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Moisture adsorption of soil samples during the moisture content test

N. Erriah

University of Pretoria, Pretoria, South Africa (currently Jones & Wagener, Rivonia, South Africa)

ABSTRACT: The gravimetric method for determining the moisture content of a soil involves oven-drying a sample in order to remove all moisture to obtain its dry mass, thereafter expressing the water content of the soil as a percentage of its dry mass. Customary practice involves allowing the soil sample to cool upon being removed from the oven prior to obtaining its dry weight. It was believed that the action of allowing a sample to cool after drying was not necessary, thereby reducing the duration of the test as well as the possible influence of soil absorption during the cooling stage, which may affect the true moisture content of the soil. It was concluded that 3 hours in an oven is sufficient for drying a ± 70 g sample of wet clayey material, whereas 1 hour of drying time is sufficient for material consisting primarily of sands and silts. Furthermore, the results showed that each tested soil type gained some amount of mass in the form of moisture during the cooling stage, thereby deeming the cooling stage of the test unnecessary. It was concluded that the duration of the gravimetric moisture content test may be reduced, and more accurate results may be achieved.

1 INTRODUCTION

The basis of the gravimetric moisture content test entails removing the water content of the soil sample by drying it in an oven until the mass of the sample remains constant. The sample is first weighed prior to placement in an oven, dried, allowed to cool, and then weighed again to obtain the dry mass of the soil sample. The water content of the soil is thereafter calculated as a percentage of its dry soil mass.

Neither the scientific reasoning nor necessity behind the intermediate step of allowing a soil sample to cool prior to final weighing were understood. It was believed that the cooling stage was not necessary. This hypothesis formed the basis of the investigation.

2 MECHANISMS OF THERMAL MOISTURE TRANSPORT

2.1 Phase change processes

Moisture in soil may be in the form of a vapour or liquid. However, the flow of moisture through soil is primarily considered a molecular diffusion process.

A pure substance may be defined as a single chemical species that has a fixed chemical composition throughout.

Figure 1 (Ghoshdastidar 2012a) depicts a surface in space where p (pressure), v (specific volume) and T (temperature) are intensive properties, those of which are independent of the size of the system.

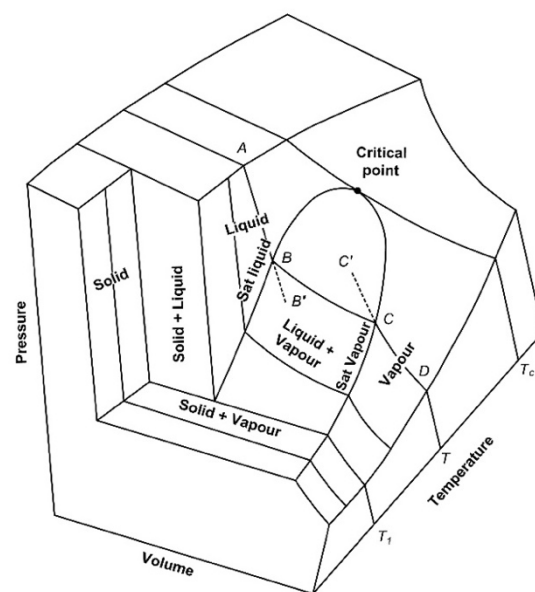


Figure 1. p - v - T surface for a pure substance (Ghoshdastidar 2012a)

The state of a pure compressible substance is determined by any two independent intensive properties, whereas extensive properties are those that depend on

the size or extent of the system. Such properties include mass (m), volume (V) and total energy (E).

The various stable equilibrium phase states are illustrated in Figure 1. The three separate stable regions are liquid, liquid and vapour, and vapour. The triple point, indicated by T_1 , and the critical point, T_c , constitute the lower limit and upper limit to a temperature range, respectively. The triple point denotes the point where all three phase states will coexist in equilibrium (Ghoshdastidar 2012a). At pressures less than the triple point pressure, no substance can exist in liquid phase in stable equilibrium. The point at which the saturated liquid and saturated vapour states cannot be distinguished defines the critical point.

