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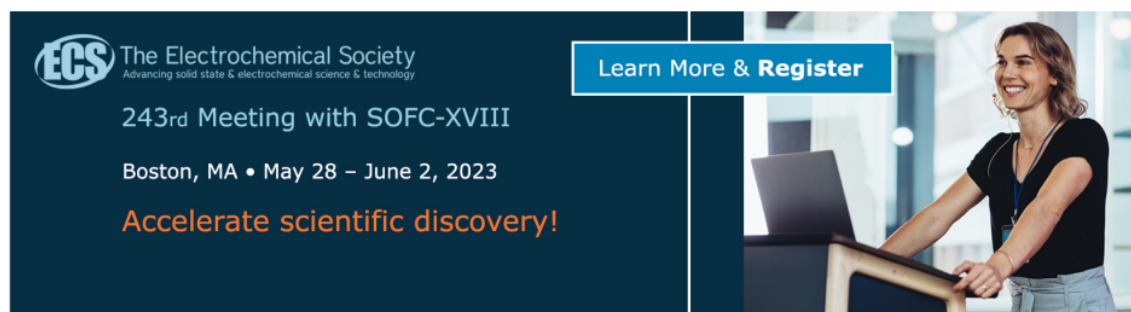
## The application of vapour equilibrium technique to control suction to study the shrinkage and water retention of compacted Claystone-Bentonite mixtures

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# The application of vapour equilibrium technique to control suction to study the shrinkage and water retention of compacted Claystone-Bentonite mixtures

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**Abstract.** Understanding soil shrinkage and retaining water is essential for learning more about the possibility of cracking of liner. Moreover, the factors that influence it are also important to know to improve the material tested as hazardous waste liners. The vapour equilibrium technique is widely used to control suction of compacted soils experiencing drying-wetting phenomena. It is considered to be inexpensive, simple, and has the ability to adequately control the suction applied to soil samples. This paper, therefore, describes its application in studying the shrinkage and water retention in compacted claystone-bentonite mixtures. This involved using five saturated salt solutions including potassium sulphate ( $K_2SO_4$ ), potassium chloride (KCl), sodium chloride (NaCl), potassium carbonate ( $K_2CO_3$ ), and magnesium chloride ( $MgCl_2 \cdot 6H_2O$ ). The sample was allowed to be in equilibrium with the relative humidity salt solution and a calliper was used to measure the dimensions every day up to when this was achieved. The results showed the bentonite in the mixture affects the amount of shrinkage and water retention while the sample's initial moisture content was also found to be very influential on the magnitude of the primary and residual shrinkage. Moreover, the sample's ability to hold water was almost the same without differentiating the initial water content at a total suction of more than 41084.91 kPa.

## 1. Introduction

Claystone is a material usually avoided and discarded due to its unfavourable behaviour and the existence of its layers considered to be a problem in construction work have been reported in some publications [1–5]. The use of this material has, however, been widely recommended, especially as a barrier to hazardous waste and this requires mixing it with bentonite to improve its hydro-mechanical properties [6–9]. Zang and Kröhn [9] reported the superiority of the claystone-bentonite mixture's hydro-mechanical properties compared to compacted pure bentonite and bentonite-sand mixture which included higher density, stiffness, and stability, and very low permeability as well as its ability to release gas at lower pressure [9]. The characteristic absorption of clay minerals in the claystone/clay shale [10] also adds to its advantage compared to the use of sand to mix the clay liners. It is even more beneficial when the claystone is obtained at the project site due to the consideration for the use of local material by several researchers in recent times [11–13].

The two mixtures are made from clay and one of the characteristics considered is their high shrinkage which was divided by Cornelis et al. [14] into four parts which are structural, normal, residual, and zero.



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Meanwhile, Mishra et al. [15] also classified shrinkage in the compacted clay and bentonite-sand mixtures into initial, primary, and residual stages. It is important to note that both structural and initial shrinkages are influenced by the bimodal pore distribution in the form of macro and micropores which were reported by Arifin [16] to be strongly influenced by the initial moisture content of the samples. Generally, the initial and residual shrinkages are not too significant and this means there is a need to study the primary shrinkage in claystone and its mixture with bentonite.

