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PHYLLITE CLAY–CEMENT COMPOSITES HAVING IMPROVED ENGINEERING PROPERTIES AND MATERIAL APPLICATIONS

E. Garzón[a,]*, M. Cano[a], Brendan C. O'Kelly[b], P.J. Sánchez–Soto[c]

[a]*Department of Engineering, High Politechnic College, University of Almería, La Cañada de San Urbano s/n, 04120-Almería, Spain.*
[d]*Department of Civil, Structural and Environmental Engineering, Trinity College Dublin, Dublin, Ireland.*
[c]*Institute of Materials Science of Sevilla (ICMS), Joint Center of the Spanish National Research Council (CSIC) and University of Sevilla (US), c/Américo Vespucio 49, 41092-Sevilla, Spain.*

*Corresponding author, Phone number: + 34 950 015529
E-mail address: egarzon@ual.es (E. Garzón)

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ABSTRACT

Phyllite clays contain clay minerals (chlorite, illite and mixed-layer illite smectite), quartz and feldspars. In this experimental laboratory study, new composites of phyllite clay and cement (5, 7 and 9 wt%) were prepared and tested to determine their Atterberg limits, dry density and optimum water content for modified Proctor (MP) compaction, California Bearing ratio, swelling potential after soakage in water, unconfined compressive strength (UCS) and water-permeability coefficient. From the mixes investigated, the composite with 5 wt% cement was deemed most suitable for certain construction material applications, having a plasticity index of 10.5%, maximum dry density of 2.17 Mg/m^3 and optimum water content of 8% for MP compaction (undergoing no swelling under soakage), a UCS of 0.74 MPa, and very low permeability coefficient value of 7.4×10^{-11} m/s. Potential material applications for these new composites include for building construction, roofs, and flexible pavements.

Keywords: cement, compaction, permeability, phyllites, plasticity, strength

Highlights

- First report on stabilization of clay phyllites using cement
- Effect of 5–9 wt% cement on engineering properties of clay phyllites
- Most suitable stabilization achieved for phyllite clay with 5 wt% cement

1. Introduction

Phyllite clays are rocks (metamorphosed to a low extent) of slate clay materials having an abundance of fine-grained phyllosilicates, which gives them an unctuous feel and the existence of preferential cleavage makes them easily breakable into thin sheets (Adom-Asamoah and Owusu-Afrifa, 2010; Garzón et al., 2009a; Oliva-Urcia et al., 2010; Ramamurthy et al., 1993; Valera et al., 2002). Phyllite clays can range in color from beige to violet and from reddish to grey and black. Although found in several parts of the world, phyllite clays are predominant in the Almería and Granada provinces (Andalusia region, southeast Spain) (Alcántara-Ayala, 1999; Garzón et al., 2009b; Lonergan and Platt, 1995), forming a band of Permo-Triassic materials, along with slates and marble. In recent years, a few publications have reported on different applications of phyllite clays in materials technology; e.g. as a filler in plastic (Valera et al., 2002) and concrete (Adom-Asamoah and Owusu-Afrifa, 2010; Ramamurthy et al., 1993) products. In southeast Spain, phyllite clays have been used as raw materials for some specific applications on account of their compaction properties and very low permeability, including: as covering and to waterproof roofs and the central area of ponds, core material in zoned dams and also for urban waste landfill applications (Garzón et al., 2009a, 2009b, 2010). In this instance, for flat roof applications, typically several layers of clay phyllites are placed and compacted on a cane matting base, which is supported by a framework of wooden beams. For gable or hip roof applications, the compacted phyllite clay layers are typically covered by clay brick tiles or slate leaves. From previous work by the authors, compacted phyllite clays sourced from the Almería and Granada provinces (Spain) do not undergo significant swelling on soaking on account of their low values of specific surface area, porosity and water-retention ability (Garzón et al., 2009a, 2009b, 2010). However, the expansivity of these phyllite clays at low applied stress limits certain applications; e.g. as a road subgrade material.

The improvement and (or) stabilization of clayey materials by the addition of cementing agents (e.g. cement or lime) in order to obtain superior engineering properties/performance is a well-established technology. The proper design of clayey soil–cement composites includes careful identification of the soil characteristics and an experimental testing programme aimed at identifying an appropriate cementing agent and mix proportion to achieve the required properties/performance for the composite material. Composite materials having attributes superior to those of the raw soil, but produced at similar relative cost, are attractive alternatives for soil material applications, such as Construction and Building Materials, Soil Engineering, and Civil, Structural and Environmental Engineering. Different methods have been reported in the literature on the use of some industrial additives or wastes as cementing agents to improve the properties/performance of raw clayey materials (Arabi and Wild, 1986; Ayuso 1982; Bell, 1996; Gidley and Sack, 1984), laterites (Osula, 1996), soil (Attom and Shariff, 1998; Bell, 1996; Miller and Azad, 2000), clayey soil (Kolias et al., 2004; Yong and Ouhadi 2007), residual soil (Basha et al., 2005), and expansive clay/soil (Al-Rawas et al., 2005; Ayuso, 1982; Seco et al., 2011). For instance, among the industrial waste materials investigated in these studies were burned olive waste (Attom and Shariff, 1998), cement kiln dust (Miller and Azad, 2000), fly ash (Kolias et al., 2005; Seco et al., 2011), rice husk ash (Basha et al., 2005), rice husk fly ash (Seco et al., 2011), artificial pozzolan (Al-Rawas et al., 2005), and coal bottom ash, natural gypsum and aluminatum filler (Seco et al., 2011).