The vapour phase exists exclusively at pressures greater than the critical pressure due to the indistinct liquid-to-vapour phase change process. As indicated in

Figure 1, for a constant temperature T , the pressure and volume vary along line ABCD. The liquid phase exists on its own along line AB, while only the vapour state exists along line BC. The saturation vapour curve separates the two-phase state and the superheated vapour state, whereas the saturation liquid curve separates the two-phase state and the subcooled liquid state. The specific volume (v) which is determined by its enthalpy, varies along line BC while the pressure remains invariant along this line.

The two-phase liquid + vapour surface is projected in the pressure-temperature plane in order to outline the vapour pressure curve which relates pressure and temperature between T_1 and T_c . The temperature and pressure values on this curve are referred to the saturation temperature (T_{sat}) and saturation pressure (p_{sat}), respectively.

Only stable equilibrium states are represented on the p - v - T diagram; however, other metastable or unstable states can occur where the coordinates of pressure (p), volume (v), and temperature (T) do not lie on any of the surfaces. A superheated liquid refers to an unstable liquid state achieved by the process of *superheating*, which involves meticulously heating a liquid above saturation temperature corresponding to the imposed static pressure on the liquid (Ghoshdastidar 2012a). The equivalent cooling process leading to the non-formation of liquid is called *supersaturation*. A supersaturated vapour refers to the metastable vapour state (Ghoshdastidar 2012a). The metastable states lie along an extrapolation of AB to B' (superheated liquid) and DC to C' (supersaturated vapour).

2.1.1 Molecular diffusion and evaporation

Vapour and liquid phases can coexist in unstable equilibrium, where the liquid and vapour pressures in close proximity to the interface are no longer equal (Ghoshdastidar 2012a). Since the vapour pressure is greater than liquid pressure, the number of molecules

making contact and being absorbed by the interface by the vapour phase is also greater. Therefore, in order to maintain equilibrium, the number of molecules emitted through the interface from the liquid phase must increase correspondingly. Ghoshdastidar concluded that some amount of superheating is required in order for vapour bubbles to form.

Consider a partially saturated soil specimen in the process of drying in an oven. The air voids in the soil serve as nucleation sites for the formation of vapour bubbles. When the sample is subjected to an input of heat, the gas or vapour residue expand until a critical size corresponding to the size of a viable bubble is reached (Ghoshdastidar 2012a). A vapour bubble can then develop further as a result of superheating of the water, until the point where buoyancy and dynamic forces become larger than the adhesion (such as forces of surface tension). This is followed by the bubble lifting off, leaving a small remnant of gas or vapour in the cavity. Subsequently, this remnant of vapour will be cooled by the cold water coming from the interior of the soil specimen. This transported water will then be heated by the addition of heat from the surrounding solid soil particles. This results in the formation of a new nucleus for the growth of a vapour bubble. This process will continue until all moisture is removed from the sample during the oven-drying process.

2.1.2 Relative humidity in unsaturated soil mechanics

Relative humidity may be described as the state of thermodynamic equilibrium between air and water (Lu & Likos 2004b). It is the amount of water vapour present in the air expressed as a percentage of the amount of water vapour needed for complete saturation at the same temperature. This ratio is the same as the ratio of vapour pressure in equilibrium with a solution and the saturated vapour pressure in equilibrium with free water. Relative humidity in the atmosphere and in the pores of unsaturated soil has the potential to vary significantly and as a result, phase changes commonly occur in the form of evaporation and condensation. Condensation occurs in the form of dew when the relative humidity of the pore air reaches 100%. Dew formation refers to a phase change from vapour to liquid.

Should the temperature of an environment drop, but the vapour pressure remains constant, a state of 100% relative humidity will occur when the temperature reaches a critical point known as the dew point temperature.

Figure 3 illustrates the manner in which the dew point temperature is reached once the cooling path intersects the saturated vapour pressure curve. The air void in an unsaturated soil is shown at a certain initial condition (point A) by temperature T_0 and vapour pressure u_{vA} . The cooling path is presented by a horizontal route from point A to point B.

