ABSTRACT

Water retention curve (WRC) is a nonlinear function that describes the relationship between the matric suction ($\psi$) and water content ($\theta$) of a porous material evaluated under unsaturated conditions. WRC contains fundamental information about hydro-mechanical behavior of unsaturated porous materials of different composition. In this context, this paper presents the laboratory results and procedures used to determine the WRC of a soil-cement composite material used in road pavement construction. With this purpose, water content and matric suction measurements in a soil-cement composite with 3.5% of Portland cement by using pressure cells were developed. Fitting values of the WRC were calculated according to the theoretical four-parameter van Genuchten model. Hence, the best fit parameters of the van Genuchten water retention curve were estimated using a nonlinear fitting approach based in minimizing the sum of squared-error in nonlinear functions. Results show that the water retention curve and its parameters according to the van Genuchten model determined in this study can be used as reference values of the unsaturated parameters of soil-cement composite material used in road pavement construction. Moreover, soil-cement water retention curve shown in this study corresponds to one of the first results published in the literature on this composite material.

Keywords: Soil-cement composite, matric suction, water content, nonlinear analysis.

RESUMEN

La curva de retención de agua es una función no lineal que describe la relación entre la succión matricial y el contenido de agua de un material poroso evaluado bajo condiciones no saturadas. La curva de retención contiene información fundamental sobre el comportamiento hidromecánico de los materiales porosos. Este trabajo presenta los resultados de laboratorio y los procedimientos utilizados para determinar la curva de retención de un material compuesto de suelo-cemento utilizado en la construcción de pavimentos de carreteras. Con este propósito se han desarrollado mediciones del contenido de agua y la succión matricial utilizando células de presión en un compuesto de suelo-cemento con un 3,5% de cemento Portland. Los valores de ajuste de la curva de retención fueron calculados de acuerdo con el modelo teórico de cuatro parámetros de Van Genuchten. Los mejores parámetros de ajuste de la curva de Van Genuchten fueron estimados utilizando un enfoque de ajuste no lineal basado en la minimización de la suma de errores al cuadrado. Los resultados muestran que la curva de retención y sus parámetros, de acuerdo con el modelo de Van Genuchten, se pueden utilizar como valores de referencia de los parámetros no saturados del material compuesto suelo-cemento. Por otra parte, la curva de retención del suelo-cemento que se muestra en este estudio corresponde a uno de los primeros resultados publicados en la bibliografía sobre este tipo de material.

Palabras clave: Compuesto suelo-cemento, succión matricial, contenido de agua, análisis no lineal.
INTRODUCTION

Study of water flow through saturated and unsaturated porous materials is a topic of great interest in materials engineering [1]. In porous materials under unsaturated conditions the water flow through them is primarily by capillarity or suction, while under saturation gravity it is the primary force causing water movement. Suction is a particular physical property of unsaturated porous materials, such as compacted road soils, cementitious materials, geocomposites, some composite materials, etc. This property describes the potential with which a material adsorbs and retains water in the interior of the pores. Therefore, a water retention curve (WRC) describes the relationship between the matric suction and water content or degree of saturation of a specific porous material [2-4].

In this way, total suction ($\psi_t$) is defined as the sum of two main components: matric suction ($\psi$) and osmotic suction ($\psi_o$). However, in most problems concerning porous materials under unsaturated conditions, e.g., road compacted soils [1], the changes in the total suction correspond to changes in matric suction, not being necessary to consider the osmotic component. Matric suction or suction ($\psi$) in unsaturated porous material is defined as the difference between the pore-air pressure and the pore-water pressure ($u_a-u_w$). However, usually in unsaturated materials the pore-air pressure is atmospheric ($u_a=0$) being pore-water pressure negative with respect to the atmospheric pressure. In addition, water content can be expressed as gravimetric water content ($w$), volumetric water content ($\theta$), or degree of saturation ($S$). However, in civil engineering applications considering geomaterials [5-6] results are commonly represented as volumetric water content. As it can be seen in Figure 1, WRC is a nonlinear graphical relationship characterized by singular points (i.e., associated variables) where the slope of the curve changes abruptly. These points are called the air-entry value (AEV) and the residual water content value ($\theta_r$). The AEV corresponds to the value of suction (or matric suction) at which the air begins to enter into the largest pores of the material. The residual water content value is the one that requires large changes in the suction to cause a further reduction of water content. In addition, another characteristic value of the WRC is the saturated water content value ($\theta_s$) which represents the point where all the available spaces in the pores of the porous material matrix are filled with water and the suction at this point is zero. Nevertheless, the general form of the water retention curve can also be influenced by other different parameters associated mainly at porous material type [7]. Additionally, the WRC form (see Figure 1) can be divided into three zones according to the state of water retention, such as [3]: the capillary zone, the continuous capillary zone, and the residual zone; where the water content or liquid inside the pores is almost zero.

