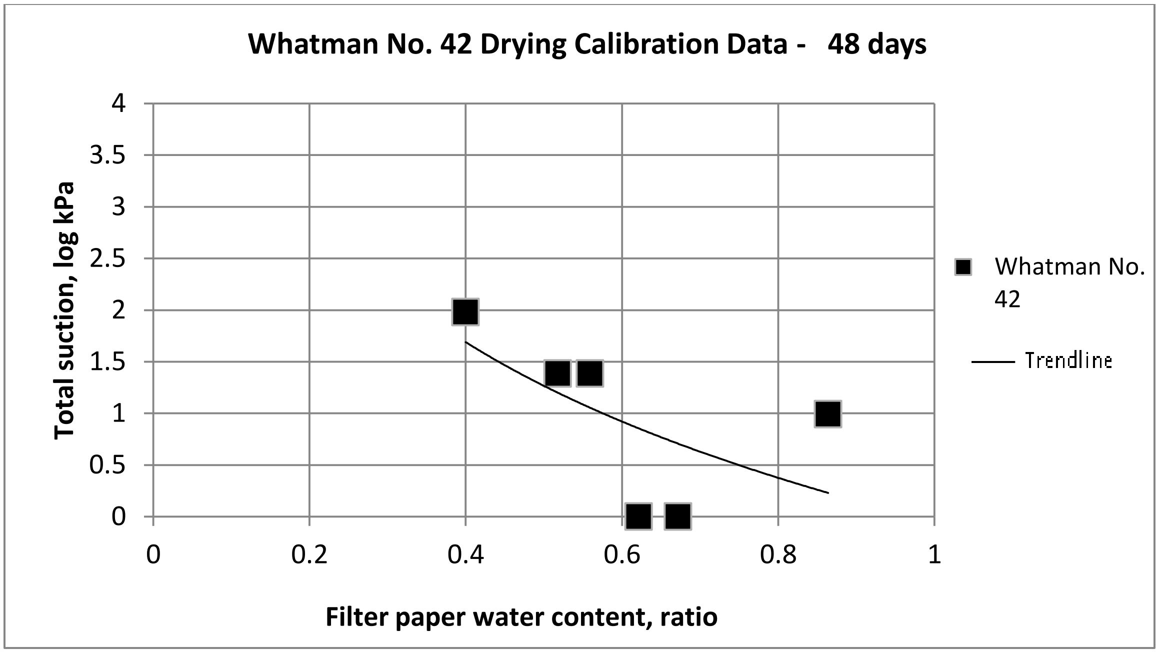


Figure 13: Drying calibration data for Whatman No. 42 filter paper (Equilibration period - 48 days)

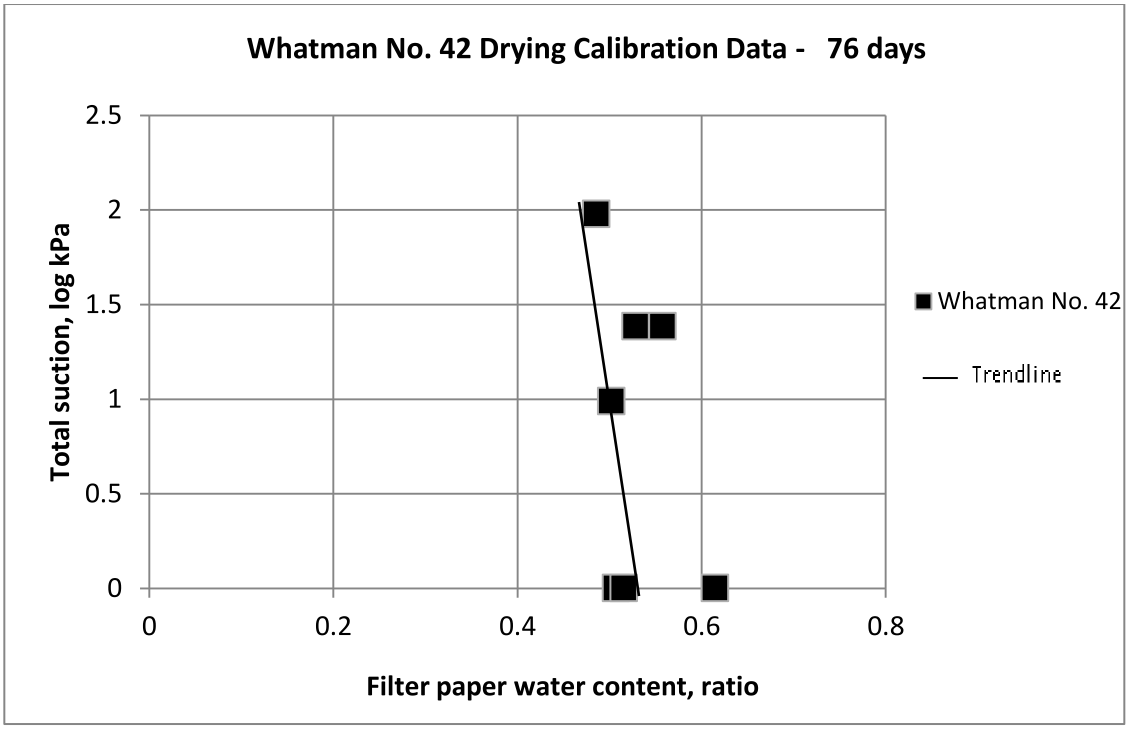


Figure 14: Drying calibration curve for Whatman No. 42 filter paper (Equilibration period - 76 days)

Although overall the drying calibration data showed scatter and high water content values in the lower suction regions, it was observed that the more time the filter papers were allowed to equilibrate with the salt solutions, the more the curves shifted inward – water content values decreased – and the scatter also diminished. Figure 15 shows the drying calibration data for each of the four sets of experiments for Whatman No. 42 filter paper.

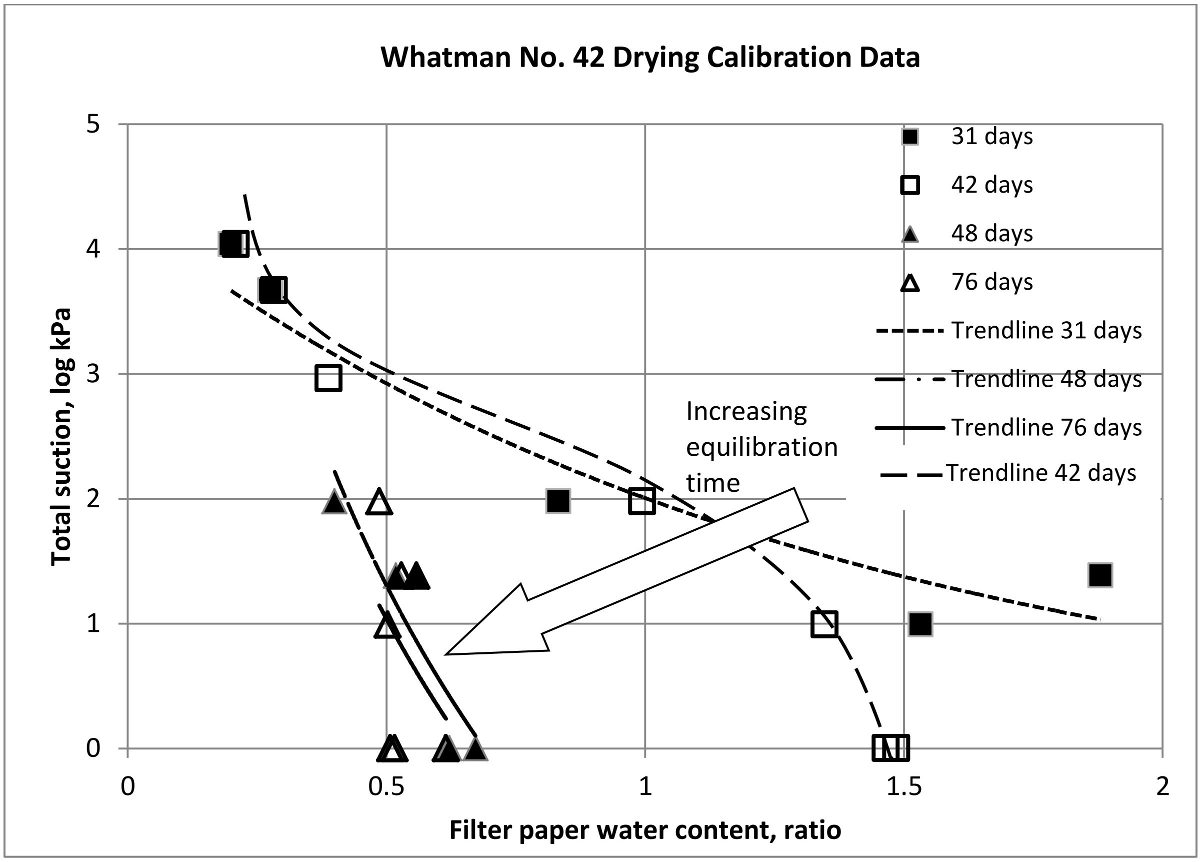


Figure 15: Drying calibration curves for Whatman No. 42 filter paper

It can clearly be seen from Figure 15 that with more equilibration time allowed, the curves move ‘inward’ i.e. water content values decreased and the data get closer to each other for the longer equilibration periods.

4.1.2 Schleicher and Schuell No. 589^2 Filter Papers

The drying calibration data for Schleicher and Schuell No. 589^2 filter papers are shown in Figures 16, 17, 18 and 19 for equilibration periods of 31, 42, 48, and 76 days, respectively.

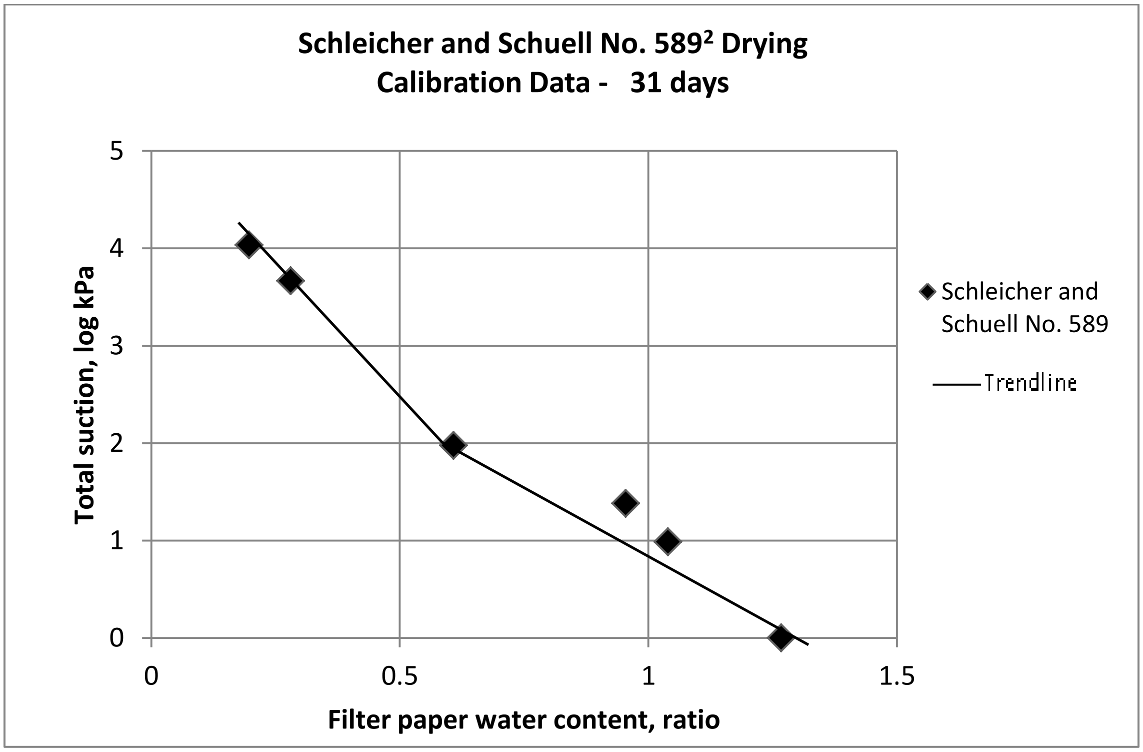


Figure 16: Drying calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period - 31 days)

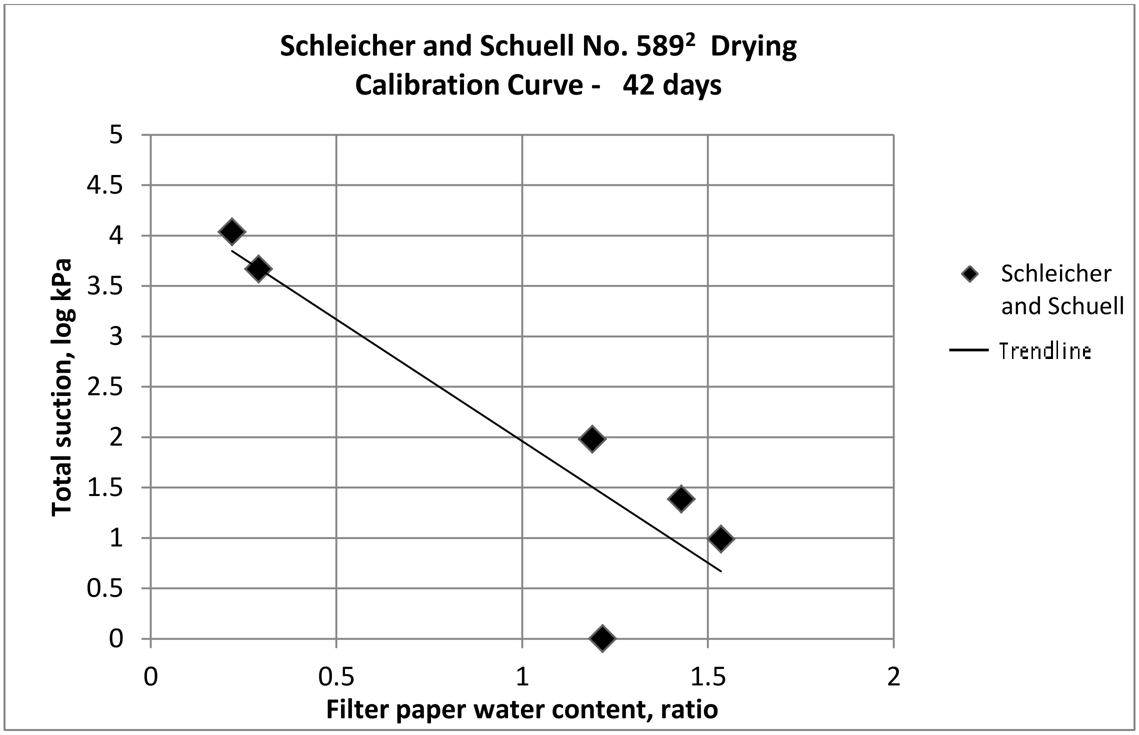


Figure 17: Drying calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period - 42 days)

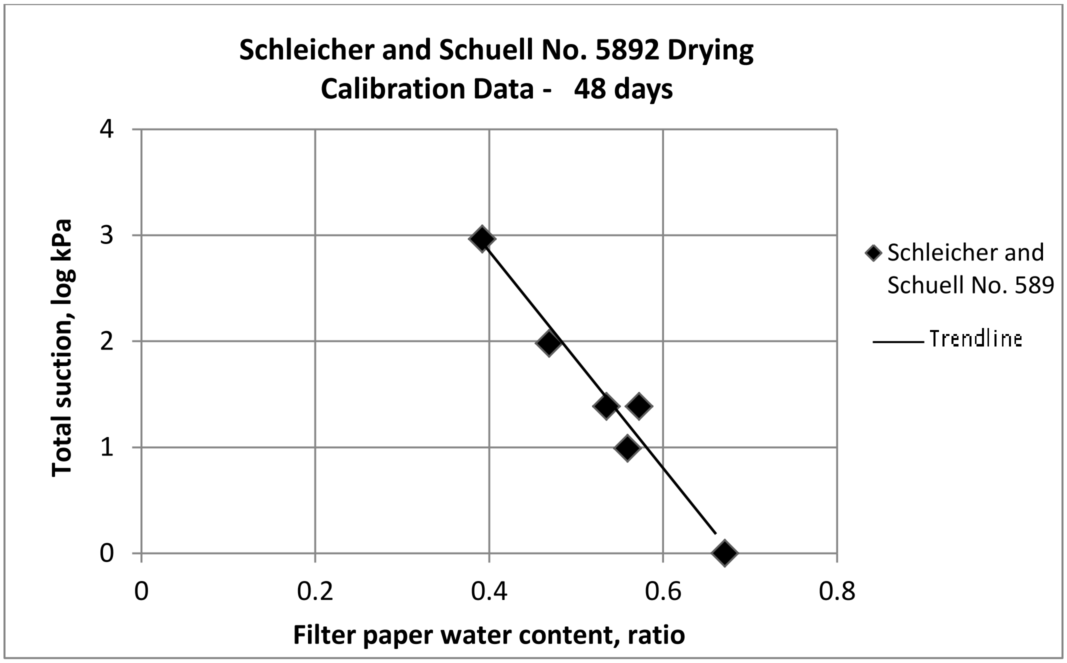


Figure 18: Drying calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period - 48 days)