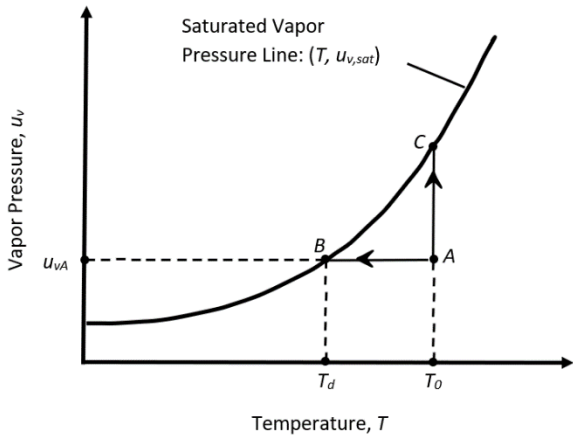


Figure 2. Graphical determination of the dew point temperature (Lu & Likos 2004b)

The dew point temperature, T_d , may be calculated from Equation 1:

$$T_d = \frac{36 \ln(u_v) - 4700}{\ln(u_v) - 16.78} \quad (1)$$

where u_v is the vapour pressure in kilopascals.

2.1.3 Effect of relative humidity on condensation

Condensation occurs when the temperature of a vapour is reduced below its saturation temperature. Two forms of condensation may occur, depending on the condition of the surface.

Figure 3a depicts film condensation, whose characteristic surface condition is that of a clean, uncontaminated surface. A liquid film covers the entire condensing surface and, thereafter, flows continuously from the surface due to the action of gravity.

Figure 3b depicts dropwise condensation (Ghoshdastidar 2012a).

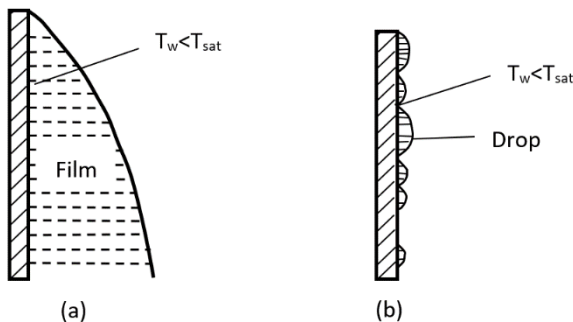


Figure 3. a) Film condensation, b) Dropwise condensation (Ghoshdastidar 2012a)

However, if the surface inhibits wetting, distinct droplets of condensate will form on the surface that has been cooled below the vapour saturation temperature.

Dropwise condensation was applicable for this study. The rate of condensation is governed by external factors, such as the relative humidity of the environment in which the samples were allowed to cool.

Development of droplets occurs in three stages (Castillo et al. 2015):

Initially, a uniform layer of small droplets grows by direct condensation on the interfaces of the droplets.

As the droplets increase in size, they coalesce and the distance between adjacent droplets consequently increases with time.

Small droplets grow in the exposed space between the coalesced droplets, thereby increasing the average surface coverage to a constant value. The droplets slide off the surface once a critical size is reached, allowing for new droplets to develop in the cleared off spaces.

Castillo et al. performed experiments to analyse the effects of relative humidity on dropwise condensation on a vertically oriented, hydrophobic surface. The controlled parameters were the relative humidity and surface sub-cooling temperature. The distribution and growth of water droplets were monitored across the surface at relative humidity of 45%, 50%, 55% and 70% at a constant surface sub-cooling temperature of 15 °C below the ambient temperature of 20 °C. Figure 4 and Figure 5 display diagrammatic results of the experiments conducted by Castillo et al.

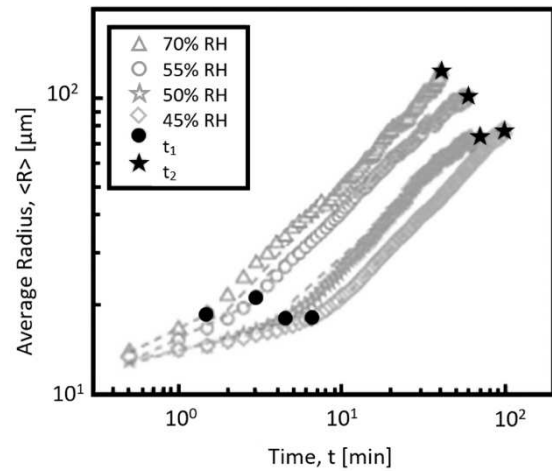


Figure 4. Average radius versus time at $\Delta T_{sub} = 15^\circ C$ and 45 %, 50 %, 55 % and 70 % relative humidity (Castillo et al. 2015)

It was found that the droplet growth dynamics exhibited a strong dependency on relative humidity in the early stages of development during which there was a substantial population of small droplets on the surface. It was also noted that single droplet growth dominated over coalescence effects in the early stages. It was found that, at later stages of droplet growth, the dynamics of droplet development were not affected by relative humidity due to the dominance of coalescence effects.