![Figure 1. Nonlinear water retention curve form and its associated variables [1].](image-url)
to model the behavior of unsaturated composite materials, for example by numerical simulation models, it is necessary that the relationships between matric suction and water content are described by continuous functions [11]. In this context, various researchers [12-13] have proposed different equations to describe the WRC knowing experimental data. However, theoretical four-parameter van Genuchten model [13] is the most widely used. This model has been used in this study because of its simplicity and its greater flexibility compared to other models, in this way, van Genuchten is defined as follows:

\[ \theta(\psi) = \theta_r + \left( \frac{\theta_s - \theta_r}{1 + (\alpha \psi)^n} \right)^m \]

Where, \( \theta \) is the volumetric water content in \([L^3 L^{-3}]\), \( \psi \) is the suction in \([L]\), \( \theta_r, \theta_s \) are the residual and saturated water contents in \([L^3 L^{-3}]\), \( \alpha \) is a curve fitting parameter related to air-entry pressure in \([L^{-1}]\), and \( n, m \) are dimensionless curve fitting parameters related to pore-size distribution, where the relationship, \( m=1-1/n \) is often assumed. It can be seen in Equation 1 that the water retention curve according to van Genuchten is nonlinear. Thus for the determination of the characteristic curve parameters, the use of a nonlinear fitting approach is a necessary step. With this aim, different nonlinear fitting methods have been implemented in several commercial codes such as RECT code [14] or SWRC-Fit program [15]. In this study, SWRC-Fit program was used because explicit input of the initial estimate is not required. This program shows a good agreement between the experimental results and fit values obtained when the actual initial parameters of the material are unknown [1,5]. Finally, this paper has been prepared with the objective of experimentally measure the water retention curve of soil-cement composite material used in road pavement construction. With this objective, water content and matric suction measurements on a soil-cement with 3.5% Portland cement by using pressure cells, have been developed.

MATERIALS AND METHODS

Materials
Soil-cement composite material used as subgrade improvement layer in road pavement construction has been analyzed in this study. A representation of the structure of this soil is shown in Figure 2. The soil was extracted from the construction of a road section located in the city of Santander, Northern Spain [1]. For this reason, identification and classification of the material was carried out according to the recommendations of the European standards. Soil-cement composite material consisted on a mix of silty-clay soil with a 3.5% Portland cement, not showing significant voids or fractures. According to Munsell scale classification its color is pale red and white identification number 2.5YR 7/6, see Figure 2. Finally, carbonate content into artificial soil was not significant.

Test samples preparation
Nine undisturbed samples of soil-cement were prepared in order to measure the matric suction of the material in laboratory conditions. With this purpose, undisturbed samples were extracted directly from the corresponding field sample, with unaltered structure and water content. The sample dimensions were 70 mm diameter and 20 mm height. Finally, each sample was kept in an airtight container until the sample was tested.

Physical and chemical characterization
Soil-cement was characterized by their specific gravity, Atterberg limits, maximum dry density and optimum water content of the modified Proctor compaction test, porosity of the soil and lastly the micro-porosity and total specific surface area using mercury intrusion porosimetry. In addition, size characteristic parameters and grain size distribution curves were obtained using sieving analysis. Moreover, the chemical properties and mineralogical composition of the soil were obtained using X-Ray
florescence and X-Ray diffraction. Finally, due to the reaction of the soil-cement in contact with water, the specific gravity of composite material has been determined using a gas pycnometer.