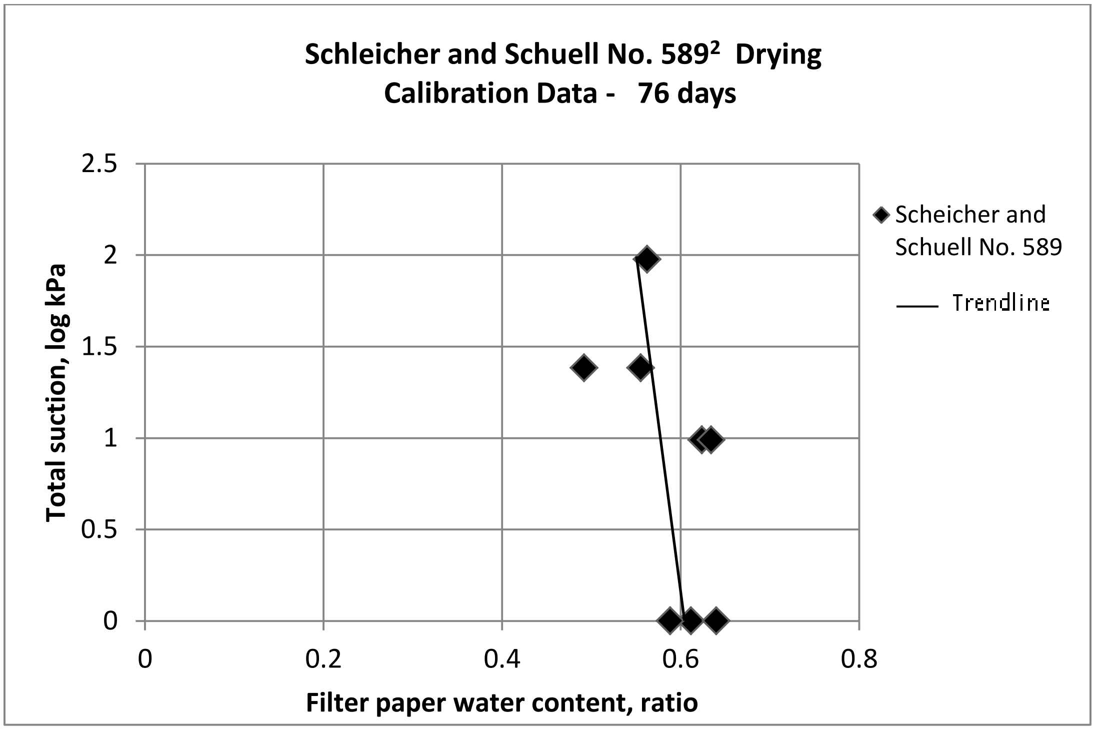


Figure 19: Drying calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period - 76 days)

As with the Whatman No. 42 papers, obvious outliners have been omitted from the water content versus total suction curves for the Schleicher and Schuell No. 589^2 papers shown in the preceding pages. The measured water content values for Schleicher and Schuell No. 589^2 filter papers show less scatter and a more defined trend than those of Whatman papers. As with the Whatman No. 42 filter papers, the curves shifted ‘inward’ with increase in equilibration period with the exception of the calibration curve from the 42-day equilibration period which shifted outward (moved to the right of the 31-day curve). Figure 20 shows the drying calibration data for each of the four sets of Schleicher and Schuell No. 589^2 filter papers studied.

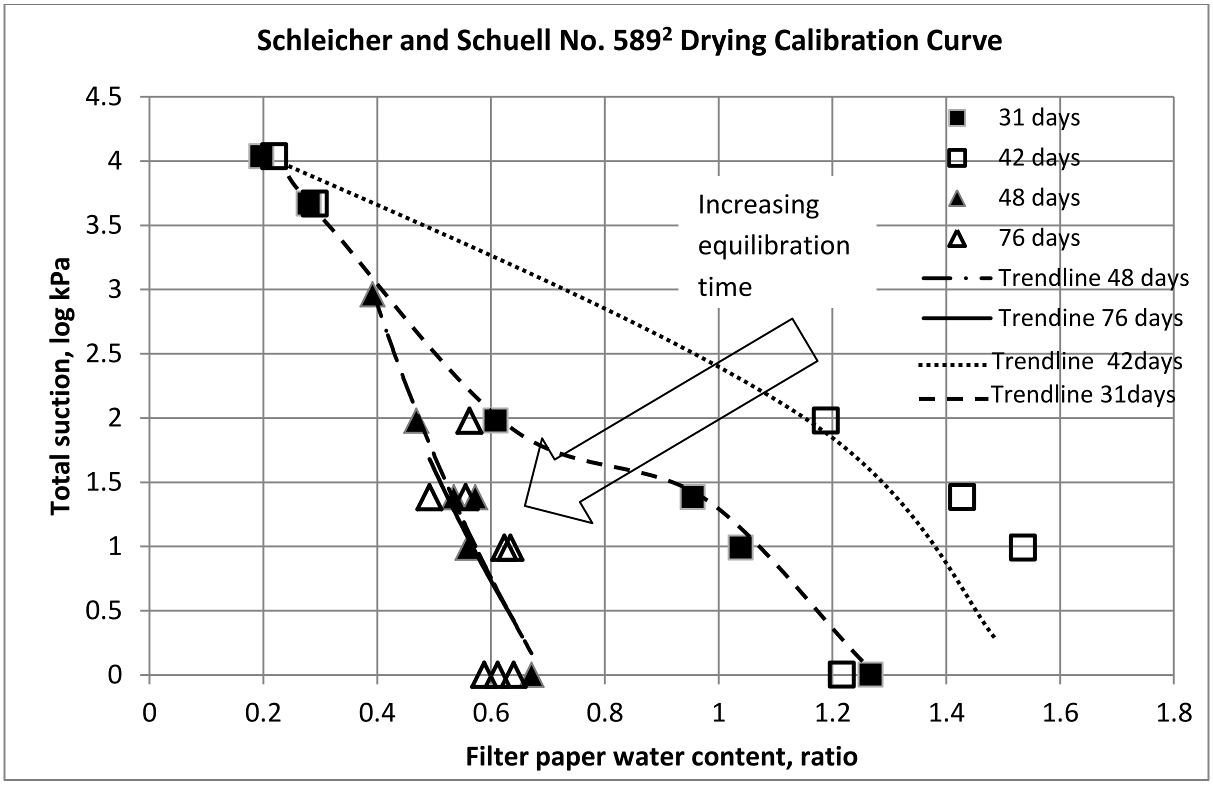


Figure 20: Drying calibration curves for Schleicher and Schuell No. 589^2 filter papers

4.2 Wetting Calibration Data and Trends

4.2.1 Whatman No. 42 Filter Papers

The water content versus total suction data for the wetting calibration data of Whatman No. 42 filter papers are shown in Figures 21, 22, 23, and 24 for equilibration periods of 31, 42, 48, and 76 days, respectively. The values shown are measured values for each filter paper (not average values).

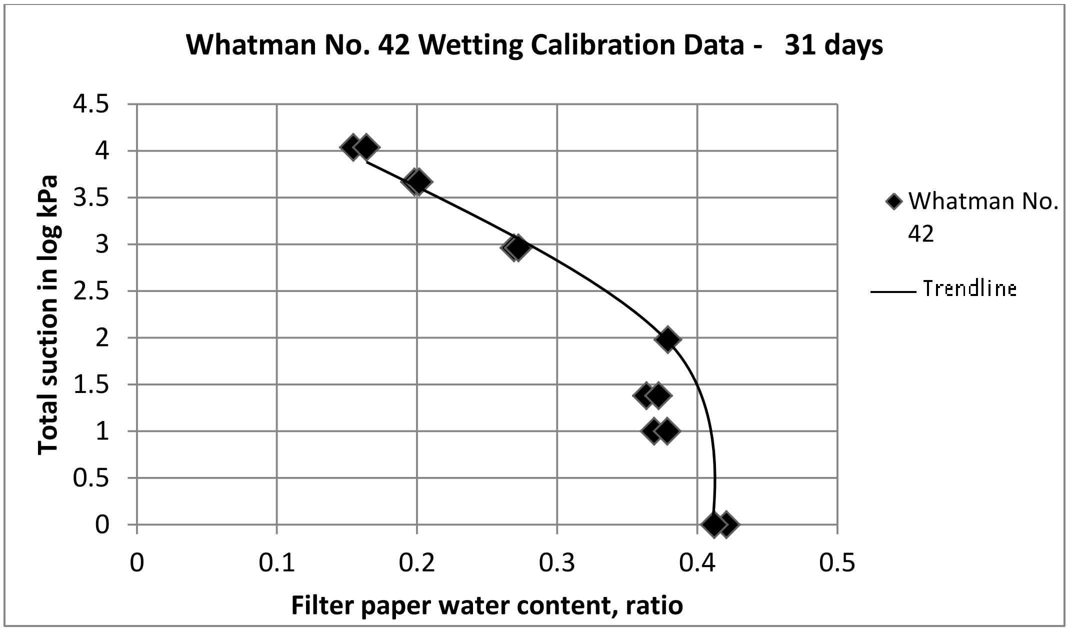


Figure 21: Wetting calibration data for Whatman No. 42 filter paper (Equilibration period – 31 days)

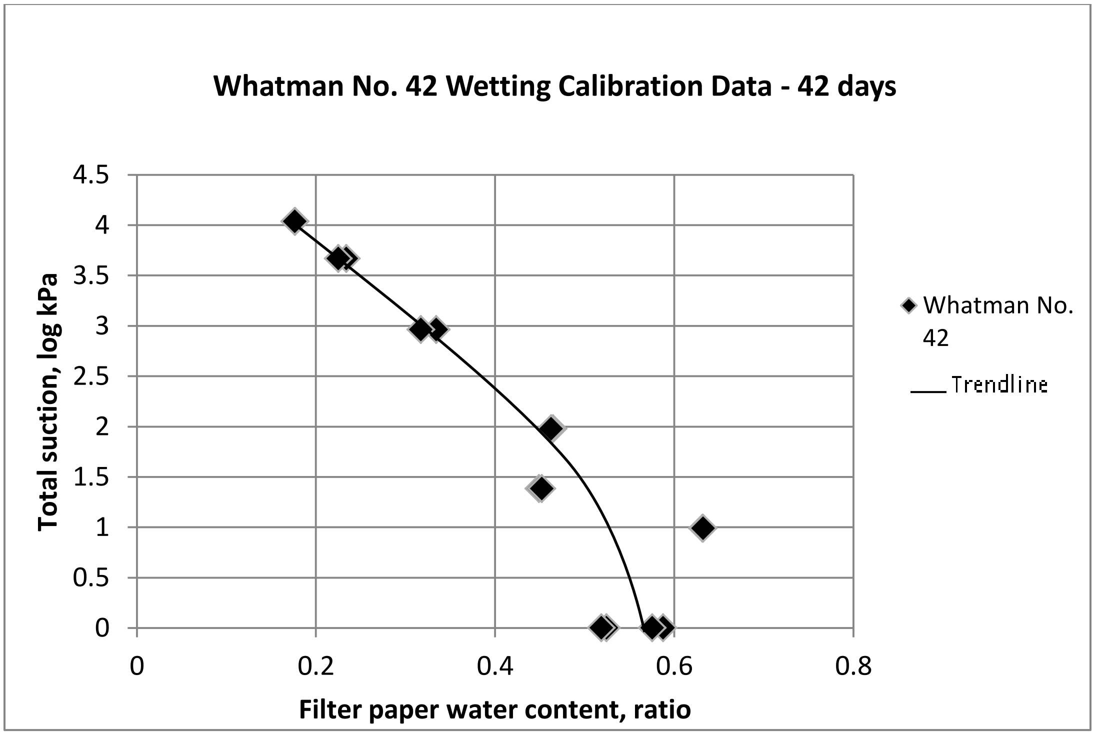


Figure 22: Wetting calibration data for Whatman No. 42 filter paper (Equilibration period – 42 days)

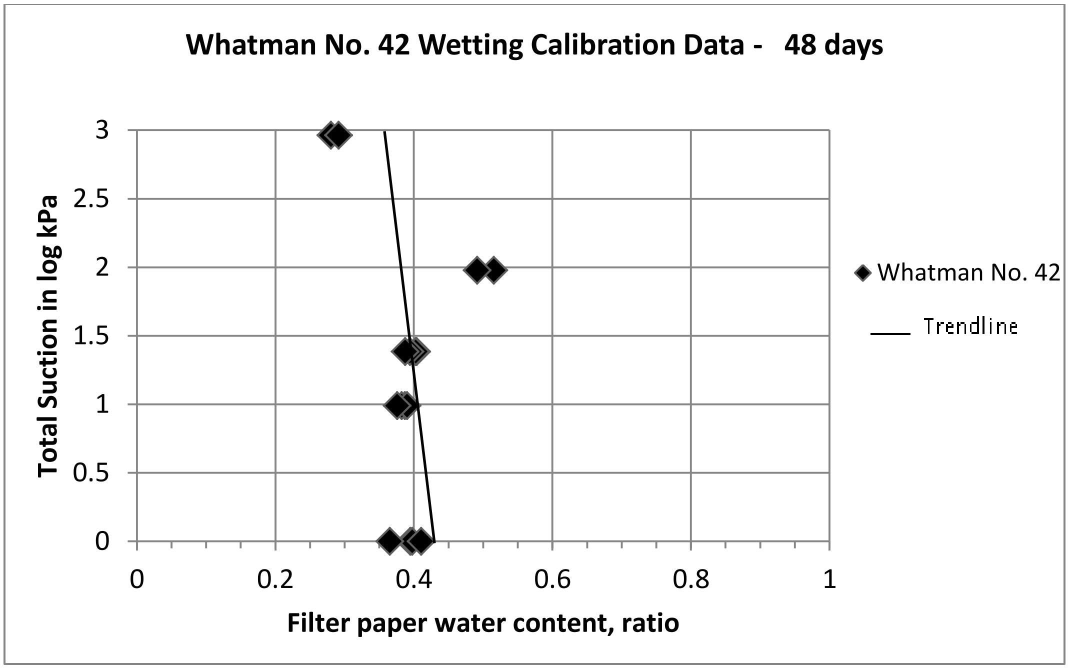


Figure 23: Wetting calibration data for Whatman No. 42 filter paper (Equilibration period – 48 days)

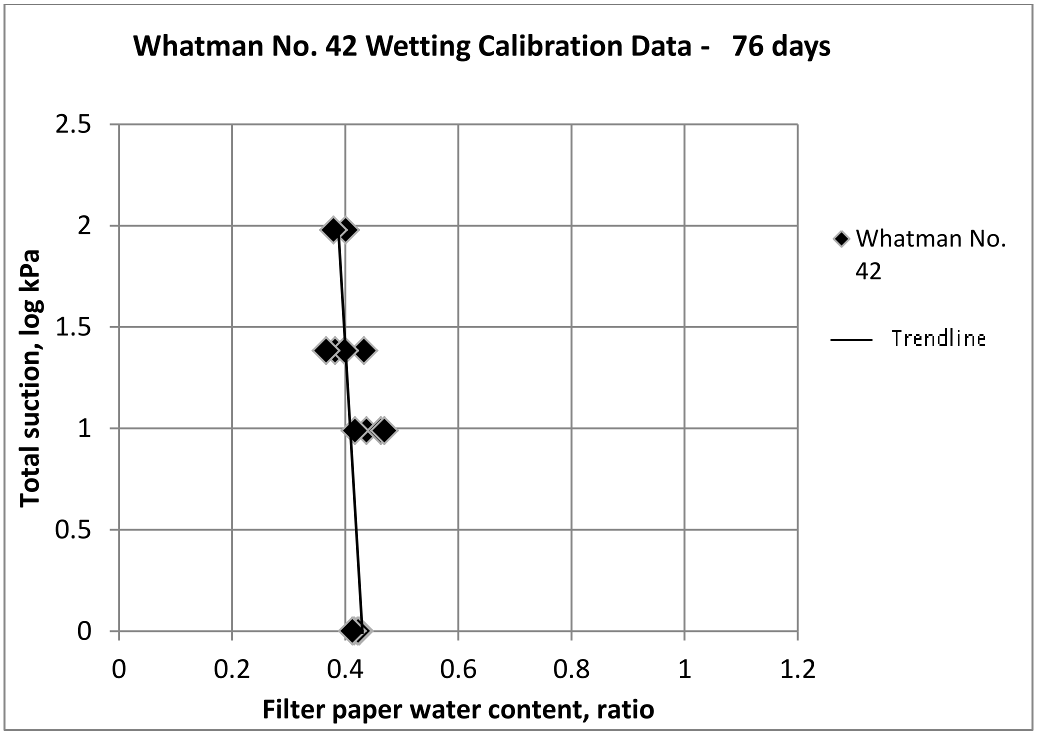


Figure 24: Wetting calibration data for Whatman No. 42 filter paper (Equilibration period – 76 days)

4.2.2 Schleicher and Schuell No. 589^2 Filter Papers

The water content versus total suction data for the wetting calibration data of Schleicher and Schuell No. 589^2 filter papers are shown in Figures 25, 26, 27, and 28 for equilibration periods of 31, 42, 48, and 76 days, respectively. The values shown are measured values (not average values).