Regarding clay–cement composites, Chang et al. (2007) studied the material properties of Portland cement paste with nano-montmorillonite additive. They reported that the composites comprising 0.6% and 0.4% of montmorillonite by weight of cement produced the optimum values for compressive strength and the permeability coefficient, respectively, with an increase in compressive strength of ~13% and a decrease in the permeability coefficient of ~50% produced. Hakamy et al. (2014) studied the characteristics of hemp fabric (HF) reinforced clay–cement composites. They reported an optimum replacement of ordinary Portland cement with 1 wt% clay decreased the porosity and significantly increased the density, flexural strength and fracture toughness of HF-reinforced nanocomposite. Potential building applications include the construction of sandwich panels, ceilings, roofing sheets, on-ground floors and concrete tiles. Wei and Meyer (2014) reported the partial replacement of Portland cement by a combination of metakaolin and clay (1, 3 and 5 wt%) in sisal fiber-reinforced cement composites enhanced mechanical properties.

However, we found no published work in the literature concerning the engineering or hydraulic properties of composites prepared using phyllite clays and cement additive. The present investigation reports an original experimental laboratory study on phyllite clay–cement composites undertaken by the authors to examine the improvement in selected engineering properties compared with the phyllite clay material itself.

2. Experimental

In the present investigation, selected phyllite clay samples, sourced from Berja (Almería, Spain), and white cement (CEM V/A 32.5 N/mm^2 (EN 197-1: CEN, 2000)) were used. In its natural state, the phyllite clay material had a very low gravimetric water content ranging 1–2% (mean of 1.8%), a void ratio (volume of voids to volume of solids) of ~0.39, and a dry density of 2.03 Mg/m^3 (Garson et al., 2010). The sampled phyllite clay material was oven dried at 105–110ºC to constant mass, allowed to cool to ambient laboratory temperature, disaggregated and then dry sieved to obtain the fraction passing < 125 μm aperture sieve. Using this size fraction of the phyllite clay, batches comprising 0, 5, 7 and 9 wt% cement were prepared for geotechnical index, compaction, unconfined compressive strength (UCS) and permeability testing. In preparing the composite materials, the phyllite clay and cement were dry mixed for a 1 h period to achieve homogeneity.

As part of the present investigation, the sampled phyllite clay was characterized by X-ray fluorescence (XRF), X-ray diffraction (XRD) and thermogravimetry**.** A sample was taken through successive quartering, crushed, lightly ground, sieved to obtain the fraction passing the 63 μm aperture sieve (No. 230 ASTM sieve), oven dried at 105–110ºC for a 24 h period, and then allowed to cool in a desiccator to ambient laboratory temperature. Aliquots of dried material (1–2 g) were then gently ground using an agate mortar for further analysis. For the XRF analysis, an Axios spectrometer (PANanalytical B.V., Germany) was used; with the experimental test conditions, standard certified materials and data processing required previously reported by Garzón et al. (2009a). For the XRD analysis, an X´PERT PRO X-ray diffractometer (PANanalytical B.V., Germany), was used at 36 kV and 26 mA settings, with Ni-filtered CuKα radiation and graphite monochromator. Oven-dried phyllite clay sub-samples were gently ground in an agate mortar and a random-oriented powder mount specimen prepared for XRD testing (Niskanen, 1964; Sánchez-Soto et al., 1993). The XRD instrument, with X'Celerator detector, had the following settings: 2θ range of 3–70º; step size of 0.03º (2θ); scan speed of 0.05/240 (2θ/s); counting time of 240 s; divergence slit of ½ (º2θ) and antiscatter slit of ¼ (º2θ). The identification of crystalline phases, according to the files by the Joint Committee for Powder Diffraction Standards, was performed using the software provided by the equipment.

The phyllite clay and the composites of phyllite clay and cement were characterized by their liquid limit (LL), plastic limit (PL) and plasticity index (PI) values, which were determined in accordance with standard sample preparation and testing procedures (ASTM D4318, 2005). Modified Proctor (MP) compaction tests and California Bearing Ratio (CBR) tests were performed over a range of compaction water contents in accordance with the sample preparation and testing procedures given in ASTM (2014). From the MP data, the optimum water content for compaction, and corresponding maximum dry density value, of the phyllite clay and composite materials were determined. The CRB test method is used to evaluate the potential strength of subgrade, subbase, and base course materials, for use in the design of road and airfield pavements. CBR values were determined by measuring the force required to cause the CBR plunger to penetrate at a specified rate into MP compacted specimens which had been allowed to soak in a water bath for 4 d. The swelling potential of the MP compacted specimens was determined from the measured longitudinal dimensional change of the compacted soil cylinders under soakage (ASTM, 2014). Unconfined compression tests (ASTM, 2013) and water-permeability testing under constant confining stress and controlled-gradient conditions in the triaxial cell were performed on MP compacted specimens (50 mm in diameter by 100 mm long) of the phyllite clay and the composites of phyllite clay and cement; these specimens having been allowed to cure in a wet chamber for a 7 d period before performing these tests).