**Morphology of soil-cement**

The surface aspect of the soil-cement composite material was studied by using a Scanning Electron Microscope (JEOL JSM-6610/LV) available at the Center for Biomaterials and Nanotechnology from University of Bío-Bío.

**Matric suction measurement**

Matric suction on nine soil-cement samples was measured according to standard ASTM D3152-72 [16]. In this way, pressure cell apparatus with semi-permeable regenerated cellulose membranes have been used, see Figure 3. The development of the tests was as follows. Firstly, the sample was taken out of the airtight container and placed on the pervious part of the pressure cell (saturated cellulose membrane) located above a porous disk of coarse grains saturated in contact with water at atmospheric pressure. Saturation during the test was achieved through the water movement (water circuit) between the inlet and outlet valves during the test. Thus, when the whole set was closed, air was introduced into the cell at the tested pressure using a nitrogen bottle (N₂). The test pressure of the soil-cement samples ranged from 0.1 to 2000 kPa. Pressure was monitored using a gauge placed at the top of the pressure cell. Therefore, the air pressure in the soil-cement pores (in contact with the membrane) increased to the amount to which the air pressure rose in the cell, producing a moisture transfer (or water content) between the sample and the drainage system, until the water pressure in the soil-cement was equal to atmospheric pressure. When the equilibrium was reached (final moisture) the air pressure was equal to the soil-cement suction and this was equal to the matric suction. This procedure is known as the axis translation technique [10]. Additionally, the time necessary to achieve the moisture equilibrium is associated with the type of drainage system used, which in turn will display higher impedance to the passage of water as greater ranges of suction. For the tests carried out the average equilibrium time registered in the different tested samples was 330 hours, approximately. Therefore, when the equilibrium was reached, the cells were removed and the samples extracted to find their final moisture or equilibrium water content. The final gravimetric water content calculated was converted to volumetric water content (θ).

![Figure 3. Pressure cell apparatus to measure matric suction and based on the axis translation technique.](image)

**WRC according to van Genuchten model**

Volumetric water content (θ) and suction (ψ) values obtained by experimental procedures were used to estimate the WRC function and its four-parameters according to the van Genuchten model shown in equation (1). The best-fit parameters according to van Genuchten model were determined using the nonlinear fitting program SWRC-Fit [15]. Thus, quality of fit using the van Genuchten model and experimental values model were verified by calculating the sum of squared residual (SSR) values according to equation (2) given by:

$$SSR = \sum_{i=1}^{n} w_i (\theta_i - \hat{\theta}_i)^2$$

![Figure 4. Grain-size distribution curve.](image)
Where, according to this study, \( w_i \) is a dimensionless weighting factor and equal to 1 in this study, \( \theta_i \) is the volumetric water content measured experimentally for a given value of suction, and \( \hat{\theta}_i \) is the volumetric water content estimated by fitting van Genuchten model with a suction value equal to \( \theta_i \). According to the recommendations of Leong and Rahardjo [17] a SSR value less than \( 10^{-3} \) can be considered satisfactory for a good fit.

Table 1. Physical properties of soil-cement.