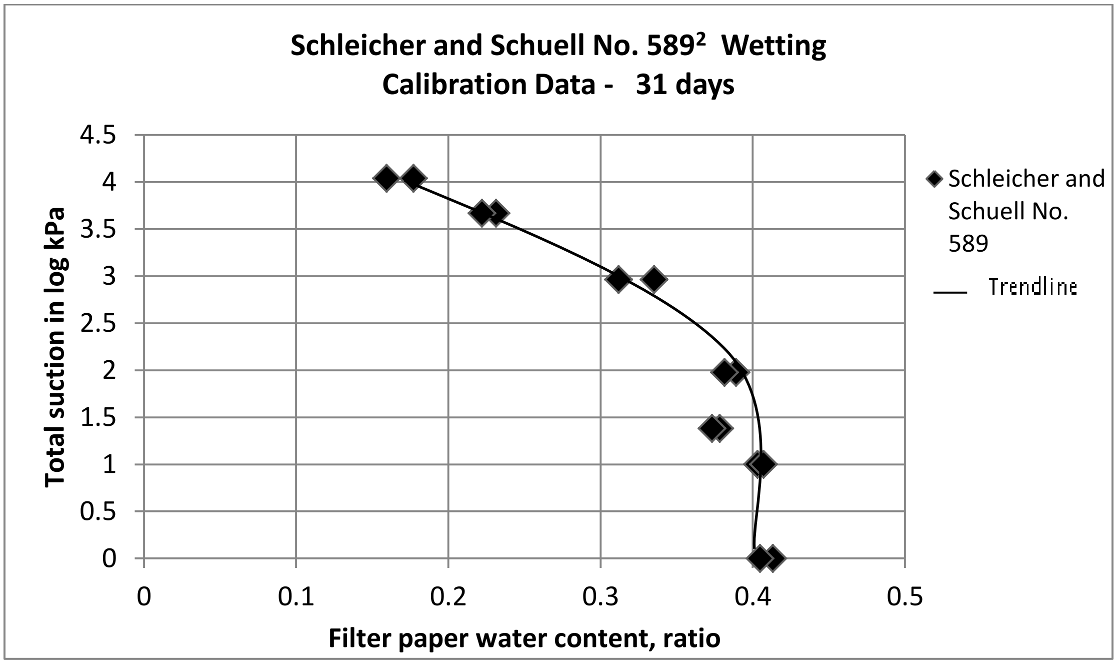


Figure 25: Wetting calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period – 31 days)

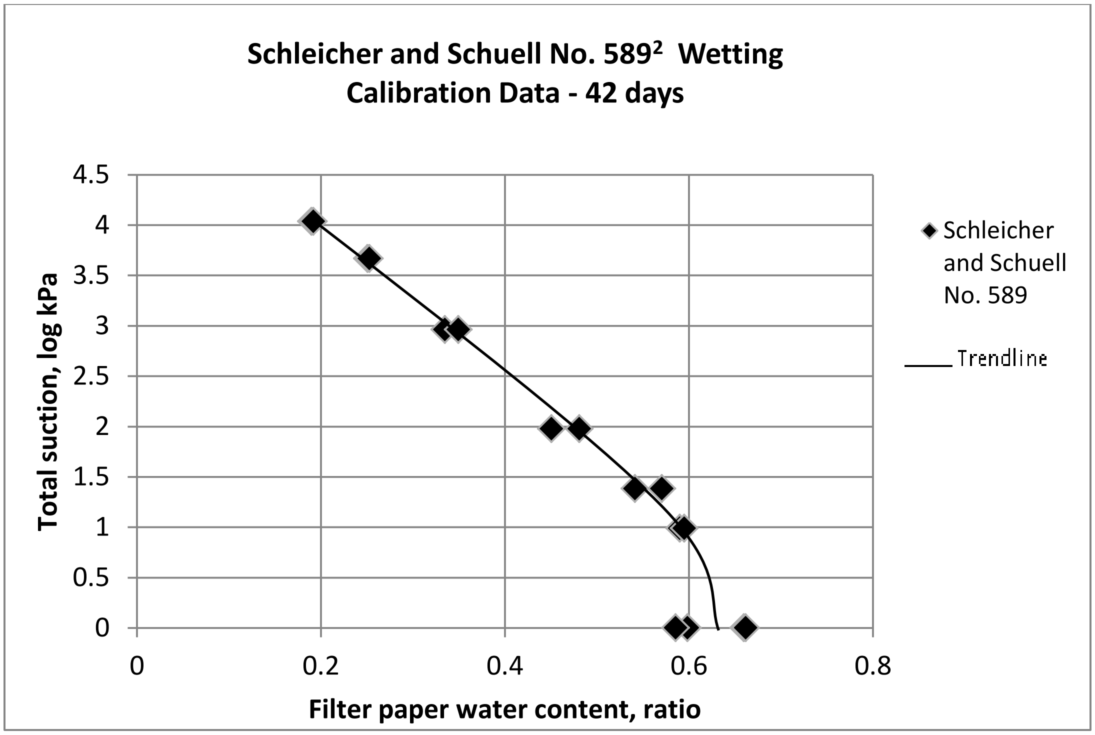


Figure 26: Wetting calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period – 42 days)

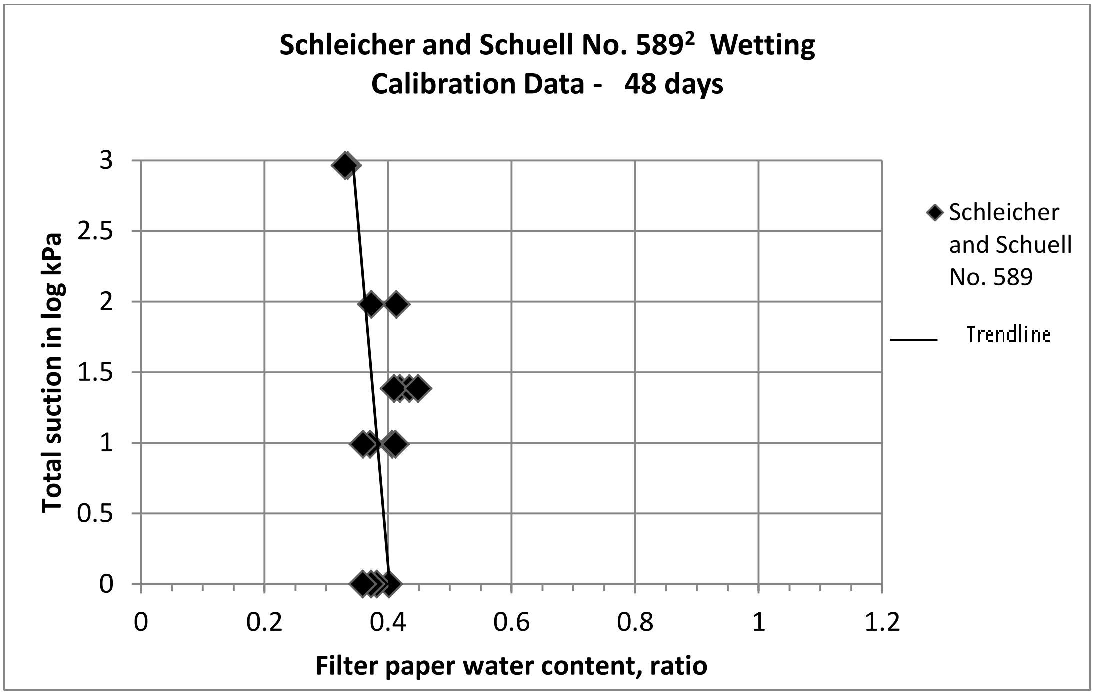


Figure 27: Wetting calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period – 48 days)

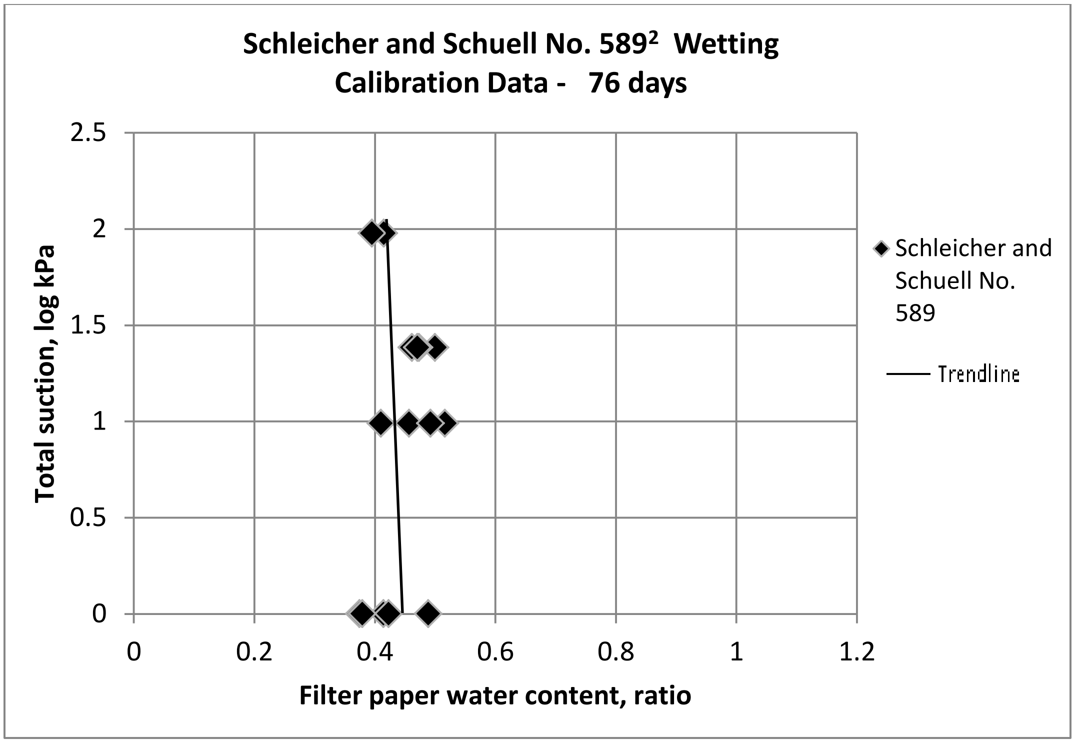


Figure 28: Wetting calibration data for Schleicher and Schuell No. 589^2 filter paper (Equilibration period – 76 days)

The wetting data show less scatter than the drying data as can be seen from Figures 21 to 24 and Figures 25 to 28 for Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers, respectively. However, more scatter between data values for a particular suction value can be seen in the low suctions region. This can clearly be seen in Figures 29 and 30 for Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers, respectively which show the calibration data for the respective filter papers from all the four sets of experiments conducted in this research. The data points in these figures represent the value of water content measured for individual filter papers from all four sets of experiments, i.e., the values shown are not average values of water contents of filter papers in the same jar.

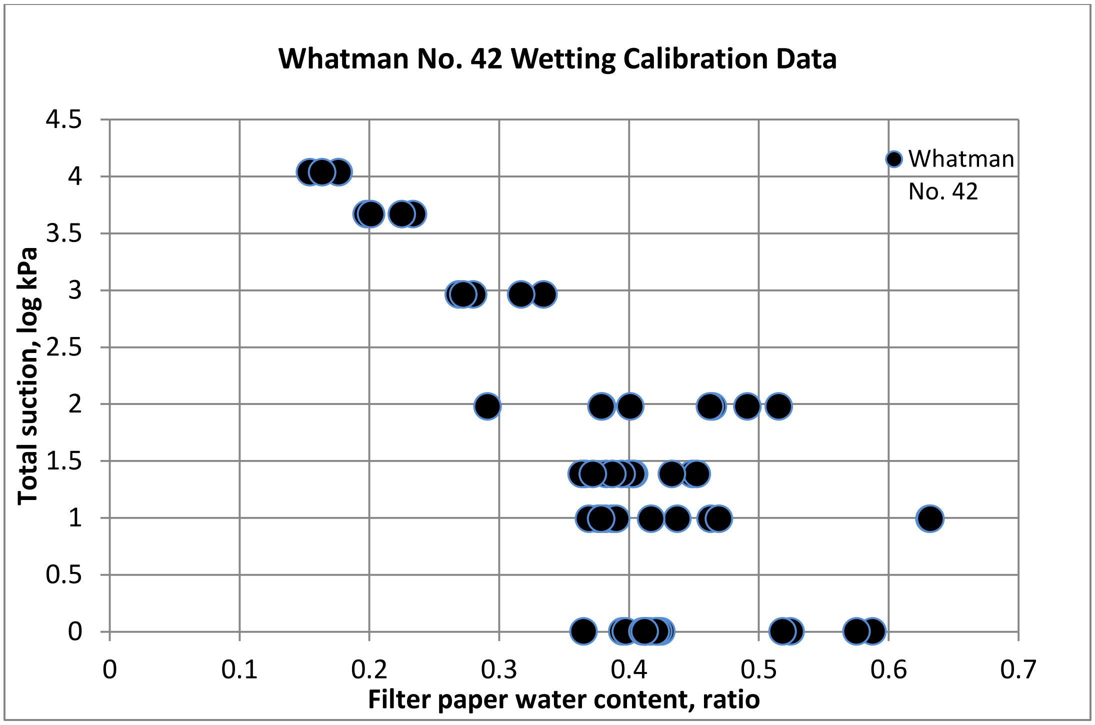


Figure 29: Wetting calibration data for Whatman No. 42 filter papers from all four sets of experiments

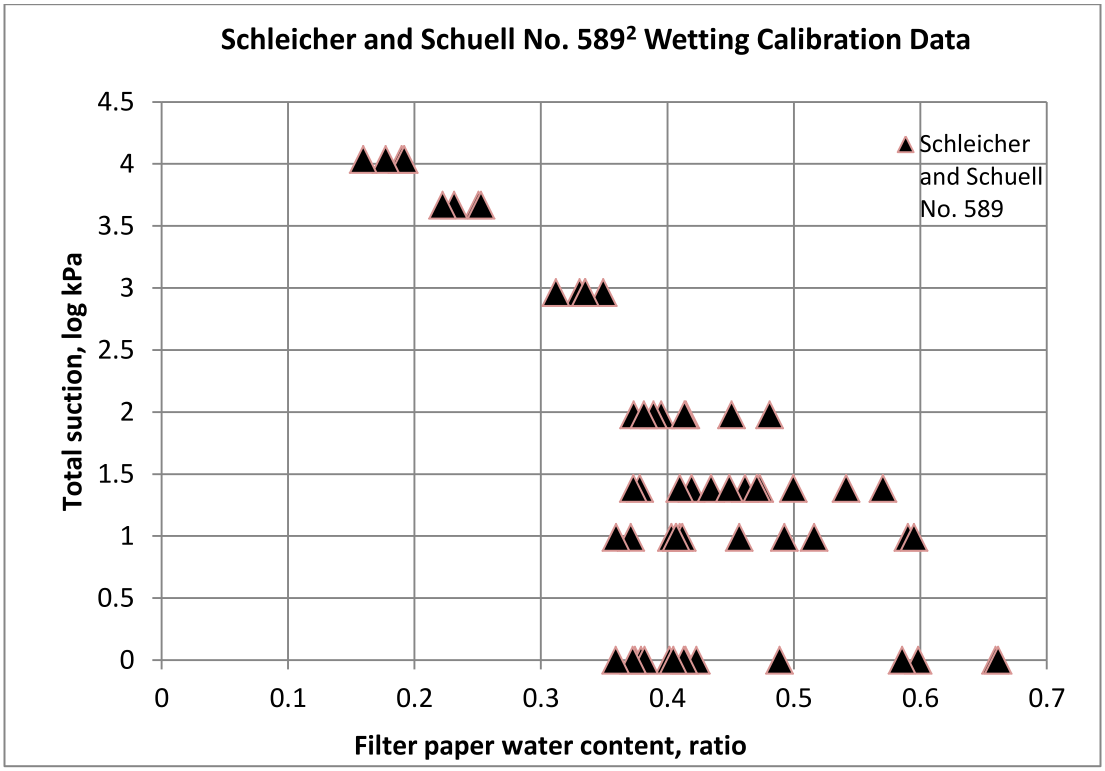


Figure 30: Wetting calibration data for Schleicher and Schuell No. 589^2 filter paper for all four sets of experiments

Figures 29 and 30 show that the wetting calibration data for both Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers show a smooth trend with increasing suction values.