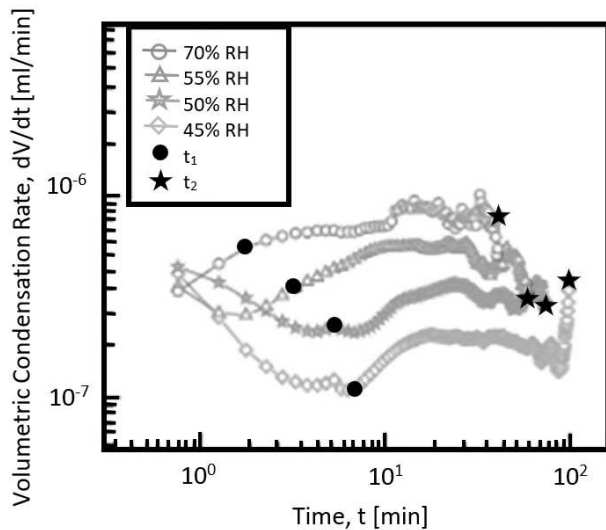


Figure 5. Volumetric rate of condensation on the surface as a function of time at $\Delta T_{\text{sub}} = 15$ °C and 45 %, 50 %, 55 % and 70 % relative humidity (Castillo et al. 2015)

Castillo et al. observed that the condensation rate decreases as droplets grow on the surface and coalesce. This trend only remained true until the gaps between the droplets became large enough to cause nucleation of new small droplets on the surface, thereby increasing the net condensation rate. This overall behaviour proved to be highly sensitive to the relative humidity. At lower ambient relative humidity, the sample surface is more easily deprived of water vapour, therefore prolonging the period of reduced condensation rates. On the contrary, at higher ambient relative humidity, the rate of growth of droplets on the surface is enhanced.

In the context of the gravimetric moisture content test, if the oven temperature is too low, the relative humidity in the oven would not be low enough, resulting in samples that are not completely dry. A lower equilibrium moisture content may be achieved by raising the temperature in the oven, consequently lowering the relative humidity of the ambient air within which the soil samples are drying. Ideally, the oven should be set to between 105 °C and 110 °C, thereby minimizing the variability due to ambient conditions. By meeting the minimum temperature range requirements, moisture content results from an oven in a humid, tropical climate are within 0.3 % - 0.4 % of that from an oven in a cold, dry climate (Reeb et al. 1999).

2.2 Adsorptive potential of soil

2.2.1 Soil Water Retention Curves

As soil goes through the process of drying (or wetting), the suction within the soil changes. The relationship between soil water content and suction is referred to as the Soil Water Retention Curve (SWRC), as shown in Figure 6.

Soil Water Retention Curves plot the volumetric water content, θ , or degree of saturation, S_r against suction.

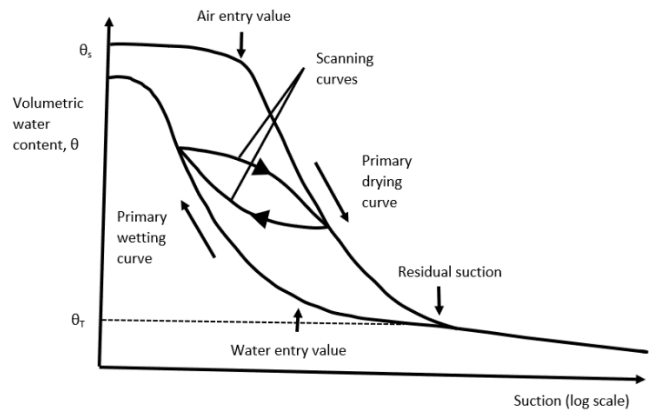


Figure 6. Typical Soil Water Retention Curve (Toll 2012)

Soil will follow the Primary Drying Curve if the soil were to start from a saturated state and is then subjected to drying. The largest pores within the soil will reach their limiting value of suction as it increases, which will cause the pores to desaturate. Air starts to enter the soil once the limiting value of suction, referred to as the Air Entry Value, is exceeded. Past the air entry value, the soil begins to lose moisture, as the pores begin to desaturate, where finer pores desaturate in succession to larger pores. At the Residual Suction value, which corresponds to the residual water content, the Soil Water Retention Curve will flatten. This is indicative of significantly smaller changes in volumetric moisture content which result from an increase in suction (Toll 2012).