Mishra et al. [15] conducted used a drying process to obtain a shrinkage curve and this involved allowing the sample to be air-dried for two months. It is, however, difficult to implement this method in areas with high relative humidity (*RH*) such as in Indonesia due to the possible absorption of water from the air by the sample when the *RH* rises. Therefore, a better method is to control the *RH* around the sample using the Vapor Equilibrium Technique (VET) to ensure the sample is balanced at a lower *RH*. This approach has also been widely applied to test water retention behaviour or water characteristic curves of soils, especially clay apart from its application in shrinkage behaviour [16–18]. It has also been applied in testing the hydro-mechanical behaviour of unsaturated compacted clay to control the suction applied to the sample [7], [16], [19–21]. VET, however, generally requires using a saturated salt solution with some researchers observed to have used molal salt solutions such as sodium chloride [16], [18]. The sample also needs to be in equilibrium with the relative humidity above the solution [7], [16–20], [22] and this means temperature needs to be controlled due to its strong influence on the *RH*. The method is also usually combined with the axis translation technique at low suction [16], [21] and the changes in the temperature gradient affect the *RH* with further causes a variation in the total suction being applied [16], [23], [24]. This paper, therefore, discusses the shrinkage behaviour and water retention of compacted claystone-bentonite mixtures using VET.

## 2. Methods and Procedures

### 2.1. Materials Used

The materials used in this study include claystone and bentonite with the claystone obtained from the Banjarbaru city area while the bentonite is available in the market. The Engineering Properties of the two materials are summarized in Table 1 and the claystone observed to have a liquid limit (LL) of 50.76% and a plasticity index (PI) of 20.95% while and these are twice as much as those obtained from Belencito formation in eastern Andes mountain, Columbia, as reported by Espitia et al. [1].

### 2.2. Sample preparation

The Standard Proctor compaction test (ASTM D698-07) was conducted to determine the relationship between water content and claystone density used as a primary material and the optimum moisture content and maximum density were found to be 15% and 16kN/cm<sup>3</sup>, respectively. All the samples were prepared using these values and those with lower and higher water contents at 10% and 15% were used to study the effect of water content.

Claystone was mixed with 5%, 10%, 15%, and 20% bentonite on dry weight basis while water was added to reach the pre-selected level of 10%, 15%, and 20% and value. Meanwhile, the water contained in each sample was also calculated before mixing. The mixed sample was left in equilibrium for one day and statically compacted to reach a density of 16 kN/m<sup>3</sup> and the dimension used was, however, thinner than usual as observed with the 10 mm height and 64.2 mm diameter.

There are two possible conditions in the clay liner or cover layer while on the field and the first involves the experience of an increase in water content due to rain in the sample which means the initial process is wetting. It is, however, possible to also experience immediate drying due to the exposure to air, thereby, leading to shrinkage. The sample was simulated in this study and this led to the second condition with the vapour equilibrium technique (VET) method.

The total suction of the initial sample was determined before being tested by measuring the *RH* using a sensor corrected for accuracy using the chilled-mirror hygrometer technique which has been confirmed to be the most reliable tool to measure *RH* [16], [23–25]. Meanwhile, the total suction sample was

calculated using Equation 1 while the initial conditions of the claystone and bentonite mixture are presented in Table 2.

**Table 1.** Engineering properties of materials used

Properties		Claystone	Bentonite
Specific gravity		2.60	2.71
Water content	%	2.75	14.17
Grain size distribution:			
Gravel (> 2 mm)	%	0.02	0.00
Coarse sand (0.6-2.0 mm)	%	0.08	0.00
Medium sand (0.2-0.6 mm)	%	0.10	0.00
Fine sand (0.05-0.2 mm)	%	4.30	1.39
Silt and Clay (0.002-0.05)	%	43.94	8.33
Clay (<0.002mm)	%	51.55	90.28
Atterberg Limits:			
Liquid limit	%	50.76	351.71
Plastic limit	%	20.95	44.68
Shrinkage limit	%	9.74	41.89
Plasticity Index	%	29.81	307.03

$$s_t = -\frac{\rho_w RT}{M_w} \ln\left(\frac{RH}{100}\right) \quad (1)$$

where  $s$  is total suction,  $\rho_w$  is the volumetric weight of water,  $R$  is a universal gas constant which is 8.31432 J/mol.K,  $T$  is the temperature in Kelvin, and  $M_w$  is molecule mass of water vapour which is 18.016 g/mol.