4.3 Initial Water Content of Saturated/Soaked and Out-of-the Box Filter Papers

Table 17 in Appendix C presents the results of the saturated/soaked water content measurement while Table 18 in the same Appendix shows the out-of-the-box water content values for the two types of filter papers studied. It can be referred from Table 16 that the average water content values of the saturated Whatman papers are about 1.84 (184%), 1.92 (192%) and 1.95 (195%) for the papers soaked for 26 hours, 4 days and 18 days, respectively. The respective values for the Schleicher and Schuell No. 589^2 filter papers are 2.05 (205%), 2.25 (225%) and 2.15 (215%).

The values show that although there is a slight difference between the water content values soaked for different lengths of time, the difference is not significant. Therefore, it can be safely concluded that the longest (18-day) soaking period used did not have an impact on the values measured after equilibration. The maximum saturation water content calculated was 1.97 for the Whatman No. 42 papers soaked for 18 days while a maximum value of 2.31 was measured for the Schleicher and Schuell No. 589^2 filter paper after a 4-day soaking period. However, when measuring the water content values for the drying calibration data, a few higher water content values have been observed. These might be measurement errors as otherwise this would imply the soaked/saturated papers absorbed water from the solutions over which they were placed to come to equilibrium with which would contradict the laws of thermodynamics. These values were deemed outliers and were excluded from the calibration curve.

It should be noted that the water content values measured for the soaked filter papers (average of 1.9 and 2.15 for Whatman No. 42 and Schleicher and Scheull No. 589[2], respectively) are very high values. These values are not the “true” saturation values as the filter papers were soaked in the water and contained a significant amount of free water on them thus raising the water content values beyond the actual saturation values.

As can be seen in Table 18, the out-of-the box filter paper water content values were averaged to be 9.17% and 10.11%. The papers were stored in the lab which has a fairly constant relative humidity that ranges between 30-40%. All the papers were in their boxes and in the same lab environment. Although this might not have an impact on the calibration curves, these values are considered significant and any future use of the calibration curves should take into account this initial water content the filter papers have before they were set-up to equilibrate.

CHAPTER V

CALIBRATION CURVES

The wetting calibration data have been fitted with the general equation Fredlund and Xing (1994) proposed for soil-water characteristic curve. This equation was chosen because of the similarity observed between its curve and the trend of the wetting experimental data of this research. Fredlund and Xing's equation gives a relationship between volumetric water content and suction as shown in Equation (4).

$$\theta = \theta_s \left[\frac{1}{\ln\left[e + (\psi/a)^n\right]}\right]^m \tag{4}$$

where θ= volumetric water content of the soil at a given suction value ψ; θ_s= saturated volumetric water content of the soil; a, n, and m are three different soil parameters.

The water content values of the experimental data of this research are gravimetric. Therefore, the volumetric water contents in Equation (4) are changed into gravimetric values. The conversion between volumetric and gravimetric water content values is given by Equation (5).

$$\theta = w \, \frac{\rho_d}{\rho_w} \tag{5}$$

where w = gravimetric water content; ρ_d = dry density and ρ_w = density of water.

Fredlund and Xing's 3-parameter equation (Equation 4) for soil-water characteristic curve gives the value of θ in terms of ψ. This equation has been rewritten to give the value of ψ, total suction in terms of w, gravimetric water content to be used as a model for the calibration curves of the wetting calibration data of this research.

$$\theta = \theta_s \left[\frac{1}{\ln\left[e + (\psi/a)^n\right]}\right]^m \tag{4}$$

$$w = w_s \left[\frac{1}{\ln\left[e + (\psi/a)^n\right]}\right]^m \tag{4a}$$

$$(\frac{w}{w_s})^{\frac{1}{m}} = \frac{1}{\ln\left[e + (\psi/a)^n\right]} \tag{4b}$$

$$(\frac{w_s}{w})^{\frac{1}{m}} = \ln\left[e + (\psi/a)^n\right] \tag{4c}$$

$$e^{(\frac{w_s}{w})^{\frac{1}{m}}} = e + (\psi/a)^n \tag{4d}$$

$$(e^{(\frac{w_s}{w})^{\frac{1}{m}}} - e)^{\frac{1}{n}} = (\psi/a) \tag{4e}$$

$$\psi = a * [e^{(\frac{w_s}{w})^{\frac{1}{m}}} - e]^{\frac{1}{n}} \tag{5}$$

$$\psi = a * [e^{(\frac{w_s}{w})^{M}} - e]^{N} \tag{5a}$$

where M = 1/m and N = 1/n. Leong et al. (2002) also used Fredlund and Xing's (1994) soil-water characteristic curve equations to fit their total and matric suction calibration data.

Nonlinear regression using Statistical Product and Service Solutions (SPSS) was employed to fit Equation (5a) to the wetting calibration data of this research. Nonlinear regression is a method of finding a nonlinear model of the relationship between the dependent variable and a set of independent variables. This is accomplished using iterative estimation algorithm (SPSS Inc., 1989, 2010). To perform the nonlinear regression, SPSS requires initial values be entered for the parameters in the model expression. The parameter values of the total suction calibration equations proposed by Leong et al. (2002) based on Fredlund and Xing's soil-water characteristic curve have been adopted as initial values for the nonlinear regression to start its iteration with. The procedure followed to fit the data using SPSS is outlined in the Appendix.

The following equations have been fitted to the calibration data of the experimental results of this research (average water content values of all four sets of experiments have been used to fit the data):

Whatman No. 42 filter papers:

$$\psi = 23145[e^{\left(\frac{45}{w}\right)^{0.218}} - e]^{2.445} \tag{6}$$

Schleicher and Schuell No. 589^2:

$$\psi = 352596[e^{\left(\frac{47}{w}\right)^{0.077}} - e]^{2.270} \tag{7}$$

Where ψ = Total suction in kPa; and w = water content of the filter papers in %.

The plots of these equations are presented in Figures 31 and 32 along with the experimental data for Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers.

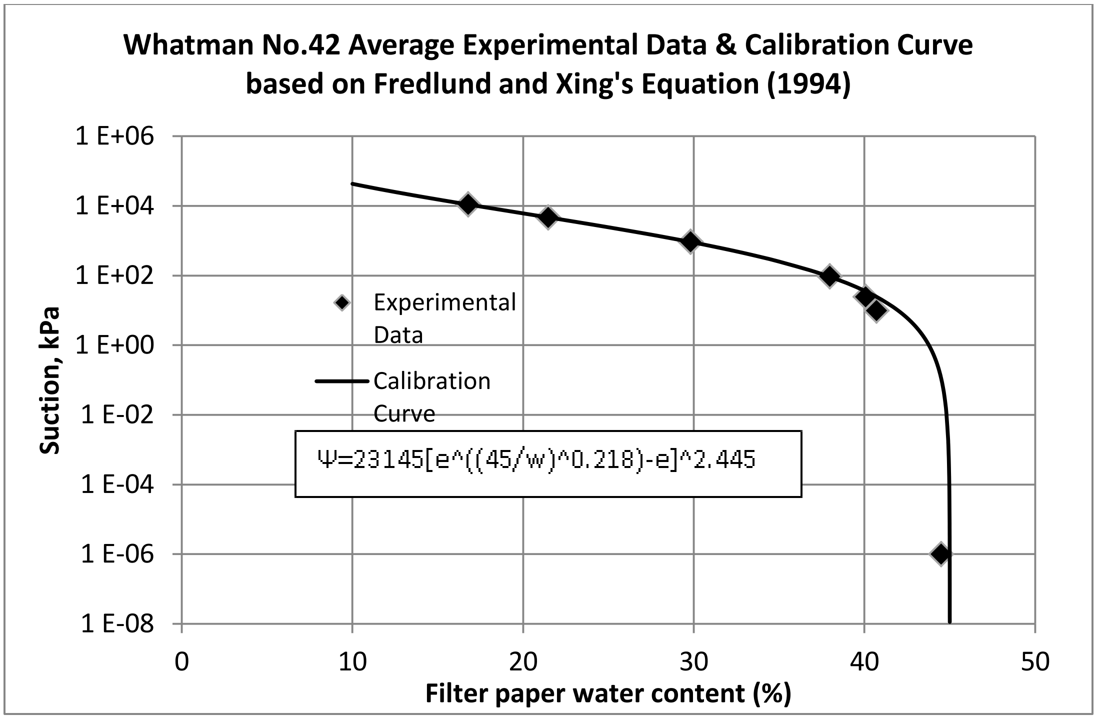


Figure 31: Wetting calibration data for Whatman No. 42 filter papers and calibration curve based on Fredlund and Xing's Equation (1994)

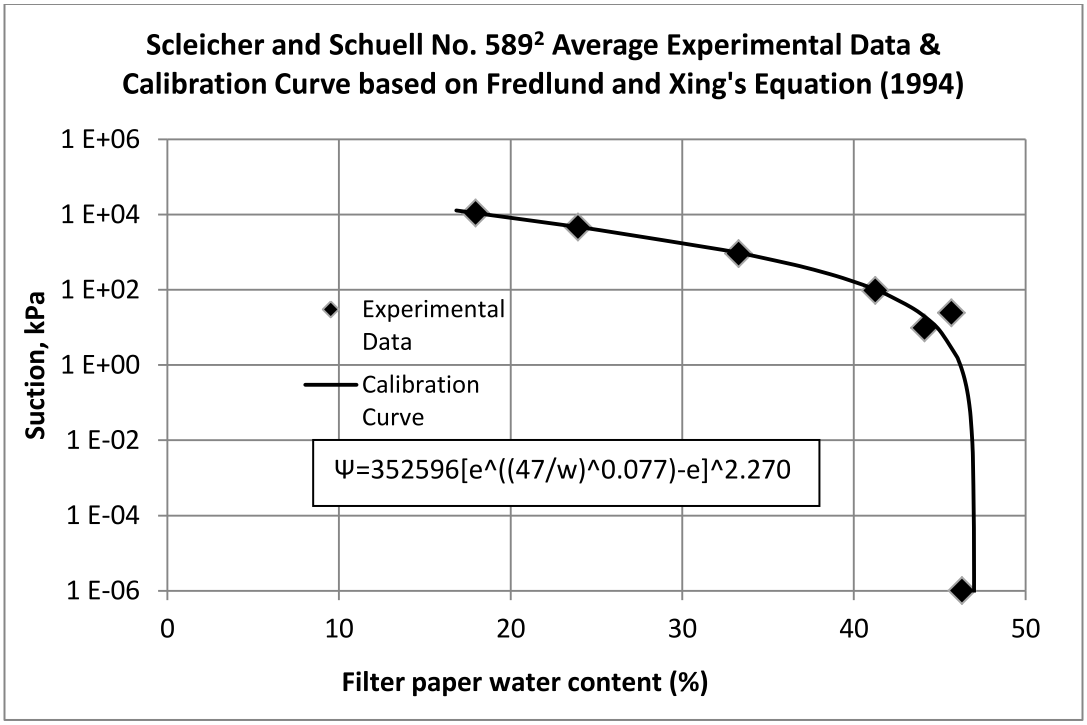


Figure 32: Wetting calibration data for Schleicher and Schuell No. 589^2 filter papers and calibration curve based on Fredlund and Xing's Equation (1994)

A comparison has been made of the calibration equations from this research, the calibration equations of Leong et al. (2002) and that of Bulut et al. (2001) for low and high suction values. Table 8 presents this comparison between different calibration equations.

Table 8 Comparison between Different Calibration Equations

Calibration Equations	Low Suction at w =35%		High Suction at w = 20%	
	kPa	log kPa	kPa	log kPa
This study				
Whatman No. 42	252.13	2.40	6107.43	3.79
Schleicher and Schuell No. 589^2	666.55	2.82	8254.02	3.92
ASTM D5298 – 10(2010)				
Whatman No. 42	398.57	2.60	5874.89	3.77
Schleicher and Schuell No. 589^2	995.41	3.00	7585.78	3.88
Leong et al. (2002)				
Whatman No. 42	11.22	1.05	3443.12	3.54
Schleicher and Schuell No. 589^2	288.97	2.46	7052.58	3.85
Bulut et al. (2001)				
Schleicher and Schuell No. 589^2	345.26	2.54	5959.37	3.78

Low suction comparison:

For both Whatman No. 42 and Schleicher and Schuell No. 5892 filter papers, the values from the calibration equations proposed in this study and those of ASTM D5298 – 10 (2010) are higher than those of the equations of Leong et al. (2002) which are on the lower side. The calibration equation of Bulut et al. (2001) gave an intermediate suction value. It can be seen from the values obtained from all the calibration equations that the suction values of Schleicher and Schuell No. 5892 are higher than those of Whatman No. 42 filter papers.

High suction comparison:

The values from all the calibration equations are fairly close to each other except for the lower suction value for Whatman No. 42 obtained using the calibration equations of Leong et al. (2002). The suction values obtained using the calibration equations from this study for both types of filter papers are higher than each of the values obtained using the other equations.

CHAPTER VI

DISCUSSIONS

The results presented and discussed in Chapter IV show that the drying and wetting calibration data are different. The data show that the water content values of the drying experiments are higher than those of the wetting experiments for the same suction values. The drying calibration experiments started with soaked filter papers having water content values as high as 1.9 (190%) and 2.15 (215%) for Whatman No. 42 and Schleicher and Schuell No. 589[2], respectively. Therefore, there was a tremendous amount of water in the papers that was to be lost and consequently, the filter papers had not most likely reached equilibrium during the two sets of tests with equilibration periods of 31 and 42 days. This led to higher water content values of the drying calibration data as compared to the wetting calibration data. In addition to the high initial water content values of the soaked filter papers, hysteresis is also a factor that contributed to the discrepancy between the wetting and drying calibration data as discussed in section 2.3.2.4.

It has been stated in Section 2.3.2.3 that Marinho (1994a) believed the distance between the filter paper and the suction source affects the equilibration time and that he recommended the distance be at most 1 cm. This gap has been about 2 cm in all the experiments conducted in this research. This is because the gap is too small to affect the equilibration period. Also, the gap does not affect the equilibration period since gravity is not a factor as the universal gas law is independent of gravity and consequently the total suction equation (Equation 1) given in terms of RH is independent of gravity.

The calibration curves given by ASTM D5298-10 (2010) have been presented in Figure 4 in section 2.4.4. The curves are captioned in the ASTM standard as “Calibration curves for wetting of filter paper”. However, none of the calibration curves or data from this study presented in Chapters III to V shows the trend shown by the ASTM curves. Also, the bi-linear curves of the ASTM are a combination of wetting and drying curves (McQueen and Miller, 1968). Researchers, nonetheless, have refuted this on account of hysteresis– the drying and wetting curves cannot be combined to give a single calibration curve that can be used for suction measurement using filter paper method through a wetting or drying experiment (Bulut et al., 2001; Leong et al., 2002). The results of this research have also confirmed that drying and wetting calibration curves are different.

6.1 Drying Calibration Data

The drying calibration data have not been fitted with a single calibration curve as the data from the different equilibration periods showed considerable scatter. As explained in the first paragraph of this chapter, the drying calibration data obtained in the two sets of lower equilibration period experiments did not represent equilibrium water content values as the filter papers would still lose water as observed from the larger equilibration period experiments. Therefore, a single calibration curve could not be fitted for the drying data.

Figure 15 and Figure 20 show the drying calibration data for each of the four sets of experiments for Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers, respectively. A shift to the left (inward) is observed in both types of filter papers. This shifting inward of the drying calibration data with increasing equilibration period reaffirms that hysteresis does decrease with increase in the equilibration time allowed, as also shown by Leong et al. (2002).

The difference between the calibration data trends of the four sets of experiments as shown in Figure 15 and Figure 20 indicates that equilibration period is a critical factor in the calibration process. These figures connote that equilibrium was not actually reached at equilibrium periods of 31 and 42 days. It can be seen, however, that the calibration data for the second and third sets are very close to each other. It has been discussed in Chapter 2 that different calibration periods have been used and recommended by researchers. The minimum period is minutes recommended by Hamblin (1981) and the maximum is 25 – 30 days recommended by Harrison and Blight (1998) for drying calibration curve using the contact method. The results obtained in this research, however, imply otherwise for drying non-contact vapor transfer method. However, it should be noted that the drying experiments were conducted with soaked filter papers with high water content values which contributed to the longer equilibration period.