Upon wetting from an oven-dried state, the soil will follow the *Primary Wetting Curve*. At the point known as the *Water Entry Value*, the water content increases significantly as additional pores begin to saturate. Once the suction value decreases to zero, the final volumetric water content may be lower than the initial saturated value, θ_s . Air bubbles being trapped within the soil may be the cause of such behaviour, or it possibly could be as a result of irrecoverable shrinkage of the soil.

Possible states within which the soil can exist are defined by an envelope created by the primary drying and wetting curves.

Representative Soil Water Retention Curves of clay, silt and sand are illustrated in Figure 7 (Lu & Likos 2004a), showing their general behaviour, which is highly dependent on soil type.

The surface adsorption regime in the high suction range for sandy soil is generally very limited due to the surface charge and specific surface properties of sand being relatively small (Lu & Likos 2004a). Capillarity is the governing suction mechanism over most of the unsaturated water content range, ending at an air-entry pressure that is relatively low.

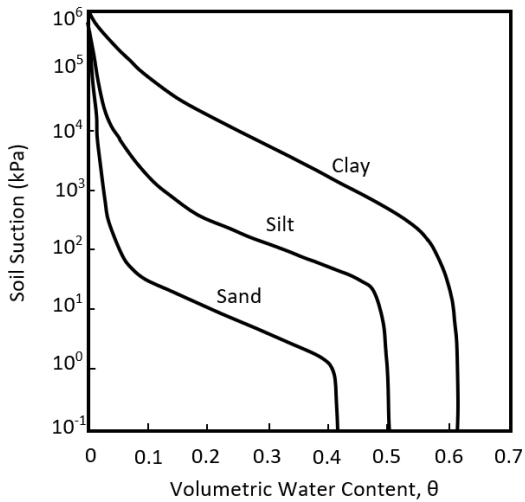


Figure 7. Representative Soil Water Retention Curves for sand, silt and clay (Lu & Likos 2004a)

The fairly large pore throats formed between the sand particles is what determines the air-entry pressure. The overall shape and slope of the capillary regime is predominantly determined by the pore size distribution of the soil. Flat retention curves are indicative of soils with a relatively narrow pore size distribution due to most of the pores being drained over a relatively narrow range of suction.

Due to the specific surface area of silt being considerably larger than that of sand, a silty soil may absorb a considerably larger amount of water under short-range adsorption mechanisms compared to a sandy soil. The relatively small pores in silt cause a high air-entry pressure.

Due to clay particles having charged surfaces and a significantly high specific surface area, it has the highest capacity for water adsorption under short-range surface interaction effects. Highly expansive clays are capable of adsorbing as much as 20% water by mass during the initial surface adsorption regime and may sustain exceedingly high suction over a wide range of water content. On the contrary, non-expansive clays adsorb less water in the high suction regime compared to that of an expansive clay (Lu & Likos 2004a).

3 EXPERIMENTAL PROCEDURE

The gravimetric moisture content test was studied in detail in order to closely observe the behaviour of the chosen soil samples during the various stages of the oven-drying test. The areas of interest were the drying stage, the cooling stage, and the temperature response of the samples during each stage of the test.

Thermistors were embedded within the soil samples to monitor the temperature change of the soil for the duration of the oven-drying and the cooling period. Three representative samples of each soil type

were tested at a time to provide a reference to each other. It was necessary to solder the thermistors to wires to form a connection to a data taker, which was used to log the temperature of the soil samples every minute. The samples had to be manually removed from the oven at 90-minute intervals and weighed on a digital scale with an accuracy of 0.01 grams. The scale was insulated with a measured sheet of polystyrene to eliminate the effect of the heat from the sample dishes affecting the readings on the digital scale. The cable providing the extended connection to the data taker had to be detachable from the petri dishes which contained the embedded thermistors. This was for the purposes of weighing the dishes with ease and accuracy, without the cable causing additional and varying weight to the petri dishes containing the samples. Therefore, further soldering of wire connections was done prior to being insulated with shrink tape and then secured with insulation tape to the glass petri dishes.