<table>
<thead>
<tr>
<th>Property</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity (kg/m(^3))</td>
<td>( G_s )</td>
<td>2,720</td>
</tr>
<tr>
<td>Liquid limit (%)</td>
<td>( w_L )</td>
<td>37</td>
</tr>
<tr>
<td>Plastic limit (%)</td>
<td>( w_p )</td>
<td>18.10</td>
</tr>
<tr>
<td>Plasticity index (%)</td>
<td>( PI )</td>
<td>18.90</td>
</tr>
<tr>
<td>Maximum dry density (kg/m(^3))</td>
<td>( \gamma_d )</td>
<td>2,040</td>
</tr>
<tr>
<td>Optimum water content (%)</td>
<td>( w_{opt} )</td>
<td>10</td>
</tr>
<tr>
<td>Porosity by ( G_s, \gamma_d )</td>
<td>( n )</td>
<td>25</td>
</tr>
<tr>
<td>Micro-porosity (%) Particles &lt;0.08 mm</td>
<td>( \mu n )</td>
<td>58.8</td>
</tr>
<tr>
<td>Total specific surface area (m(^2)/g) Particles &lt;0.08 mm</td>
<td>SSA</td>
<td>7.104</td>
</tr>
<tr>
<td>Effective particle size (mm)</td>
<td>( D_{10} )</td>
<td>–</td>
</tr>
<tr>
<td>Particle size (Pass. 30%) (mm)</td>
<td>( D_{30} )</td>
<td>0.08</td>
</tr>
<tr>
<td>Particle size (Pass. 60%) (mm)</td>
<td>( D_{50} )</td>
<td>4.16</td>
</tr>
</tbody>
</table>

RESULTS AND DISCUSSION

Soil-cement characterization

The results of the physical and chemical characterization of soil-cement composite material analyzed in this study are shown in Table 1 and Table 2. Additionally, in Figure 4 it can be seen the grain-size distribution curve of soil-cement material. From this Figure, it can be observed that the shape of the curve was unimodal, so the water retention curve would be expected to be unimodal too. Moreover, as the proportion of fine particles goes down in the soil-cement material, the water retention capacity would be expected to show the same trend. However, this will also depend on the morphology of the composite material. In this context, Figure 5 shows microstructural analysis on a soil-cement sample determined by SEM. Figure 5 (a) shows the porous surface aspect of soil (left) and cement (right), and Figure 5 (b) shows the cracking in soil-cement surface, due to the effect of the cement drying shrinkage. This cracking can also be observed in the lower part of the Figure 2.

Table 2. Chemical properties and mineralogical composition in the soil-cement.

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Oxide content (% by weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{Al}_2\text{O}_3 )</td>
<td>16.134</td>
</tr>
<tr>
<td>( \text{CaO} )</td>
<td>1.802</td>
</tr>
<tr>
<td>( \text{Fe}_2\text{O}_3 )</td>
<td>3.626</td>
</tr>
<tr>
<td>( \text{K}_2\text{O} )</td>
<td>3.591</td>
</tr>
<tr>
<td>( \text{MgO} )</td>
<td>0.792</td>
</tr>
<tr>
<td>( \text{MnO} )</td>
<td>0.009</td>
</tr>
<tr>
<td>( \text{Na}_2\text{O} )</td>
<td>0.273</td>
</tr>
<tr>
<td>( \text{P}_2\text{O}_5 )</td>
<td>0.061</td>
</tr>
<tr>
<td>( \text{SO}_3 )</td>
<td>0.106</td>
</tr>
<tr>
<td>( \text{SiO}_2 )</td>
<td>68.710</td>
</tr>
<tr>
<td>( \text{TiO}_2 )</td>
<td>0.984</td>
</tr>
<tr>
<td>Minerals content into soil-cement (%)</td>
<td>60% Quartz, 33% Clays micas and 7% Clays kaolinites</td>
</tr>
</tbody>
</table>

Figure 5. Microstructural analysis of soil-cement composite material by SEM: (a) surface aspect of soil and cement, and (b) cracking in soil-cement surface.
Besides, as it can be observed in Table 2, chemical properties and mineralogical composition of the materials show that soil-cement material is mainly composed of quartz (SiO$_2$/69%) and clay micas (Al$_2$O$_3$/16%), typical of the mineralogy composition of the Northern Spain. These results can be considered as the standard value of the properties of this material. Nevertheless, their values can be different depending on the cement content into the soil matrix. In this study it was 3.5% of Portland cement on matrix weight.