6.2 Wetting Calibration Data

The wetting calibration data show a smooth trend for both Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers. A scatter, however, is observed for lower suction values between data points of the same suction value.

Not much shifting of the calibration data trend has been observed with increase in equilibration period for both Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers with the exception of the calibration data from the fourth set of experiments with equilibration period of 42 days which showed a slight shift to the right compared to the others. This indicates that for the wetting calibration data, the first equilibration period allowed for equilibrium (31 days) is probably sufficient for the non-contact method. The equilibration period could also be less, which this study has not investigated as the minimum equilibration period used in this research was 31 days.

The average water content value at a suction value of 95 kPa (equivalent to log kPa value of 1.98), is about 38% for Whatman No. 42 filter papers. The average water content value at 95 kPa for Schleicher and Schuell No. 589^2 filter papers is about 41%. This can be seen in Figures 28 and 29 in Chapter V. It can be seen from these figures that the curve shows a very abrupt change at this break point. The range of bound water values determined by Nelson (1977) for different paper samples is 0.21 to 0.27g/g (or 21 to 27%), whereas it is 0.265 for Whatman No. 1 filter paper. The water content values of both the Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers at the break points are higher than the bound water ranges for paper samples given by Nelson (1977). They are also higher than the roughly determined bound water value of 0.2g/g for cotton linters by Froix and Nelson (1975). The fiber saturation point (FSP) has been determined to be 50% using DSC by Nelson (1975). Felby et al. (2008) indicated FSP values between 33 – 43%. The water content values 38% and 41% for the Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers, respectively, at the break point are closer to the FSP values given above. At the break point, all the bound water the filter papers could take up has been sorbed but also there is free water in the filter papers. It has been mentioned in Chapter II that free water exists in a water-cellulose system at very low water content values. The free water incorporation point has been determined to be 5% using DSC for cotton linters which agrees well with results obtained by NMR measurements (Nelson, 1977). The lower portion of the calibration curve beyond the break point may be explained by the filter papers approaching “true” saturation.

ASTM D5298-10 (2010) attributes the two-segmented nature of typical calibration curves to the change in sorption mechanism, i.e., the upper part of the curve represents moisture retained as water films adsorbed to particle surfaces, while the lower segment represents moisture retained by capillary or surface tension forces between particles. The ASTM standard states that the break points are 45.3% and 54% for Whatman No. 42 and Schleicher and Schuell 5892, respectively (McQueen and Miller, 1968; Fawcett and Collis-George, 1967; Greacen et al., 1987). Although the change in sorption mechanism is a plausible explanation, attributing water content values as high as 45% and 54% to only moisture retained as water films adsorbed to particle surfaces contradicts with the definition of the fiber saturation point (FSP) - which has been determined to be between 33-50% (Nelson, 1977; Felby et al., 2008). Christensen and Giertz (1965) defined FSP as the amount of water required to completely fill the cell wall without filling the lumen. It has been discussed in Section 2.4.3 that at the cell wall level bonding occurs either by capillary forces, by hydrogen bonds to hydroxyl groups on hemicelluloses and lignin or by hydrogen bonds to hydration layers already adsorbed (Felby et al., 2008).

The lower part of the calibration curve can be explained by free water in the lumens and water layers adsorbed in layers to the microfibril surfaces but sufficiently far from the cellulose being shielded from its effect by the inner most hydration layers.

There is a resemblance between the calibration curves presented in Chapter V and the typical sorption isotherm for cellulose fibers presented in Figure 4. However, the water content values in Figure 4 are significantly lower than the experimental data of this research. This may in part be explained by a difference in the crystallinity of the materials considered as the maximum water uptake of cellulose is determined by its degree of crystallinity (Olsson and Salmén, 2004).

CHAPTER VII

CONCLUSIONS AND RECOMMENDATIONS

7.1 Conclusions

The experiments of this research showed that the equilibration period to be allowed for the filter paper and salt solution to come to equilibrium depends on whether the filter papers are to equilibrate from the dry (wetting process) or the wet (drying process) initial state. Also, for the drying calibration experiments, the equilibration period depends on the initial water content of the filter paper.

It has been reaffirmed that equilibration time does affect how much hysteresis is exhibited. Hysteresis was less with increased equilibration periods.

This study could not address the issue of filter paper quality/ consistency among different batches as filter papers from the same batch have been used for all sets of experiments.

Wetting calibration curves were developed for the Whatman No. 42 and Scleicher and Schuell No. 589^2 filter papers based on Fredlund and Xing's equation (1994). Break points were observed in the wetting calibration curves of both the Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers. The water content values at these break points were associated with the type of water in the filter papers at these points. Specifically, the break points 38% and 41% for the Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers were associated with the fiber saturation point of cotton linters which has been estimated to be between 33-50%. Accordingly, at the break points, all the bound water the filter papers could take up has most likely been sorbed and some amount of free water has also been taken up. The nature of the curve beyond this break point is associated with the filter papers approaching “true” saturation.

The drying filter papers had higher values of water content than the wetting filter papers for the same suction. This is in part attributable to the initial high water content of the soaked/saturated filter papers which required longer periods of equilibration than the wetting filter papers. The difference in water content values between the wetting and drying filter papers is also attributable to hysteresis.

No calibration curves were fitted to the drying experimental data. However, observation of the trend of the drying calibration data for the different sets of experiments showed that the curves shifted inward (showed a decrease in water content values) with longer equilibration periods.

A comparison has been made between the calibration equations from this study and calibration equations of ASTM D5298 – 10 (2010), Leong et al. (2002) and Bulut et al. (2001). Accordingly, for the low suction water content value, the equations from this study gave higher suction values than those of Leong et al. (2002) and Bulut et al. (2001) but lower values than those of the ASTM D5298 – 10 (2010) for both Whatman No. 42 and Schleicher and Schuell No. 589^2 filter papers. For the high suction water content value, the values obtained using the calibration equations from this study for both types of filter papers are higher than each of the values obtained using the other equations.

7.2 Recommendations

Review of the literature on the interaction between cellulose fibers and water and the experimental results obtained in this research showed that the filter paper method should also be looked into from the perspective of the molecular interaction between the filter paper and the water in addition to the thermodynamics that determine the equilibrium conditions. This might require observing the water uptake by and loss from the filter papers as it is taking place during the equilibration period. This could be achieved by devising a way to interface the temperature-controlled chamber wherein the filter papers are sealed to equilibrate with an instrument such as an NMR apparatus.

Filter papers for drying calibration experiments took longer time to reach equilibrium as the experiments were conducted on soaked filter papers with high water content values with a significant amount of free water in them and giving artificially high saturated water content values. Based on experience gained from this research, it is recommended that future drying calibration data be obtained by conditioning the soaked filter papers using apparatus such as desiccators to lower the water content values to the “true” saturated values.

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APPENDICES

APPENDIX A – LABORATORY EXPERIMENTS

Table 9 Filter Paper Water Content Measurement Worksheet for 1st Set of Experiments (Equilibration Period = 31 days)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 10/15/12
Equilibration Period: 31 days

Test Date:
11/15/2012

Moisture tin No.:		A1	A2	A3	A4	A5	A6	A7	A8	A9	B1
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.94134	36.86496	36.72025	36.98631	36.77998	36.69133	36.78415	36.78557	37.04523	36.75005
Mass of wet filter paper + cold tare mass, g	M_1	37.15425	37.07644	36.98682	37.25316	37.06908	36.96214	37.00232		37.60842	37.43256
Mass of dry filter paper + hot tare mass, g	M_2	37.1064	37.03097	36.92934	37.19663	36.99223	36.89126	36.9449		37.20868	36.9239
Hot tare mass, g	T_h	36.93014	36.85265	36.70656	36.97461	36.76584	36.67859	36.7745		37.02917	36.7369
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.17626	0.17832	0.22278	0.22202	0.22639	0.21267	0.1704		0.17951	0.187
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.03665	0.03316	0.04379	0.04483	0.06271	0.05814	0.04777		0.38368	0.49551
Water content of filter paper, g (M_w/M_f)	W_f	0.207931	0.185958	0.196562	0.201919	0.277	0.273381	0.28034		2.137374	2.6497861
Suction, log Kpa	h_1	4.04	4.04	4.04	4.04	3.67	3.67	3.67		2.96	2.96
		Drying		Drying		Drying		Drying		Drying	
		2.2M, Schleicher & Scheull No. 589[2]		2.2M, Whatman No. 42		1.0M, Whatman No. 42		1.0M, Schleicher & Scheull No. 589[2]		0.2M, Schleicher & Scheull No. 589[2]	

A8 - Filter paper slipped into the solution and so not used.

B1- Filter paper showed some discoloration - it looked like it was burnt slightly at a few spots.

Table 9 Filter Paper Water Content Measurement Worksheet for 1st Set of Experiments (Equilibration Period = 31 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET										
Date experiment set up: 10/15/12 Equilibration Period: 31 days										Test Date: 11/15/2012
Moisture tin No.:	B2	B3	B4	B5	B6	B7	B8	B9	C1	C2
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.93152	36.9066	36.97579	36.7775	36.8293	36.85696	36.92423	36.71331	36.93604	36.85022
Mass of wet filter paper + cold tare mass, g M_1	37.2397	37.21255	37.17884	36.98392	37.0801	37.1114	37.14183	36.93075	37.2035	37.11734
Mass of dry filter paper + hot tare mass, g M_2	37.13914	36.9165	37.13543	36.94961	37.0312	37.05999	37.08361	36.87742	37.14255	37.05592
Hot tare mass, g T_h	37.11313	36.89231	36.96295	36.77158	36.81396	36.84139	36.9069	36.69952	36.91932	36.83363
Mass of dry filter paper, g (M_2 -T_h) M_f	0.02601	0.02419	0.17248	0.17803	0.21724	0.2186	0.17671	0.1779	0.22323	0.22229
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.28217	0.28176	0.03057	0.02839	0.03356	0.03584	0.04089	0.03954	0.04423	0.04483
Water content of filter paper, g (M_w/M_f) W_f	10.84852	11.64779	0.177238	0.159468	0.154484	0.163952	0.231396	0.22226	0.198136	0.20167349
Suction, log Kpa h_1	2.96	2.96	4.04	4.04	4.04	4.04	3.67	3.67	3.67	3.67
	Drying		Wetting		Wetting		Wetting		Wetting	
	0.2 M, Whatman No. 42		2.2 M, Schleicher & Scheull No. 589^2		2.2 M, Whatman No. 42		1.0 M, Schleicher & Scheull No. 589^2		1.0 M, Whatman No. 42	

B8 - Filter paper touched the lid of the moisture tin.

Table 9 Filter Paper Water Content Measurement Worksheet for 1st Set of Experiments (Equilibration Period = 31 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET									
Date experiment set up: 10/15/12 Equilibration Period: 31 days									Test Date: 11/15/2012
Moisture tin No.:		C3	C4	C5	C6	C7	C8	C9	D1
Top or bottom filter paper		T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.89294	36.84131	36.74358	36.61643	36.89201	36.94423	36.80167	36.87721
Mass of wet filter paper + cold tare mass, g	M_1	37.12611	37.06753	37.0308	36.89811	37.29539	37.35814	37.14653	37.22508
Mass of dry filter paper + hot tare mass, g	M_2	37.05255	36.99826	36.95659	36.8204	37.09797	37.15281	36.96612	37.03718
Hot tare mass, g	T_h	36.87792	36.82582	36.73029	36.59903	36.87757	36.92718	36.78668	36.86217
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.17463	0.17244	0.2263	0.22137	0.2204	0.22563	0.17944	0.17501
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.05854	0.05378	0.06092	0.06031	0.18298	0.18828	0.16542	0.17286
Water content of filter paper, g (M_w/M_f)	W_f	0.335223	0.311877	0.2692	0.27244	0.830218	0.834464	0.921868	0.98771499
Suction, log Kpa	h_1	2.96	2.96	2.96	2.96	1.98	1.98	1.38	1.38
		Wetting		Wetting		Drying		Drying	
		0.2 M, Schleicher & Scheull No. 589[2]		0.2 M, Whatman No. 42		0.02 M, Whatman No. 42		0.005 M, Schleicher & Scheull No. 589[2]	

Table 9 Filter Paper Water Content Measurement Worksheet for 1st Set of Experiments (Equilibration Period = 31 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET											
Date experiment set up: 10/15/12 Equilibration Period: 31 days											Test Date: 11/15/2012
Moisture tin No.:		D2	D3	D4	D5	D6	D7	D8	D9	E1	E2
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.89852	36.94559	36.35937	36.38757	36.47036	36.64587	36.41828	36.41816	36.72346	36.92522
Mass of wet filter paper + cold tare mass, g	M_1	37.17922	37.23007	36.95932	37.06874	36.70966	36.89223	36.72249	36.72692	36.96624	37.16842
Mass of dry filter paper + hot tare mass, g	M_2	37.05753	37.10545	36.56559	36.59318	36.62669	36.80719	36.69179	36.62571	36.88547	37.08648
Hot tare mass, g	T_h	36.88274	36.92886	36.3429	36.37104	36.45442	36.62887	36.40036	36.40182	36.70933	36.90938
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.17479	0.17659	0.22269	0.22214	0.17227	0.17832	0.29143	0.22389	0.17614	0.1771
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.10591	0.10789	0.37726	0.45903	0.06703	0.06804	0.01278	0.08487	0.06664	0.0661
Water content of filter paper, g (M_w/M_f)	W_f	0.605927	0.610963	1.694104	2.0664	0.389099	0.381561	0.043853	0.37907	0.378335	0.37323546
Suction, log Kpa	h_1	1.98	1.98	1.38	1.38	1.98	1.98	1.98	1.98	1.38	1.38
		Drying		Drying		Wetting		Wetting		Wetting	
		0.02 M, Schleicher & Scheull No. 589^2		0.005 M, Whatman No. 42		0.02 M, Schleicher & Scheull No. 589^2		0.02 M, Whatman No. 42		0.005 M, Schleicher & Scheull No. 589^2	

Table 9 Filter Paper Water Content Measurement Worksheet for 1st Set of Experiments (Equilibration Period = 31 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET										
Date experiment set up: 10/15/12 Equilibration Period: 31 days										Test Date: 11/15/2012
Moisture tin No.:	E3	E4	E5	E6	E7	E8	E9	F1	F2	F3
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.87326	36.81623	37.05722	36.81874	36.62756	36.73738	36.97959	37.01288	36.67545	36.90356
Mass of wet filter paper + cold tare mass, g M_1	37.17622	37.11255	37.4179	37.18314	37.18388	37.30098	37.22275	37.26731	36.97982	37.20346
Mass of dry filter paper + hot tare mass, g M_2	37.07896	37.0149	37.21588	36.97887	36.83068	36.94471	37.13576	37.1752	36.88564	37.10175
Hot tare mass, g T_h	36.85681	36.799	37.03797	36.80123	36.61063	36.72258	36.96247	36.99439	36.66336	36.88424
Mass of dry filter paper, g (M_2 -T_h) M_f	0.22215	0.2159	0.17791	0.17764	0.22005	0.22213	0.17329	0.18081	0.22228	0.21751
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.08081	0.08042	0.18277	0.18676	0.33627	0.34147	0.06987	0.07362	0.08209	0.08239
Water content of filter paper, g (M_w/M_f) W_f	0.363763	0.372487	1.027317	1.05134	1.528153	1.537253	0.403197	0.407168	0.369309	0.37878718
Suction, log Kpa h_1	1.38	1.38	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99
	Wetting		Drying		Drying		Wetting		Wetting	
	0.005 M, Whatman No. 42		0.002 M, Schleicher & Scheull No. 589^2		0.002 M, Whatman No. 42		0.002 M, Schleicher & Scheull No. 589^2		0.002 M, Whatman No. 42	

Table 9 Filter Paper Water Content Measurement Worksheet for 1^{st} Set of Experiments (Equilibration Period = 31 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET								
Date experiment set up: 10/15/12 Equilibration Period: 31 days								Test Date: 11/15/2012
Moisture tin No.:	F4	F5	F6	F7	F8	F9	G1	G2
Top or bottom filter paper	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.79211	36.94934	36.64828	36.77392	36.81877	36.71879	36.91577	35.98127
Mass of wet filter paper + cold tare mass, g M_1	37.13278	37.30117	37.0408	37.19116	37.13173	37.03262	37.15983	36.22734
Mass of dry filter paper + hot tare mass, g M_2	36.99266	37.15994	36.8082	36.94268	37.022	36.92678	37.07248	36.13903
Hot tare mass, g T_h	36.77402	36.93525	36.63339	36.76044	36.80174	36.70457	36.89978	35.96387
Mass of dry filter paper, g (M_2 -T_h) M_f	0.21864	0.22469	0.17481	0.18224	0.22026	0.22221	0.1727	0.17516
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.12203	0.12714	0.21771	0.235	0.0927	0.09162	0.07136	0.07091
Water content of filter paper, g (M_w/M_f) W_f	0.558132	0.565846	1.245409	1.289508	0.420866	0.412313	0.413202	0.40482987
Suction, log Kpa h_1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Drying		Drying		Wetting		Wetting	
	De-ionized water, Whatman No. 42		De-ionized water, Schleicher and Schuell No. 589^2		De-ionized water, Whatman No. 42		De-ionized water, Schleicher and Schuell No. 589^2	