### 2.3. Vapour Equilibrium Technique

The five saturated salt solutions used in this study include potassium sulfate ( $K_2SO_4$ ), potassium chloride (KCl), sodium chloride (NaCl), potassium carbonate ( $K_2CO_3$ ), and magnesium chloride ( $MgCl_2 \cdot 6H_2O$ ). The  $RH$  or total suction of each solution calculated using Equation 1 is summarized in Table 3. The solution was later placed in an airtight glass equipped with rubber seals and clamp lids as shown in Figure 1.

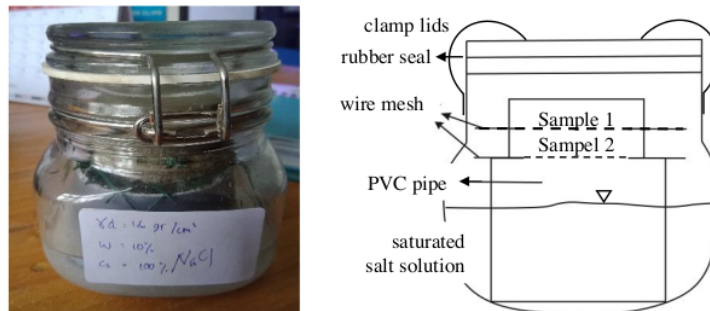
**Table 2.** Initial conditions of claystone-bentonite mixtures

Sample code	Bentonite content (%)	Dry density ( $kN/m^3$ )	Water content (%)	Total suction (kPa)
100CS-1	0	16	10	2663.56
100CS-2	0	16	15	2237.56
100CS-3	0	16	20	1816.35
95CS-5B-1	5	16	10	4089.92
95CS-5B-2	5	16	15	2664.36
95CS-5B-3	5	16	20	2104.87
90CS-10B-1	10	16	10	5231.79
90CS-10B-2	10	16	15	4233.12
90CS-10B-3	10	16	20	3236.72

**Table 3.**  $RH$  and total suction of saturated salt solution

Solution	$K_2SO_4$	KCl	NaCl	$K_2CO_3$	$MgCl_2 \cdot 6H_2O$
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$RH$ (%)	95.2	81.5	74.3	46	34.2
$T$ (°C)	27.7	28.1	27.8	28.2	28.0
Total suction (kPa)	6801.21	28318.56	41084.91	107528.77	148484.33



**Figure 1.** (a) Photo of an airtight glass used, and (b) sketch of components in the glass

The VET was generally conducted using a desiccator [17], [22] and, apart from the larger volume, the samples placed in the desiccator influenced each other and this slowed down the time to reach equilibrium [16].

The compacted samples were placed into a 615 cm<sup>3</sup> glass containing a saturated salt solution, as shown in Figure 1, and allowed to stay until they attain equilibrium. The weight as well as the dimensions including the height and diameter of the samples were measured every day using Vernier callipers through the direct measurement method [26]. They were placed in the oven for 24 hours to obtain the dry weight and two samples were tested to represent the conditions presented in Table 1. Moreover, smaller glasses were used to ensure each is filled with different solutions and due to its ability to allow concurrent testing of identical samples to reduce the time needed for the test.