**Water retention curve of soil-cement**

Water retention curve and the best-fit four-parameters according to the van Genuchten model (VG-Model) for a soil-cement composite material with 3.5% of Portland cement are shown in Figure 6. Additionally, relationship between the values of volumetric water content experimentally measured and calculated by using a fit program (i.e., SWRC-Fit) are shown in Figure 6. As result, a nonlinear coefficient of determination (R$^2$) of 0.970 for the soil-cement material shows a fairly good correlation with the van Genuchten model [13].

Moreover, in Figure 6 it can be observed that saturated water content ($\theta_s$) should be close to the porosity, although when this parameter is characterized experimentally this value can be slightly different. For the analyzed material, the value of saturated water content ($\theta_s$) in the fitted retention curve and the porosity value ($n$), shown in Table 1, have been significantly coincident. Besides, the suction corresponding to zero water content was considered equal to $10^6$ kPa, according to Fredlund and Xing [12]. Moreover, Figure 6 shows that the soil-cement material presented a high adsorption capacity for high water suction (greater than 100 kPa). This happened due to the high surface area and surface charge of the soil-cement, see SSA in Table 1. Thus, recorded air-entry pressure was 83 kPa for the analyzed material. For this reason, materials with uniform grain-size such as soil-cement are characterized by flat characteristic curve from saturation to the air-entry point, because the majority of pores are drained within a narrow range of suction, showing a well-defined air-entry value [12].

Additionally, the parameter $n$ in the van Genuchten formulation, equation (1), indicates a greater uniformity in the pore-size distribution. For this reason, a higher value of $n$ is associated with a greater slope of the curve in the central section (see Figure 1), while a lower value is associated with a gentler slope, as is the case of the soil-cement. Additionally, in reference [18] it was shown that water retention curves for compacted fine soils in field and laboratory conditions can result very similar. The authors concluded that when the soil material is compacted under the same energy conditions (e.g., Proctor conditions) and water content, the compaction method exerts little influence on the water retention curve. This conclusion suggests that the determination of water retention curves in the laboratory, as in this study, adequately represents the behavior of soil porous material under field conditions.

Furthermore, Figure 7 shows a really close correlation between the values of volumetric water content (VWC) measured in laboratory and values adjusted by using the van Genuchten theoretical model. In this Figure, it can be seen that the sum of squared residuals values (SSR) was of $1.1 \times 10^{-4}$, showing a value less than $10^{-3}$. Therefore, it confirms that the fitting of the water retention curve according to van Genuchten model can be considered very satisfactory according to studies of Leong and Rahardjo [17] and Miller [18] on fine soil of similar texture and behavior to soil-cement composite material. It is suggested that the water retention curve and its parameters according to the van Genuchten model determined in this study, can be used as reference values for the parameters of unsaturated soil-cement material used in road pavement construction. Furthermore,
nonlinear fitting program used for obtaining the parameters, showed an excellent agreement between the experimental results and fit values obtained when the actual initial parameters of the material are not known. Finally, water retention curve and its parameters measured in the laboratory (see Figure 6) can be successfully used in numerical codes to assess unsaturated water flow through soil-cement in future analysis, as shown in [5].

CONCLUSIONS

This paper has presented the experimental results and procedures used to determine the water retention curve (WRC) of soil-cement composite material with a 3.5% of Portland cement. Matric suction (ψ) and water content (θ) measurements on the composite material have been successfully developed via pressure cells based on the axis translation technique. As a result, water retention curve shown in this study corresponds to one of the first results published in the literature on this composite material type.

Additionally, the following remarks have been obtained based on this research:

Fitting parameters of the WRC (θ_s, θ_r, α, n) were determined using the non-linear fitting program SWRC-Fit, and they were consistent with experimental results, even where soil-cement real initial parameters were unknown. This result was shown by the high coefficient of determination value (R^2) and the low Sum of Squared Residuals value (SSR) obtained.

Therefore, WRC and its best-fit parameters determined according to van Genuchten model can be used as reference values for the parameters of unsaturated soil-cement material used in road pavement construction.

Finally, WRC determined in this study can be included in numerical code for Finite Element Analysis to evaluate hydro-mechanical behavior of soil-cement material under unsaturated conditions.

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