Table 10 Filter Paper Water Content Measurement Worksheet for 2nd Set of Experiments (Equilibration Period = 48 days)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 12/06/12

Equilibration Period: 48 days Test Date: 01/23/13-01/24/13

Moisture tin No.:	A1	A2	A3	A4	A5	A6	A7	A8	A9	B1
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.93843	36.86356	36.71969	36.98534	36.77919	36.69029	36.78315	36.78488	37.04464	36.74949
Mass of wet filter paper + cold tare mass, g M_1	37.22969	37.15993	37.0244	37.28551	37.08133	36.99608	37.08696	37.08621	37.3407	37.05029
Mass of dry filter paper + hot tare mass, g M_2	37.12925	37.06143	36.91873	37.17915	36.97806	36.89296	36.98348	36.98517	37.23769	36.94875
Hot tare mass, g T_h	36.92046	36.84436	36.70074	36.96634	36.76026	36.67303	36.76713	36.76911	37.02663	36.73194
Mass of dry filter paper, g (M_2 -T_h) M_f	0.20879	0.21707	0.21799	0.21281	0.2178	0.21993	0.21635	0.21606	0.21106	0.21681
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.08247	0.0793	0.08672	0.08736	0.08434	0.08586	0.08746	0.08527	0.085	0.08399
Water content of filter paper, g (M_w/M_f) W_f	0.39499	0.36532	0.397816	0.410507	0.387236	0.390397	0.404252	0.394659	0.402729	0.38738988
Suction, log Kpa h_1	0.00	0.00	0.00	0.00	0.99	0.99	1.38	1.38	1.38	1.38
	Wetting		Wetting		Wetting		Wetting		Wetting	
	De-ionized water		De-ionized water		0.002M		0.005M		0.005M	
	Whatman No. 42		Whatman No. 42		Whatman No. 42		Whatman No. 42		Whatman No. 42	

Table 10 Filter Paper Water Content Measurement Worksheet for 2nd Set of Experiments (Equilibration Period = 48 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 12/06/12

Equilibration Period: 48 days

Test Date: 01/23/13-01/24/13

Moisture tin No.:	B2*	B3*	B4	B5	B6	B7	B8	B9	C1	C2
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.93089	36.90606	36.97495	36.77699	36.82868	36.85633	36.92398	36.71282	36.9352	36.84985
Mass of wet filter paper + cold tare mass, g M_1	37.18502	37.14686	37.22291	37.02204	37.13174	37.15717	37.17591	36.95856	37.17677	37.09047
Mass of dry filter paper + hot tare mass, g M_2	37.09192	37.05858	37.12957	36.92599	37.03031	37.05724	37.08243	36.87066	37.08918	37.00849
Hot tare mass, g T_h	36.91286	36.88778	36.95674	36.75688	36.81107	36.83864	36.90336	36.69662	36.91686	36.8332
Mass of dry filter paper, g $(M_2 - T_h)$ M_f	0.17906	0.1708	0.17283	0.16911	0.21924	0.2186	0.17907	0.17404	0.17232	0.17529
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.07507	0.07	0.07513	0.07594	0.08382	0.08224	0.07286	0.0717	0.06925	0.06533
Water content of filter paper, g (M_w/M_f) W_f	0.419245	0.409836	0.434705	0.449057	0.382321	0.376212	0.40688	0.411974	0.401869	0.37269667
Suction, log Kpa h_1	1.38	1.38	1.38	1.38	0.99	0.99	0.99	0.99	0.00	0.00
	Wetting		Wetting		Wetting		Wetting		Wetting	
	0.005 M		0.005 M		0.002 M		0.002 M		De-ionized water	
	Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2		Whatman No. 42		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2	

* B2 & B3 - Wet weight measurement took a little more time than usual.

Table 10 Filter Paper Water Content Measurement Worksheet for 2nd Set of Experiments (Equilibration Period = 48 days) (Cont’d)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 12/06/12

Equilibration Period: 48 days

Test Date: 01/23/13-01/24/13

Moisture tin No.:		C3*	C4*	C5	C6	C7	C8	C9	D1	D2	D3
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.89294	36.84107	36.74339	36.61627	36.89148	36.94399	36.80143	36.87694	36.89824	36.9452
Mass of wet filter paper + cold tare mass, g	M_1	37.1983	37.22262	36.98875	36.84992	37.12161	37.17529	37.08517	37.16137	37.13817	37.18118
Mass of dry filter paper + hot tare mass, g	M_2	37.09337	37.03848	36.90061	36.77055	37.04479	37.0957	37.00776	37.08068	37.05667	37.10092
Hot tare mass, g	T_h	36.87523	36.82086	36.72704	36.60042	36.87239	36.92186	36.78611	36.8604	36.88167	36.92735
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.21814	0.21762	0.17357	0.17013	0.1724	0.17384	0.22165	0.22028	0.175	0.17357
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.08722	0.16393	0.07179	0.06352	0.05773	0.05746	0.06209	0.06415	0.06493	0.06241
Water content of filter paper, g (M_w/M_f)	W_f	0.399835	0.753286	0.413608	0.373362	0.334861	0.330534	0.280126	0.29122	0.371029	0.35956675
Suction, log Kpa	h_1	1.98	1.98	1.98	1.98	2.96	2.96	2.96	2.96	0.99	0.99
		Drying		Wetting		Wetting		Wetting		Wetting	
		0.02M		0.02M		0.2M		0.2M		0.002M	
		Whatman No. 42		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2		Whatman No. 42		Schleicher and Schuell No. 589^2	

* C3 & C4 - Wet weight measurement took a little more time than usual.

Table 10 Filter Paper Water Content Measurement Worksheet for 2nd Set of Experiments (Equilibration Period = 48 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 12/06/12

Equilibration Period: 48 days

Test Date: 01/23/13-01/24/13

Moisture tin No.:		D4*	D5	D6	D7	D8	D9	E1	E2	E3	E4
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.35914	36.38731	36.46986	36.64557	36.41798	36.41812	36.72333	36.92505	36.8729	36.8156
Mass of wet filter paper + cold tare mass, g	M_1	36.60364	36.62598	36.7295	36.90441	36.77026	36.77453	37.08793	37.29843	37.14505	37.0761
Mass of dry filter paper + hot tare mass, g	M_2	36.51935	36.54464	36.62851	36.80404	36.61267	36.6246	36.92544	37.12938	37.03036	36.9684
Hot tare mass, g	T_h	36.34243	36.36905	36.45234	36.62729	36.39664	36.40354	36.70726	36.90618	36.85399	36.7978
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.17692	0.17559	0.17617	0.17675	0.21603	0.22106	0.21818	0.2232	0.17637	0.17059
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.06758	0.06308	0.08347	0.08209	0.13625	0.13535	0.14642	0.15018	0.09578	0.08984
Water content of filter paper, g (M_w/M_f)	W_f	0.381981	0.359246	0.473804	0.464441	0.630699	0.612277	0.671097	0.672849	0.543063	0.52664
Suction, log Kpa	h_1	0.00	0.00	1.98	1.98	0.00	0.00	0.00	0.00	1.38	1.38
		Wetting		Drying		Drying		Drying		Drying	
		De-ionized water		0.02M		De-ionized water		De-ionized water		0.005M	
		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42		Whatman No. 42		Schleicher and Schuell No. 589[2]	

*D4 - Lid fell down before the mass of wet filter paper + cold tare mass was taken.

Table 10 Filter Paper Water Content Measurement Worksheet for 2nd Set of Experiments (Equilibration Period = 48 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET											
Date experiment set up: 12/06/12											
Equilibration Period: 48 days											Test Date: 01/23/13-01/24/13
Moisture tin No.:		E5	E6	E7	E8	E9	F1	F2	F3	F4	F5
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	37.05704	36.8185	36.62735	36.73728	36.97937	37.01266	36.6752	36.90315	36.79167	36.94881
Mass of wet filter paper + cold tare mass, g	M_1	37.38596	37.14521	36.92016	37.02028	37.23923	37.26144	36.95418	37.18529	37.1873	37.36194
Mass of dry filter paper + hot tare mass, g	M_2	37.25938	37.02058	36.78794	36.89219	37.1369	37.16395	36.83378	37.06646	36.98755	37.14989
Hot tare mass, g	T_h	37.04234	36.80153	36.61435	36.72127	36.96055	36.99218	36.65738	36.88602	36.77266	36.93097
Mass of dry filter paper, g $(M_2 - T_h)$	M_f	0.21704	0.21905	0.17359	0.17092	0.17635	0.17177	0.1764	0.18044	0.21489	0.21892
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.11188	0.10766	0.11922	0.11208	0.08351	0.07701	0.10258	0.1017	0.18074	0.19421
Water content of filter paper, g (M_w/M_f)	W_f	0.515481	0.491486	0.686791	0.655745	0.473547	0.448332	0.581519	0.563622	0.841081	0.887128
Suction, log Kpa	h_1	1.98	1.98	0.00	0.00	0.00	0.00	1.38	1.38	0.99	0.99
		Wetting		Drying		Drying		Drying		Drying	
		0.02M		De-ionized water		De-ionized water		0.005M		0.002M	
		Whatman No. 42		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42	

Table 10 Filter Paper Water Content Measurement Worksheet for 2nd Set of Experiments (Equilibration Period = 48 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 12/06/12

Equilibration Period: 48 days

Test Date: 01/23/13-01/24/13

Moisture tin No.:		F6*	F7*	F8	F9	G1	G2	G3	G4	G5	G6	G8	G9
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.6478	36.7735	36.8182	36.7183	36.91665	36.98077	36.91672	36.7522	36.73573	36.06813	36.6604	36.7458
Mass of wet filter paper + cold tare mass, g	M_1	37.3036	37.5509	37.1479	37.0474	37.16577	37.22751	37.19128	37.0258	37.0986	36.42466	37.0035	37.0895
Mass of dry filter paper + hot tare mass, g	M_2	36.8493	36.9690	37.0139	36.9161	37.07629	36.13745	37.07492	36.9083	36.89064	36.22079	36.8623	36.9484
Hot tare mass, g	T_h	36.6313	36.7541	36.7988	36.6971	36.89781	35.95972	36.90455	36.7319	36.71675	36.04773	36.6426	36.7276
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.21802	0.21496	0.21509	0.21897	0.17848	0.17773	0.17037	0.17634	0.17389	0.17306	0.21966	0.22082
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.4378	0.56247	0.1146	0.1102	0.07064	0.06901	0.10419	0.09726	0.18898	0.18347	0.12341	0.12292
Water content of filter paper, g (M_w/M_f)	W_f	2.00807	2.61662	0.5328	0.50326	0.395787	0.388286	0.611551	0.55154	1.086779	1.060152	0.56182	0.55665
Suction, log Kpa	h_1	0.99	0.99	1.38	1.38	2.96	2.96	0.99	0.99	0.99	0.99	1.38	1.8
		Drying		Drying		Drying		Drying		Drying		Drying	
		0.002M		0.005M		0.2		0.002M		0.002M		0.005M	
		Whatman No. 42		Whatman No. 42		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42	

* Papers were too wet.

Table 11 Filter Paper Water Content Measurement Worksheet for 3rd Set of Experiments (Equilibration Period = 76 days)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 02/19/13

Equilibration Period: 76 days Test Date: 05/06/13-05/07/13

Moisture tin No.:	A1	A2	A3	A4*	A5	A6	A7	A8	A9	B1
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.9393	36.864	36.7198		36.779	36.691	36.7838	36.7851	37.0454	36.7488
Mass of wet filter paper + cold tare mass, g M_1	37.2272	37.147	37.0078		37.121	37.031	37.102	37.1098	37.36	37.0689
Mass of dry filter paper + hot tare mass, g M_2	37.1078	37.027	36.8849		36.985	36.897	36.9792	36.9898	37.2408	36.951
Hot tare mass, g T_h	36.93	36.851	36.7075		36.767	36.677	36.769	36.7735	37.0299	36.7348
Mass of dry filter paper, g (M_2 -T_h) M_f	0.17778	0.1765	0.17734	0	0.218	0.22	0.21018	0.21632	0.21086	0.21624
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.11008	0.1067	0.11064	0	0.1235	0.1209	0.10802	0.10835	0.10368	0.10381
Water content of filter paper, g (M_w/M_f) W_f	0.61919	0.6044	0.62389		0.5663	0.5494	0.51394	0.50088	0.4917	0.48007
Suction, log Kpa h_1	0.00	0.00	0.99	0.99	1.38	1.38	0.00	0.00	1.98	1.98
	Drying		Drying		Drying		Drying		Drying	
	De-ionized water		0.002M		0.005M		De-ionized		0.02M	
	Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42		Whatman No. 42		Whatman No. 42	

* A4 - paper slipped into solution.

Table 11 Filter Paper Water Content Measurement Worksheet for 3rd Set of Experiments (Equilibration Period = 76 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 02/19/13

Equilibration Period: 76 days

Test Date: 05/06/13-05/07/13

Moisture tin No.:		B2	B3	B4	B5	B6	B7	B8	B9	C1	C2
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.9299	36.9064	36.9757	36.7778	36.8292	36.857	36.924	36.7136	36.936	36.8499
Mass of wet filter paper + cold tare mass, g	M_1	37.255	37.243	37.3047	37.1046	37.1862	37.2017	37.2042	36.99	37.2178	37.1308
Mass of dry filter paper + hot tare mass, g	M_2	37.1324	37.1157	37.1787	36.9776	37.0338	37.0566	37.0868	36.8771	37.0976	37.014
Hot tare mass, g	T_h	36.9175	36.8939	36.9599	36.7598	36.8143	36.8416	36.9083	36.6994	36.9221	36.8352
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.21483	0.22176	0.2188	0.21779	0.21951	0.215	0.17847	0.1777	0.1755	0.17885
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.11024	0.11487	0.11021	0.10904	0.13752	0.12965	0.10171	0.09871	0.10635	0.10204
Water content of filter paper, g (M_w/M_f)	W_f	0.51315	0.51799	0.5037	0.50067	0.62649	0.60302	0.5699	0.55549	0.60598	0.57053
Suction, log Kpa	h_1	0.00	0.00	1.98	1.98	0.00	0.00	1.98	1.98	0.00	0.00
		Drying		Drying		Drying		Drying		Drying	
		De-ionized water		0.002M		De-ionized water		0.02M		De-ionized water	
		Whatman No. 42		Whatman No. 42		Whatman No. 42		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]	

Table 11 Filter Paper Water Content Measurement Worksheet for 3rd Set of Experiments (Equilibration Period = 76 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 02/19/13

Equilibration Period: 76 days

Test Date: 05/06/13-05/07/13

Moisture tin No.:		C3	C4	C5	C6	C7	C8	C9	D1*	D2	D3
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.8926	36.8415	36.7437	36.6147	36.8919	36.9445	36.8016	36.8771	36.8989	36.9457
Mass of wet filter paper + cold tare mass, g	M_1	37.2943	37.2375	37.0667	36.9407	37.1688	37.2066	37.0929	37.1605	37.2005	37.2356
Mass of dry filter paper + hot tare mass, g	M_2	37.0981	37.0444	36.9404	36.8173	37.053	37.0968	36.9643	37.038	37.0648	37.1039
Hot tare mass, g	T_h	36.8808	36.8281	36.7319	36.6012	36.8761	36.9272	36.7854	36.8653	36.883	36.9251
Mass of dry filter paper, g $(M_2 - T_h)$	M_f	0.21733	0.21635	0.20852	0.21614	0.17689	0.16964	0.17896	0.1727	0.18183	0.17881
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.1843	0.17966	0.11452	0.1098	0.10009	0.09247	0.11235	0.11071	0.11977	0.11111
Water content of filter paper, g (M_w/M_f)	W_f	0.84802	0.83041	0.5492	0.508	0.56583	0.5451	0.62779	0.64105	0.65869	0.62139
Suction, log Kpa	h_1	0.99	0.99	1.38	1.38	1.38	1.38	0.99	0.99	0.00	0.00
		Drying		Drying		Drying		Drying		Drying	
		0.002M		0.005M		0.005M		0.002M		De-ionized water	
		Whatman No. 42		Whatman No. 42		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2	

* D1 - Paper touched the side of the jar.