### 3. Results and Discussion

#### 3.1. Time to reach equilibrium

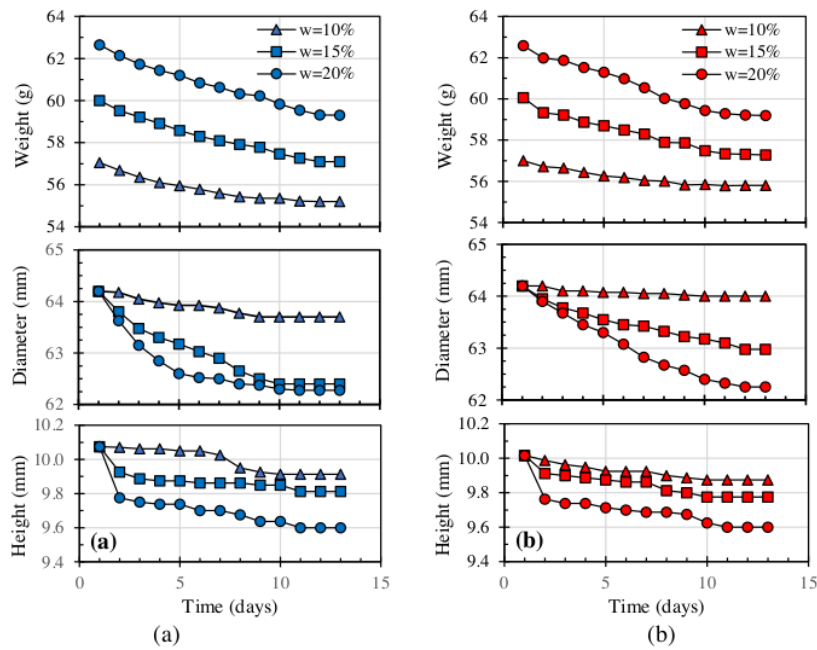
The samples were considered to have reached equilibrium condition when no change was recorded in their weight and dimensions. Figure 2 shows a typical change in the weight, diameter, and height of the samples 95CS-5B in (a) and 90CS-10B in (b) for a glass containing a saturated K<sub>2</sub>SO<sub>4</sub> solution with a total suction of 6848.64 kPa. This means the weight decreases by time followed by the diameter and height. Meanwhile, the weight at 95CS-5B-3 and 90CS-10B-3 with an initial water content of 20% was constant on the 11th and 12th day while the value was also observed to be constant on the 9th day for 95CS-5B-1 and 90CS-10B-1 with an initial water content of 10%. Furthermore, the diameter and height measured for the 95CS-5B-1 and 90CS-10B samples -1 were relatively constant at the 9th and 10th days while it was 11th and 12th day for 95CS-5B-3 and 90CS-10B-3. These results showed the equilibrium time was affected by the initial water content such that a longer time is required to achieve equilibrium at a higher value for the initial moisture content.

Figure 3 shows the change in the weight by time at higher total suction of 39055.36 kPa for 95CS-5B in (a) and 90CS-10B in (b). The weights of the 95CS-5B-1, 95CS-5B-2, and 95CS-5B-3 were observed to be constant at the 7th, 8th, and 9th day while those for 90CS-10B-1 to 90CS-10B-3 were on the 8th, 9th, and 10th day. This means the time to reach equilibrium was relatively faster at high suction than lower suction.

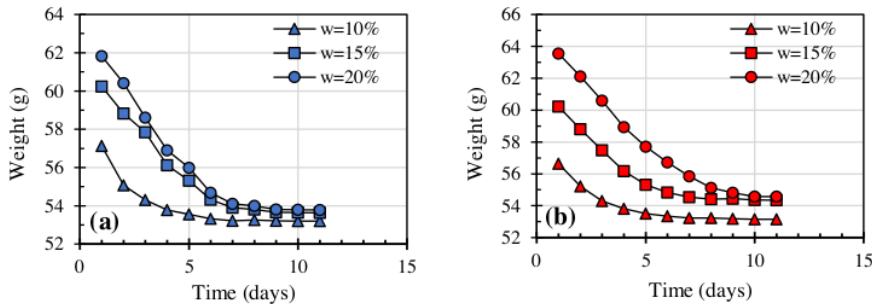
Figure 3 also shows the sample's weight under equilibrium conditions was almost the same without differentiating the initial moisture content. This result is typical for samples which allowed equilibrium at a total suction of more than 41084.91 kPa.