The resistance of each pair of wires was measured using a manometer, each pair registering approximately 10 Ω on average. This resistance was to be subtracted from the total resistance logged by the data taker, and then converted to a temperature reading using Equation (4):

$$T = \left(\frac{1}{0.00025374 \ln R + 0.0011958} \right) - 273 \quad (4)$$

where R (Ω) is the total resistance logged by the data taker minus 10 Ω ; and T is the temperature ($^{\circ}\text{C}$).

The D80 Data Taker was utilised together with a host laptop to monitor and retrieve the readings to be used for the soil temperature analysis.

The effect of the cooling stage was tested in 3 different environments. Each sample was allowed to cool to room temperature in the switched-off oven, in a desiccator and in the laboratory environment. The desiccator's lid was placed onto the base and then sealed with insulation tape to seal any gaps caused by the wires (which remained connected to the data taker until the test was concluded) protruding between the lid and the base. The temperature of the laboratory environment was also logged for the duration of each test, by attaching a thermistor to the wall of the room.

4 RESULTS

Table 1 and Table 2 summarise and compare the test results, in terms of percentage moisture gained, and the differences in moisture content (%) at the 2 stages under consideration, namely, immediately after the samples were removed from the oven and after the samples were allowed to cool to room temperature in their respective environments.

Table 1. Summary of the gravimetric moisture content tests performed on ± 30 g soil samples

Soil sample	Moisture adsorbed during cooling	Difference in moisture content
	%	%
Sand A	0.10	0.13
Sand B	0.10	0.12
Sand C	0.03	0.04
Silt A	0.10	0.12
Silt B	0.10	0.12
Silt C	0.10	0.12
Clay A	0.81	1.28
Clay B	1.44	2.23
Clay C	1.23	1.90

Table 2. Summary of the gravimetric moisture content tests performed on ± 70 g soil samples

Soil sample	Moisture adsorbed during cooling	Difference in moisture content
	%	%
Sand A	0.10	0.12
Sand B	0.10	0.13
Sand C	0.21	0.26
Silt D	0.12	0.14
Silt E	0.02	0.02
Silt F	0.08	0.10
Clay G	0.91	1.42
Clay H	1.00	1.61
Clay I	1.03	1.63

The samples which were allowed to cool in the desiccator and in the laboratory were observed to have reached room temperature within an hour after being removed from the oven, and at similar rates compared to the samples which cooled in the switched-off oven with the door left slightly ajar. However, the sample in the desiccator cooled down at a slightly slower rate than the sample in the laboratory, but always remained ± 3 °C above room temperature. The samples which cooled in the switched-off oven with the door left slightly ajar took approximately 12 hours to reach room temperature.

The ± 70 g sand and silt samples dried within 1 hour of being in the oven, whereas the clay took between 2 and 3 hours to dry.

5 CONCLUSIONS AND RECOMMENDATIONS

The clay gained the most mass (approximately 1 %) during the cooling process, whereas the sand and silt samples adsorbed approximately 0.1 % moisture, with no clear distinction between the two soil types.

Overall, the three soils responded in similar ways to the application of heat, as well as when they were removed from the source of heat.

Although it is customary to wait for soil samples to cool to room temperature before recording its dry mass, it was proven that it is not necessary, and that doing so gives a false dry mass, thereby resulting in a calculated moisture content which is lower than the

true moisture content. Irrespective of the soil type being tested, or the measures taken to prevent the material from adsorbing moisture whilst it is cooling down to room temperature (e.g. a desiccator), it was found that soil specimens will gain mass in the form of moisture during cooling.

It may be concluded that 3 hours in a 110 °C oven is sufficient for drying ± 70 g of moist clayey material. Furthermore, 1 hour of drying time in an oven is sufficient for ± 70 g of moist material consisting primarily of sands and silts.

The following recommendations are presented: The room in which the test is being conducted should be temperature controlled. The relative humidity of the various testing environments should be monitored. The thermistors must be embedded properly within the soil samples.

6 ACKNOWLEDGEMENTS

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