Table 11 Filter Paper Water Content Measurement Worksheet for 3rd Set of Experiments (Equilibration Period = 76 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 02/19/13

Equilibration Period: 76 days

Test Date: 05/06/13-05/07/13

Moisture tin No.:		D4	D5	D6	D7	D8	D9	E1	E2	E3	E4
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.3593	36.3878	36.4703	36.6457	36.4181	36.418	36.7228	36.9253	36.8734	36.8156
Mass of wet filter paper + cold tare mass, g	M_1	36.6235	36.6488	36.7208	36.8996	36.7382	36.7371	36.9786	37.166	37.1283	37.0759
Mass of dry filter paper + hot tare mass, g	M_2	36.5175	36.5441	36.6209	36.7997	36.6168	36.6188	36.8875	37.0793	37.029	36.9777
Hot tare mass, g	T_h	36.341	36.3686	36.4538	36.6273	36.398	36.4016	36.7067	36.9102	36.8546	36.8008
Mass of dry filter paper, g $(M_2 - T_h)$	M_f	0.17653	0.1755	0.16703	0.17237	0.21876	0.21714	0.18083	0.16911	0.17441	0.17699
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.0877	0.08553	0.08347	0.08155	0.10132	0.10194	0.07496	0.07152	0.08051	0.08333
Water content of filter paper, g (M_w/M_f)	W_f	0.4968	0.48735	0.49973	0.47311	0.46316	0.46947	0.41453	0.42292	0.46161	0.47082
Suction, log Kpa	h_1	1.38	1.38	1.38	1.38	0.99	0.99	0.00	0.000	1.38	1.38
		Drying		Wetting		Wetting		Wetting		Wetting	
		0.005M		0.005M		0.002M		De-ionized water		0.005M	
		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2		Whatman No. 42		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2	

Table 11 Filter Paper Water Content Measurement Worksheet for 3rd Set of Experiments (Equilibration Period = 76 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 02/19/13

Equilibration Period: 76 days

Test Date: 05/06/13-05/07/13

Moisture tin No.:	E5	E6	E7	E8	E9	F1	F2	F3	F4	F5	F6	F7
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	37.0569	36.8189	36.6276	36.7373	36.9792	37.0125	36.6734	36.9033	36.7913	36.9491	36.6479	36.7696
Mass of wet filter paper + cold tare mass, g M_1	37.3662	37.1274	36.8739	36.9798	37.2497	37.2869	36.9802	37.2059	37.0988	37.2582	36.9646	37.0775
Mass of dry filter paper + hot tare mass, g M_2	37.2536	37.0192	36.7885	36.8961	37.1399	37.1782	36.8758	37.1046	36.9885	37.1541	36.8592	36.9735
Hot tare mass, g T_h	37.0385	36.8015	36.6093	36.7203	36.9615	36.9943	36.6568	36.8853	36.7723	36.9354	36.6357	36.7571
Mass of dry filter paper, g (M_2 -T_h) M_f	0.21514	0.21768	0.1792	0.17579	0.17839	0.1839	0.21895	0.21933	0.21626	0.21873	0.22356	0.21633
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.09413	0.09084	0.06708	0.06668	0.09209	0.09057	0.08785	0.08323	0.09122	0.0904	0.09313	0.09149
Water content of filter paper, g (M_w/M_f) W_f	0.43753	0.41731	0.37433	0.37932	0.51623	0.4925	0.40123	0.37947	0.42181	0.41329	0.41658	0.42292
Suction, log Kpa h_1	0.99	0.99	0.00	0.00	0.99	0.99	0.99	0.99	0.00	0.00	0.00	0.00
	Wetting		Wetting		Wetting		Wetting		Wetting		Wetting	
	0.002M		De-ionized		0.002M		0.02M		De-ionized		De-ionized	
	Whatman No. 42		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42		Whatman No. 42		Whatman No. 42	

Table 11 Filter Paper Water Content Measurement Worksheet for 3rd Set of Experiments (Equilibration Period = 76 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 02/19/13

Equilibration Period: 76 days

Test Date: 05/06/13-05/07/13

Moisture tin No.:	F8	F9	G1	G2*	G3	G4	G5	G6	G7	G8	G9	H1
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.8179	36.7163	36.9167		36.9165	36.7515	36.7351	36.0681	35.8626	36.6602	36.7454	36.2503
Mass of wet filter paper + cold tare mass, g M_1	37.1313	37.0274	37.1708		37.2327	37.0613	36.9806	36.3197	36.1641	36.9592	36.9961	36.5034
Mass of dry filter paper + hot tare mass, g M_2	37.0192	36.9256	37.0692		37.1209	36.956	36.892	36.2301	36.0642	36.8637	36.9018	36.4122
Hot tare mass, g T_h	36.7992	36.7055	36.8985		36.9003	36.7346	36.7185	36.0498	35.846	36.6449	36.7298	36.2327
Mass of dry filter paper, g (M_2 -T_h) M_f	0.21993	0.22017	0.17068		0.22057	0.22139	0.17354	0.18035	0.21812	0.21885	0.17204	0.1795
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.09349	0.09087	0.08343		0.09559	0.08846	0.07195	0.07128	0.08345	0.0802	0.07863	0.07357
Water content of filter paper, g (M_w/M_f) W_f	0.42509	0.41273	0.48881		0.43338	0.39957	0.4146	0.39523	0.38259	0.36646	0.45704	0.40986
Suction, log Kpa h_1	0.00	0.00	0.00		1.38	1.38	1.98	1.98	1.38	1.38	0.99	0.99
	Wetting		Wetting		Wetting		Wetting		Wetting		Wetting	
	De-ionized water		De-ionized water		0.005M		0.02M		0.005M		0.002M	
	Whatman No. 42		Schleicher and Schuell No. 589[2]		Whatman No. 42		Schleicher and Schuell No. 589[2]		Whatman No. 42		Schleicher and Schuell No. 589[2]	

* G2 - paper slipped into solution.

Table 12 Filter Paper Water Content Measurement Worksheet for 4th Set of Experiments (Equilibration Period = 42 days)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 05/09/13

Equilibration Period: **42 days**

Test Date: 06/20/13 -06/21/13

Moisture tin No.:	A1*	A2*	A3	A4	A5	A6	A7	A8**	A9	B1
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.9404	36.8637	36.7201	36.9861	36.7797	36.6908	36.7837		37.0456	36.7498
Mass of wet filter paper + cold tare mass, g M_1	37.4596	37.4335	37.1139	37.3902	37.0009	36.909	37.0128		37.5003	37.2072
Mass of dry filter paper + hot tare mass, g M_2	37.1111	37.0311	36.8852	37.1583	36.9483	36.8573	36.95		37.2109	36.9191
Hot tare mass, g T_h	36.9275	36.8504	36.7048	36.9742	36.76695	36.6782	36.7725		37.0315	36.7386
Mass of dry filter paper, g (M_2 -T_h) M_f	0.18354	0.18068	0.1804	0.18409	0.18139	0.17909	0.17749		0.17936	0.18045
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.33559	0.3892	0.21342	0.21993	0.03984	0.03916	0.05162		0.27542	0.27696
Water content of filter paper, g (M_w/M_f) W_f	1.82843	2.15408	1.18304	1.19469	0.21964	0.21866	0.29083		1.53557	1.53483
Suction, log Kpa h_1	0.00	0.00	0.99	0.99	1.38	1.38	0.00		1.98	1.98
	Drying		Drying		Drying		Drying		Drying	
	0.2M		0.02M		2.2M		1.0M		0.002M	
	Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2		Schleicher and Schuell No. 589^2	

Table 12 Filter Paper Water Content Measurement Worksheet for 4th Set of Experiments (Equilibration Period = 42 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 05/09/13

Equilibration Period: **42 days** Test Date: 06/20/13 -06/21/13

Moisture tin No.:	B2	B3	B4	B5	B6	B7	B8	B9*	C1	C2
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.9316	36.9064	36.9761	36.7779	36.8299	36.8572	36.9247	36.7135	36.9363	36.8507
Mass of wet filter paper + cold tare mass, g M_1	37.3361	37.3131	37.4284	37.2776	37.2579	37.2915	37.3674	37.158	37.2429	37.1618
Mass of dry filter paper + hot tare mass, g M_2	37.1008	37.0793	37.14	36.94	36.995	37.0206	37.1324	36.9233	37.1409	37.0607
Hot tare mass, g T_h	36.9192	36.895	36.9642	36.7631	36.8174	36.8431	36.9109	36.7	36.9208	36.8359
Mass of dry filter paper, g (M_2 -T_h) M_f	0.18163	0.1843	0.17584	0.17694	0.1776	0.17753	0.22149	0.22332	0.22012	0.22478
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.22289	0.22243	0.2764	0.32278	0.25047	0.25672	0.22124	0.22116	0.08641	0.08629
Water content of filter paper, g (M_w/M_f) W_f	1.22717	1.20689	1.57188	1.82423	1.4103	1.44607	0.99887	0.99033	0.39256	0.38389
Suction, log Kpa h_1	0.00	0.00	1.98	1.98	0.00	0.00	1.98	1.98	0.00	0.00
	Drying		Drying		Drying		Drying		Drying	
	De-ionized water		De-ionized water		0.005M		0.02M		0.2M	
	Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42		Whatman No. 42	

* Paper touched the side of the jar before it was transferred to a moisture tin.

Table 12 Filter Paper Water Content Measurement Worksheet for 4th Set of Experiments (Equilibration Period = 42 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 05/09/13

Equilibration Period: **42 days**

Test Date: 06/20/13 -06/21/13

Moisture tin No.:		C3	C4*	C5	C6	C7	C8	C9	D1	D2	D3
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.8936	36.8423	36.7443	36.6169	36.8927	36.9447	36.8021	36.8779	36.8991	36.9461
Mass of wet filter paper + cold tare mass, g	M_1	37.1688	37.1213	37.0093	36.8869	37.4395	37.4877	37.3465	37.4189	37.3836	37.4521
Mass of dry filter paper + hot tare mass, g	M_2	37.0923	37.0433	36.9502	36.8272	37.0985	37.149	37.0041	37.0781	37.0906	37.1447
Hot tare mass, g	T_h	36.8781	36.8254	36.731	36.6038	36.8772	36.9282	36.7844	36.8611	36.8837	36.9295
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.21417	0.21795	0.21918	0.22338	0.22128	0.22084	0.21968	0.21702	0.20682	0.21518
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.06099	0.06106	0.04586	0.04661	0.3255	0.32215	0.32472	0.32392	0.27764	0.29086
Water content of filter paper, g (M_w/M_f)	W_f	0.28477	0.28016	0.20923	0.20866	1.47099	1.45875	1.47815	1.49258	1.34242	1.35171
Suction, log Kpa	h_1	0.99	0.99	1.38	1.38	1.38	1.38	0.99	0.99	0.00	0.00
		Drying		Drying		Drying		Drying		Drying	
		1.0M		2.2M		De-ionized water		De-ionized water		0.002M	
		Whatman No. 42		Whatman No. 42		Whatman No. 42		Whatman No. 42		Whatman No. 42	

* Paper touched the side of the jar before it was transferred to a moisture tin.

Table 12 Filter Paper Water Content Measurement Worksheet for 4th Set of Experiments (Equilibration Period = 42 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 05/09/13

Equilibration Period: **42 days** Test Date: 06/20/13 -06/21/13

Moisture tin No.:		D4	D5	D6	D7	D8	D9	E1	E2	E3	E4
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	36.3601	36.3884	36.471	36.6464	36.4194	36.4189	36.7244	36.9258	36.8744	36.8167
Mass of wet filter paper + cold tare mass, g	M_1	36.8727	36.914	36.7669	36.9496	36.6954	36.7088	37.0635	37.2653	37.2103	37.1517
Mass of dry filter paper + hot tare mass, g	M_2	36.5552	36.5869	36.6311	36.8106	36.5772	36.5883	36.9241	37.1271	37.0797	37.0238
Hot tare mass, g	T_h	36.3423	36.3722	36.4528	36.6281	36.4045	36.4054	36.7106	36.9116	36.8594	36.8032
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.21288	0.21468	0.17827	0.18245	0.17264	0.18282	0.21354	0.21551	0.22032	0.22062
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.29977	0.31098	0.11761	0.12074	0.10333	0.10707	0.1255	0.12401	0.11558	0.11442
Water content of filter paper, g (M_w/M_f)	W_f	1.40816	1.44857	0.65973	0.66177	0.59853	0.58566	0.58771	0.57543	0.5246	0.51863
Suction, log Kpa	h_1	1.38	1.38	1.38	1.38	0.99	0.99	0.00	0.00	1.38	1.38
		Drying		Wetting		Wetting		Wetting		Wetting	
		0.005M		Deionized water		Deionized water		Deionized water		Deionized water	
		Whatman No. 42		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42		Whatman No. 42	

Table 12 Filter Paper Water Content Measurement Worksheet for 4th Set of Experiments (Equilibration Period = 42 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET													
Date experiment set up: 05/09/13													
Equilibration Period: 42 days												Test Date: 06/20/13 -06/21/13	
Moisture tin No.:		E5	E6	E7	E8	E9	F1	F2	F3	F4	F5	F6	F7
Top or bottom filter paper		T	B	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g	T_c	37.0581	36.8198	36.6285	36.7383	36.9803	37.0138	36.6761	36.9041	36.7926	36.9503	36.649	36.7753
Mass of wet filter paper + cold tare mass, g	M_1	37.3386	37.1051	36.9444	37.0597	37.2405	37.2692	37.0345	37.2635	37.1097	37.2562	36.9318	37.0605
Mass of dry filter paper + hot tare mass, g	M_2	37.2216	36.989	36.8286	36.9421	37.1401	37.1729	36.8805	37.1082	36.9952	37.1415	36.8127	36.9373
Hot tare mass, g	T_h	37.043	36.8039	36.613	36.7223	36.9644	36.9969	36.6608	36.888	36.7763	36.9309	36.6348	36.7586
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.17859	0.1851	0.21567	0.21976	0.17568	0.17602	0.21965	0.22018	0.21888	0.2106	0.17784	0.17877
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.1019	0.10021	0.10027	0.10165	0.08452	0.07936	0.13876	0.13926	0.09826	0.09527	0.10498	0.10641
Water content of filter paper, g (M_w/M_f)	W_f	0.57058	0.54138	0.46492	0.46255	0.4811	0.45086	0.63173	0.63248	0.44892	0.45237	0.59031	0.59523
Suction, log Kpa	h_1	0.99	0.99	0.00	0.00	0.99	0.99	0.99	0.99	0.00	0.00	0.00	0.00
		Wetting		Wetting		Wetting		Wetting		Wetting		Wetting	
		0.005M		0.02M		0.02M		0.002M		0.005M		0.002M	
		Schleicher and Schuell No. 589[2]		Whatman No. 42		Schleicher and Schuell No. 589[2]		Whatman No. 42		Whatman No. 42		Schleicher and Schuell No. 589[2]	

Table 12 Filter Paper Water Content Measurement Worksheet for 4th Set of Experiments (Equilibration Period = 42 days) (Cont'd)

THE FILTER PAPER METHOD SUCTION MEASUREMENT WORKSHEET

Date experiment set up: 05/09/13

Equilibration Period: 42 days

Test Date: 06/20/13 -06/21/13

Moisture tin No.:	F8	F9	G1	G2	G3	G4	G5	G6	G7	G8	G9	H1
Top or bottom filter paper	T	B	T	B	T	B	T	B	T	B	T	B
Cold tare mass, g T_c	36.8196	36.7193	36.9175	35.9819	36.9171	36.7524	36.7358	36.0685	35.8632	36.66076	36.7463	36.251
Mass of wet filter paper + cold tare mass, g M_1	37.0329	36.9451	37.1712	36.2338	37.2028	37.0393	36.9838	36.3103	36.072	36.86964	37.0085	36.5139
Mass of dry filter paper + hot tare mass, g M_2	36.9732	36.8806	37.1172	36.1796	37.1156	36.9546	36.9067	36.2315	36.0209	36.8187	36.942	36.4474
Hot tare mass, g T_h	36.8027	36.7004	36.9016	35.9655	36.9015	36.7367	36.7209	36.0523	35.8453	36.64344	36.7296	36.2328
Mass of dry filter paper, g $(M_2 - T_h)$ M_f	0.17051	0.18026	0.21558	0.21417	0.21404	0.21789	0.18574	0.17918	0.17555	0.17526	0.21248	0.21461
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.04275	0.04556	0.03809	0.03777	0.07159	0.06909	0.06221	0.0626	0.03332	0.03362	0.04969	0.04832
Water content of filter paper, g (M_w/M_f) W_f	0.25072	0.25275	0.17669	0.17636	0.33447	0.31709	0.33493	0.34937	0.1898	0.191829	0.23386	0.22515
Suction, log Kpa h_1	0.00	0.00	0.00	0.00	1.38	1.38	1.98	1.98	1.38	1.38	0.99	0.99
	Wetting		Wetting		Wetting		Wetting		Wetting		Wetting	
	1.0M		2.2M		0.2M		0.2M		2.2M		1.0M	
	Schleicher and Schuell No. 589[2]		Whatman No. 42		Whatman No. 42		Schleicher and Schuell No. 589[2]		Schleicher and Schuell No. 589[2]		Whatman No. 42	