### 3.2. Shrinkage behaviour of compacted claystone-bentonite mixtures

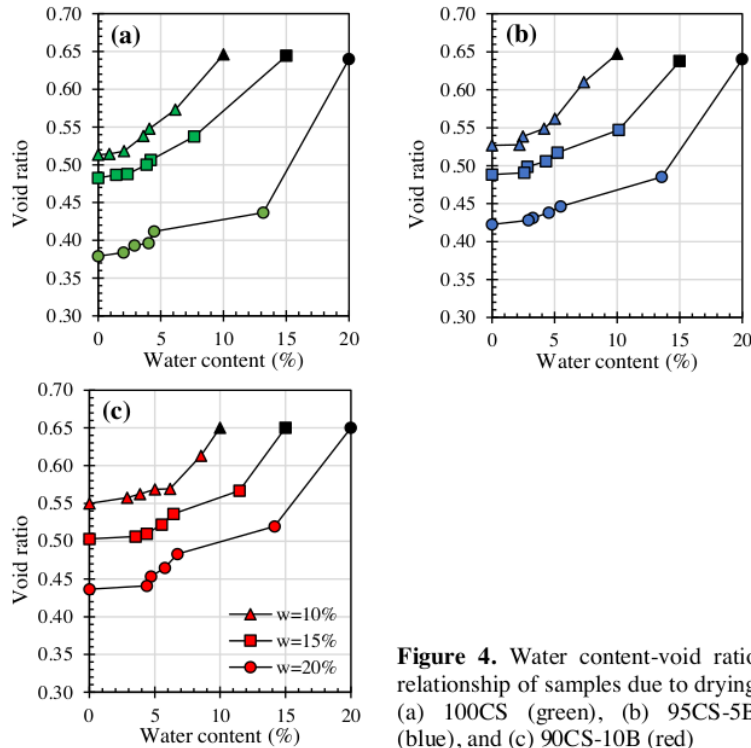
The soil shrinkage behaviour is usually depicted in the relationship between void ratio and water content [14–16, 26] and this is shown for claystone and bentonite mixture in Figure 4. The data is consistently displayed using different shape markers and colours as observed in the triangles, squares, and circles applied for the initial moisture contents at 10%, 15%, and 20% respectively. Moreover, green, blue and red colours were used to distinguish the percentage of bentonite at 100% claystone, 5% bentonite, and 10% bentonite respectively. The sample shrinkage curve is shown in the figure to only have two distinct stages which are primary and residual with no initial shrinkage obtained for all the samples tested. Generally, a sample with three stages usually has a high initial moisture content or become saturated in the swelling process as reported by Mirsha et al. [15]. A two-stage shrinkage sample was, however, found for the bentonite-sand mixture at optimum moisture content and maximum density conditions.



**Figure 2.** Typical change of weight, diameter, and height of samples at total suction of 6848.64 kPa at (a) 95CS-5B, and (b) 90CS-10B



**Figure 3.** Weight by time curves at total suction of 41084.91kPa at (a) 95CS-5B, and (b) 90CS-10B



**Figure 4.** Water content-void ratio relationship of samples due to drying (a) 100CS (green), (b) 95CS-5B (blue), and (c) 90CS-10B (red)

The water content at which the primary shrinkage ended or the residual shrinkage started ( $w_r$ ), as well as the magnitude of each, are summarized in Table 4. Moreover, the  $w_r$ ,  $PS$ , and  $RS$  are used to represent the water content at residual shrinkage, primary shrinkage, and residual shrinkage, respectively. The information on Table 4 shows samples 100CS-1, 2, and 3 with an initial water content of 10%, 15%, and 20% have a  $w_r$  of 2,068%, 4,196%, and 4,47% respectively. The content at residual shrinkage was observed to have increased with the initial value and this was also the same for the claystone samples with bentonite such as 95CS-5B and 90CS-10B.

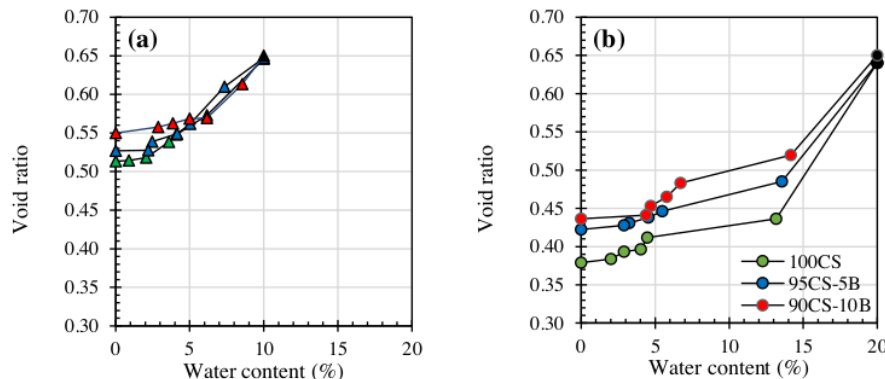
Table 4 also indicates the influence of the initial moisture content through the  $PS$  and  $RS$ . The 100CS-1, 2, and 3 experienced primary shrinkage of 19.79%, 21.37%, and 35.65%, and residual shrinkage of 0.9%, 4.74%, and 7.99%, respectively. At the same density, the  $PS$  and  $RS$  were recorded to have increased with the initial water content and the same trend was also obtained for claystone-bentonite mixtures.

**Table 4.** Shrinkage properties of claystone-bentonite mixtures

	100CS			95CS-5B			90CS-10B		
	1	2	3	1	2	3	1	2	3
$w_r$ (%)	2.068	4.196	4.478	2.827	4.313	4.542	6.160	6.436	6.725
$PS$ (%)	19.79	21.37	35.65	16.79	20.69	32.92	12.40	17.49	25.67
$RS$ (%)	0.90	4.74	7.99	2.19	3.40	3.79	3.39	6.18	9.70



A comparison was made on the samples to determine the effect of bentonite at the same initial moisture content. The  $w_r$  of 100CS-1, 95CS-5B-1, and 90CS-10B-1 at initial content of 10% were 2,068%, 2,827%, and 6.16 while the primary shrinkage was 19.79%, 16.79%, and 12.40% and the residual shrinkage was 0.9%, 2.19%, and 3.39% respectively. These data showed the  $w_r$  and  $PS$  increased while  $RS$  decreased with an increase in the bentonite percentage. The samples with 10% and 20% initial water content also showed the change in void ratio as shown in Figures 5(a) and (b). The figures also show the effect of bentonite content on the shrinkage behaviour of the samples.



**Figure 5.** Water content-void ratio relationship of samples due to drying (a)  $w=10\%$ , and (b)  $w=20\%$

### 3.3. Water retention of compacted claystone and bentonite mixtures

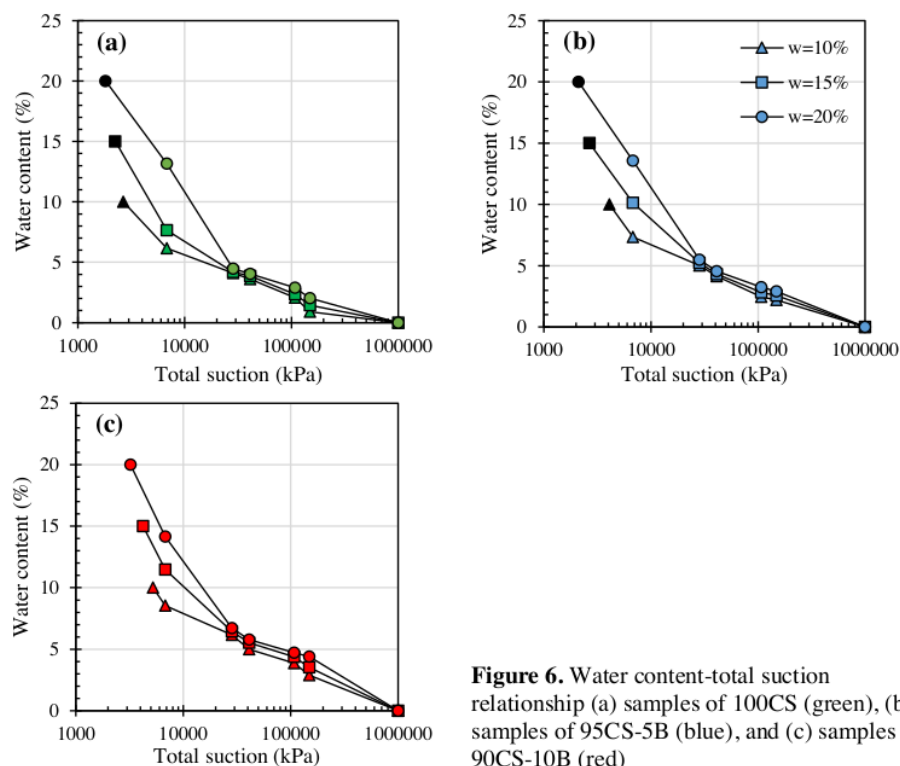
The water retention curves of claystone and bentonite mixtures are indicated in the relationship between water content and total suction as shown in Figure 6. In consistency with the previous figures, the 100CS, 95CS-5B, and 90CS-10B are displayed in green, blue, and red respectively while the initial condition is in black. Moreover, those with an initial moisture content of 10%, 15%, and 20% are presented in triangle, square, and circle respectively. Figure 6(a), therefore, shows the water content of 100CS-1, 2, and 3 was initially 20%, 15%, and 10% and later decreased as the suction was increasing. Meanwhile, the amount of water content retained by the sample is almost the same at the total suction of 41084.91 kPa and the same trend is observed in other samples as shown in Figures 6(b) and (c). Arifin [16] found a similar result for bentonite and bentonite-sand mixtures. Furthermore, dry samples from saturated conditions have almost the same water content at suction higher than the air entry value (AEV) even though they are compacted at different water levels.