APPENDIX B – SUMMARY OF THE RESULTS OF LABORATORY EXPERIMENTS

Table 13 Summary of Water Content and Total Suction Data of 1st Set of Experiments (Equilibration Period = 31 days)

Date experiment was set up: 10/15/12							
Equilibration Period: 31 days					Test Date: 11/15/2012 & 12/16/2012		
Drying - Whatman No. 42 Filter Paper							
Solution	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**1.0M**	**2.2M**
WC - Top Paper	0.558132	1.528153	1.694104	0.830218	10.84852	0.277	0.196565
WC - Bottom Paper	0.565846	1.537253	2.0664	0.834464	11.64779	0.273381	0.201919
Suction, log Kpa	0.00	0.99	1.38	1.98	2.96	3.67	4.04
Drying - Schleicher and Schuell No. 589^2 Filter Paper							
Solution	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**1.0M**	**2.2M**
WC - Top Paper	1.245409302	1.027317	0.921868	0.605927	2.137374	0.28034	0.207931
WC - Bottom Paper	1.289508341	1.05134	0.987715	0.610963	2.649786		0.185958
Suction, log Kpa	0.00	0.99	1.38	1.98	2.96	3.67	4.04
Wetting - Whatman No. 42 Filter Paper							
Solution	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**1.0M**	**2.2M**
WC - Top Paper	0.420866249	0.369309	0.363763	0.043853	0.2692	0.198136	0.154484
WC - Bottom Paper	0.412312677	0.378787	0.372487	0.37907	0.27244	0.201673	0.163952
Suction, log Kpa	0.00	0.99	1.38	1.98	2.96	3.67	4.04
Wetting - Schleicher and Schuell No. 5892 Filter Paper							
Solution	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**1.0M**	**2.2M**
WC - Top Paper	0.413202085	0.403197	0.378335	0.389099	0.335223	0.231396	0.177238
WC - Bottom Paper	0.40482987	0.407168	0.373235	0.381561	0.311877	0.22226	0.159468
Suction, log Kpa	0.00	0.99	1.38	1.98	2.96	3.67	4.04

Table 14 Summary of Water Content and Total Suction Data of 2nd Set of Experiments (Equilibration Period = 48 days)

Date experiment was set up: 12/06/12

Equilibration Period: 48 days Test Date: 01/23/13 - 01/24/13

Drying – Whatman No. 42 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.002M**	**0.005M**	**0.005M**	**0.02M**	**0.2M**
WC - Top Paper	0.6306994	0.671097	0.84108	2.00807	0.5328	0.56182	0.399835	
WC - Bottom Paper	0.6122772	0.672849	0.88713	2.61663	0.50327	0.55665	0.753286	
Suction, log Kpa	0.00	0.00	0.99	0.99	1.38	1.38	1.98	

Drying - Schleicher and Schuell No. 589^2 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.002M**	**0.005**	**0.005M**	**0.02M**	**0.2M**
WC - Top Paper	0.6867907	0.473547	1.08678	0.56182	0.54306	0.58152	0.473804	0.395787
WC - Bottom Paper	0.6557454	0.448332	1.06015	0.55665	0.52664	0.56362	0.464441	0.388286
Suction, log Kpa	0.00	0.00	0.99	0.99	1.38	1.38	1.98	2.96

Wetting – Whatman No. 42 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.002M**	**0.005**	**0.005M**	**0.02M**	**0.2M**
WC - Top Paper	0.3949902	0.397816	0.38724	0.38232	0.40425	0.40273	0.515481	0.280126
WC - Bottom Paper	0.3653199	0.410507	0.3904	0.37621	0.39466	0.38739	0.491486	0.29122
Suction, log Kpa	0.00	0.00	0.99	0.99	1.38	1.38	1.98	2.96

Wetting - Schleicher and Schuell No. 589^2 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.002M**	**0.005M**	**0.005M**	**0.02M**	**0.2M**
WC - Top Paper	0.4018686	0.381981	0.40688	0.37103	0.41924	0.4347	0.413608	0.334861
WC - Bottom Paper	0.3726967	0.359246	0.41197	0.35957	0.40984	0.44906	0.373362	0.330534
Suction, log Kpa	0.00	0.00	0.99	0.99	1.38	1.38	1.98	2.96

Table 15 Summary of Water Content and Total Suction Data of 3^{rd} Set of Experiments (Equilibration Period = 76 days)

Date experiment was set up: 02/19/13								
Equilibration Period: 76 days								Test Date: 05/06/13- 05/07/13
Drying - Whatman Filter Paper								
Solution	**Deionized Water**	**Deionized Water**	**Deionized water**	**0.002M**	**0.002M**	**0.005M**	**0.005M**	**0.02M**
WC - Top Paper	0.5139404	0.51315	0.62649	0.84802	0.5037	0.56628	0.5492	0.4917
WC - Bottom Paper	0.5008783	0.51799	0.60302	0.83041	0.50067	0.54937	0.508	0.48007
Suction, log Kpa	0.00	0.00	0.00	0.99	0.99	1.38	1.38	1.98
Drying - S& S Filter Paper								
Solution	**Deionized Water**	**Deionized Water**	**Deionized water**	**0.002M**	**0.002M**	**0.005**	**0.005M**	**0.02M**
WC - Top Paper	0.6191923	0.60598	0.65869	0.62389	0.62779	0.56583	0.4968	0.5699
WC - Bottom Paper	0.6044205	0.57053	0.62139		0.64105	0.5451	0.48735	0.55549
Suction, log Kpa	0.00	0.00	0.00	0.99	0.99	1.38	1.38	1.98
Wetting - Whatman Filter Paper								
Solution	**Deionized Water**	**Deionized Water**	**Deionized water**	**0.002M**	**0.002M**	**0.005**	**0.005M**	**0.02M**
WC - Top Paper	0.4218071	0.41658	0.42509	0.43753	0.46316	0.43338	0.38259	0.40123
WC - Bottom Paper	0.4132949	0.42292	0.41273	0.41731	0.46947	0.39957	0.36646	0.37947
Suction, log Kpa	0.00	0.00	0.00	0.99	0.99	1.38	1.38	1.98
Wetting - S& S Filter Paper								
Solution	**Deionized Water**	**Deionized Water**	**Deionized water**	**0.002M**	**0.002M**	**0.005M**	**0.005M**	**0.02M**
WC - Top Paper	0.414533	0.37433	0.48881	0.51623	0.45704	0.49973	0.46161	0.4146
WC - Bottom Paper	0.42292	0.37932		0.4925	0.40986	0.47311	0.47082	0.39523
Suction, log Kpa	0.00	0.00	0.00	0.99	0.99	1.38	1.38	1.98

Table 16 Summary of Water Content and Total Suction Data of 4th Set of Experiments (Equilibration Period = 42 days)

Date experiment was set up: 05/09/13

Equilibration Period: 42days Test Date: 06/20/13 - 06/21/13

Drying - Whatman No. 42 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**1.0M**	**2.2M**
WC - Top Paper	1.470987	1.47815	1.342423	1.408164	0.998871	0.392559	0.284774	0.209234
WC - Bottom Paper	1.458748	1.492581	1.351706	1.448575	0.990328	0.383886	0.280156	0.208658
Suction, log Kpa	0.00	0.00	0.99	1.38	1.98	2.96	3.67	4.04

Drying - Schleicher and Schuell No. 589^2 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**2.2M**	**1.0M**
WC - Top Paper	1.227165	1.571884	1.535571	1.410304	1.183038	1.82843	0.219637	0.290833
WC - Bottom Paper	1.206891	1.824234	1.53483	1.446065	1.194687	2.154085	0.218661	
Suction, log Kpa	0.00	0.00	0.99	1.38	1.98	2.96	4.04	3.67

Wetting - Whatman No. 42 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**1.0M**	**2.2M**
WC - Top Paper	0.587712	0.524601	0.631732	0.448922	0.464923	0.33447	0.233857	0.176686
WC - Bottom Paper	0.575426	0.518629	0.632483	0.452374	0.46255	0.317087	0.225153	0.176355
Suction, log Kpa	0.00	0.00	0.99	1.38	1.98	2.96	3.67	4.04

Wetting - Schleicher and Schuell No. 589^2 Filter Paper

Solution	**Deionized Water**	**Deionized Water**	**0.002M**	**0.005M**	**0.02M**	**0.2M**	**1.0M**	**2.2M**
WC - Top Paper	0.65973	0.598529	0.590306	0.570581	0.481102	0.334931	0.250718	0.189803
WC - Bottom Paper	0.66177	0.585658	0.595234	0.541383	0.450858	0.349369	0.252746	0.191829
Suction, log Kpa	0.00	0.00	0.99	1.38	1.98	2.96	3.67	4.04

APPENDIX C – SOAKED/SATURATED AND OUT-OF-THE-BOX FILTER PAPER WATER CONTENT MEASUREMENT WORKSHEETS

Table 17 Initial Water Content Measurement Worksheet of Saturated/Soaked Filter Papers

INITIAL WATER CONTENT VALUES OF SATURATED/ SOAKED FILTER PAPERS												
	Test Date: 05/09 and 10/2013				Test Date: 12/3 & 4/2012				Test Date: 12/17 and 18/2012			
	Soaking Period				Soaking Period				Soaking Period			
	26 hours				**4 days**				**18 days**			
Moisture tin No.:	A1	A2	A3	A4	A1	A2	A3	A4	A1	A2	A3	A4
Cold tare mass, g T_c	36.9399	36.8637	36.7197	36.9857	36.9392	36.8639	36.7194	36.986	36.9395	36.8637	36.7201	36.9856
Mass of wet filter paper + cold tare mass, g M_1	37.5571	37.4616	37.2787	37.5116	37.571	37.5265	37.3019	37.565	37.5733	37.4981	37.2678	37.5388
Mass of dry filter paper + hot tare mass, g M_2	37.139	37.0603	36.8853	37.1413	37.153	37.0763	36.8835	37.157	37.1385	37.068	36.8787	37.1471
Hot tare mass, g T_h	36.9217	36.8494	36.7036	36.9677	36.9337	36.8524	36.7077	36.976	36.9258	36.8504	36.7057	36.9707
Mass of dry filter paper, g (M_2 -T_h) M_f	0.21731	0.21092	0.18164	0.17356	0.21931	0.22391	0.17578	0.1816	0.2127	0.2176	0.173	0.17642
Mass of water in filter paper, g (M1 - M2 - Tc + Th) M_w	0.39985	0.38699	0.37743	0.35238	0.41243	0.4387	0.40669	0.3978	0.42114	0.4168	0.37473	0.37676
Water content of filter paper, g (M_w/M_f) W_f	1.84	1.83477	2.0779	2.03031	1.88058	1.95927	2.31363	2.1902	1.97997	1.91544	2.16607	2.13559
	Whatman No. 42		Schleicher and Schuell No. 589[2]		Whatman No. 42		Schleicher and Schuell No. 589[2]		Whatman No. 42		Schleicher and Schuell No. 589[2]	

Table 18 Out-of-the-Box Filter Paper Water Content Measurement Worksheet

INITIAL WATER CONTENT VALUES OF OUT-OF-THE-BOX FILTER PAPERS					
					Test Date: 06/23 and 24/2013
Moisture tin No.:		A1	A2	A3	A4
Cold tare mass, g	T_c	36.94008	36.86402	36.72023	36.98626
Mass of wet filter paper + cold tare mass, g	M_1	37.17454	37.09516	36.91868	37.17576
Mass of dry filter paper + hot tare mass, g	M_2	37.14049	37.05847	36.88685	37.14283
Hot tare mass, g	T_h	36.92576	36.84672	36.70702	36.97035
Mass of dry filter paper, g (M_2 -T_h)	M_f	0.21473	0.21175	0.17983	0.17248
Mass of water in filter paper, g (M1 - M2 - Tc + Th)	M_w	0.01973	0.01939	0.01862	0.01702
Water content of filter paper, g (M_w/M_f)	W_f	0.09188	0.09157	0.10354	0.09867
Average water content		**9.17265 %**		**10.11101 %**	
		Whatman No. 42		**Schleicher and Schuell No. 589[2]**	

APPENDIX D – NONLINEAR REGRESSION ANALYSIS USING SPSS

Non-linear Regression Analysis using SPSS

SPSS is a statistical software package that allows one to organize, assess, manipulate, and analyze data. SPSS was chosen because of its ease of use.

The nonlinear regression procedure using SPSS is outlined here-under.

i) Create data file

When the SPSS is opened, the initial window will ask if the user wants to open an existing file. The window can be closed by clicking the "Cancel" button.

The Data Window (Dataset) has two tabs – Data View and Variable View – in the lower left corner which can be toggled between.

Data View is used to input and access data. The Variable View is used to specify the details of each variable in the data file. In the Variable View details such as name, type, width, decimals, etc can be specified for each variable. In Variable View, each row corresponds to a variable and each column corresponds to some detail or characteristic which can be specified for each variable (http://www.unt.edu/rss/class/Jon/SPSS_SC/Module1/SPSS_M1.htm). The Variable View for the wetting experimental data had only two rows: Water Content (WC) and Total Suction (ψ).

Clicking on the Data View tab will open the data spreadsheet where data is entered. The variable names typed under the Name column in the Variable View should be at the top of the columns. In the Data View, each row represents data for one sample measurement. To enter data, the cursor is positioned in the appropriate cell and the number typed. Pressing the “enter” key will move the highlighted position down one row. Pressing the “tab” key after entering a value will move the position over one column to the right. Data can also be pasted into the Data View sheet from an Excel Spreadsheet.

ii) Nonlinear Regression

This procedure gives a nonlinear model of the relationship between the dependent and a set of independent variables by using iterative procedure (SPSS Inc., 1989, 2010). For each iteration, the procedure gives parameter estimates and residual sum of squares.

The nonlinear regression results are valid only if the function (the model) specified accurately describes the relationship between the variables. Also, the initial/starting values chosen should be good for the model to converge (http://www.unt.edu/rss/class/Jon/SPSS_SC/Module1/SPSS_M1.htm).

To perform the nonlinear regression, Analyze > Regression > Nonlinear is chosen from the menus.

A Nonlinear Regression dialog box appears. The dialog box has fields that show the list of variables, Model Expression and Parameters to be entered. One dependent variable is selected from the list of variables. A model is entered in the Model Expression field. The model is the equation that includes the variables, parameters and functions that are to be fitted into the experimental data. Equation (5a) is entered into the Model field.

$$\psi = a * [e^{(\frac{w_s}{w})^M} - e]^N \qquad (5a)$$

Parameters are the parts of the model that Nonlinear Regression procedure will estimate. Parameters to be entered are: a, M and N. The following initial values (adopted form Leong et al., (2002) calibration equations) are inputted.

Whatman No. 42 filter papers:

a= 18500; M= 0.242; and N=2.248.

Scleicher and Schuell No. 589^2 filter papers:

a= 20000; M= 0.246; and N=2.058.

The saturated water content values, w_s, are taken as the maximum average water content values of the experimental data (which occurred for de-ionized water) as follows:

Whatman No. 42 filter papers:

w_s= 45

Scleicher and Schuell No. 589^2 filter papers:

w_s= 47

These values are constants and are not be estimated by the Nonlinear Regression procedure.

To run the regression analysis, one clicks “OK”. The output appears in a new window: the Output window.

The Output Window gives the following:

- Iteration history;
- Parameter estimates;
- Correlation of parameter estimates; and
- ANOVA table; and
- R^2 value.

The parameter estimates and R^2 values for the wetting calibration data of the filter papers studied is presented below.

Whatman No. 42 filter papers:

Parameter Estimates

Parameter	Estimate	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
a	23145.461	6888.436	4020.095	42270.826
m	.218	.020	.161	.274
n	2.445	.043	2.327	2.563

R^2 = 1 - (Residual Sum of Squares) / (Corrected Sum of Squares) = 1.000.

Scleicher and Schuell No. 589^2 filter papers:

Parameter Estimates

Parameter	Estimate	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
a	352596.092	620867.368	-1371208.072	2076400.257
m	.077	.052	-.068	.222
n	2.270	.091	2.017	2.524

R^2 = 1 - (Residual Sum of Squares) / (Corrected Sum of Squares) = 1.000

VITA

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