The difference in water retention curve at low total suction is due to the pore size distribution from the samples compacted at lower and higher moisture content than the required optimum. At the lower value, the macropores became dominant while it was the micropores at high values [16]. Moreover, water does not only fill the intra-particles or micropore but also the inter-particles or macropores at low total suction and also affected by the fabric. However, at the high total suction of 41084.91 kPa recorded in this study, the water was in the intraparticle or micropore and this reduced the influence of the fabric due to the adsorption of the water with hydration force by the clay surface.

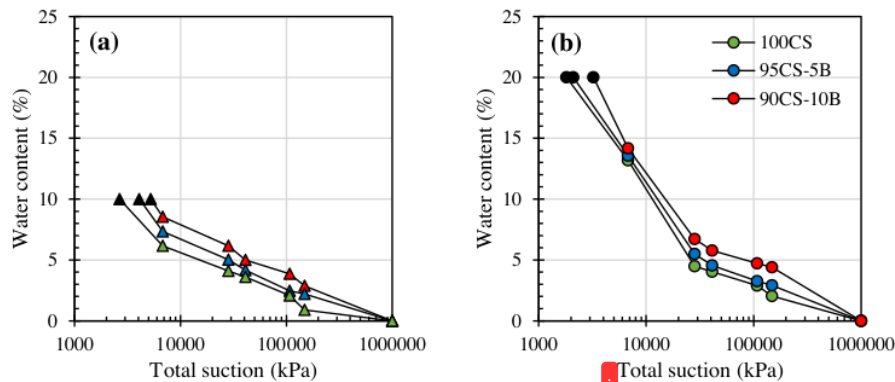
Samples with the same conditions are plotted in a graph as shown in Figure 7 to determine the effect of the bentonite with those at 10% presented in Figure 7(a) and 20% in (b). The curve of the sample containing 5% bentonite is observed to be above the curve for the sample without bentonite or 100CS while the highest curve was recorded with 10% content. Moreover, the water content was found to have increased with the bentonite content at the same total suction and similar behaviour is present in Figure 7(b) showing samples with higher bentonite content have the ability to absorb more water.

#### 4. Conclusions

The results of study on the shrinkage properties and water retention of compacted claystone and bentonite mixtures have been presented. The sample shrinkage curve has only two distinct stages which are the primary and residual shrinkage. Water content at residual shrinkage was observed to have increased with the initial water content. The amount of primary and residual shrinkages also increased with the sample's initial moisture content at the same density. The Primary shrinkage also increased with the bentonite content while the residual shrinkage was observed to have decreased. The water retention curve was strongly influenced by the initial moisture content at a total suction lower than 41084.91 kPa while the ability to hold water was almost the same at higher values. The results shows that the addition of bentonite to the claystone material increases the ability of the sample to hold water and a higher bentonite content holds more water content at the same total suction.



**Figure 6.** Water content-total suction relationship (a) samples of 100CS (green), (b) samples of 95CS-5B (blue), and (c) samples of 90CS-10B (red)



**Figure 7.** Bentonite effect on water retention behaviour of samples with the initial water content of (a) 10% and (b) 20%.

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