Finally, the thicknesses (*E*, in cm) of the flexible pavement required for road work construction using the phyllite clay and its cement composites were calculated using Peltier's equation (Dal-Ré, 1994):

$$E = (100 + 150\mathrm{P}^{1/2})/(I + 5) \qquad (1)$$

where *P* is the maximum wheel load (tonne), estimated at 3 tonne, and *I* is the CBR value, determined as described earlier.

3. Results and discussion

XRF analysis of the raw phyllite clay samples is reported in Table 1 and XRD in Figure 1. From the latter, the mineralogical composition of this material, which had a 6.8% loss in dry mass after a 1 h ignition period at 1000ºC, was identified as chlorite and illite (main clay minerals), quartz with some minor aluminosilicates, potassium feldspar, and an interstratified phase which was identified as mixed-layer illite smectite or possible chlorite smectite. Iron oxide was also detected as a minor component. This mineralogical composition agreed with the chemical composition reported in Table 1. The amount of SiO_2 is associated with the presence of quartz and silicates (illite, chlorite, feldspars and interstratified phase). The content of CaO and MgO can be mainly related to the chlorite identified by XRD. The alkaline elements (sodium and potassium) are associated with illite and feldspar because these silicates contain potassium. The 6.8% loss in dry mass on ignition is consistent with the presence of phyllosilicates having structural OH groups, which are lost by termal treatment at 1000^{o}C.

Figure 2 presents the measured LL, PL, and PI (defined as the numeric difference between the LL and PL) values for the phyllite clay and the composites of phyllite clay and cement in their remolded state. For the range of 5–9 wt% cement investigated, the addition of cement produced a step increase in the LL (from 26% to 36%) and the PL (from 17% to 24–25%) (Fig. 2a). This had the effect of producing an apparent approximately linear increase in PI (from 8.4% to 12%) with increasing cement content over the range 0–9 wt% cement (see Fig. 2b). Further, this caused a change in plasticity characterization, from low plasticity (LL < 35%) for the phyllite clay, to intermediate plasticity (LL = 35–50%) for the composites with 5, 7 and 9 wt% cement. This behavior is influenced by the presence of a relative high proportion of clay minerals (chlorite and illite) and the mixed layer in the raw phyllite clay. Hence, the addition of up to 9 wt% cement does not appear effective in reducing the sensitivity of the phyllite clay to water content variation (Bell, 1996; Kolias et al., 2004; Young and Ouhadi, 2007).

Referring to Fig. 3, MP compactive effort produced quite high maximum dry densities, which were greater than that of the in-situ phyllite clay material (2.03 Mg/m^3, Garson et al. (2010)). The MP maximum dry density reduced slightly, and approximately linearly, with increasing cement content; from 2.25 Mg/m3 for the phyllite clay to 2.14 Mg/m^3 for the composite with 9 wt% cement. Further, the addition of cement produced a moderate (and again an approximately linear) increase in the optimum water content for MP compaction; from 6.5% for the phyllite clay to 9% for the composite with 9 wt% cement (Fig. 3). This behavior is consistent with that reported previously for other clayey materials, expansive clays and soils (Al-Rawas et al., 2005; Ayuso, 1982; Basha et al., 2005; Kolias et al., 2004; Osula, 1996; Yong and Ouhadi, 2007). The slight reduction in maximum dry density values for the clay–cement composites, compared with the phyllite clay, may be explained by the lower density of the cement additive and the higher rigidity of the soil skeleton produced for the composite materials. The moderate increase in the optimum water content is consistent with the increase in plasticity caused by the addition of cement (Fig. 2b). These changes can be associated with a pozzolanic reaction (i.e. chemical reaction between the clay minerals present in the test materials), as occurs with related clay materials (Al-Rawas et al., 2005; Arabi and Wild, 1986; Ayuso, 1982; Basha et al., 2005; Gidley and Sack, 1984; Kolias et al., 2004; Miller and Azad, 2000; Osula, 1996; Seco et al., 2011; Yong and Ouhadi, 2007). In the present study, clay minerals (chlorite and illite) are the main components of the phyllite clay investigated.

Referring to Table 2, the CBR values of the composites with 5–9 wt% cement were significantly greater, 36–50% at 100% MP and 15–32% at 95% MP, compared with the corresponding values for the phyllite clay of2.5% and 1.7% respectively. Yong and Ouhadi (2007) proposed a mechanistic model on wetted-state instability of road bases founded on natural and cement-stabilized clayey soils containing phyllosilicates (illite, chlorite and kaolinite), palygorskite (attapulgite), and other minerals including quartz, gypsum, arcanite, thendernite, calcite, and dolomite. Because of the palygorskite present, the clayey material they investigated had some very unique features, with the formation of a transformation product of this fibrous silicate increasing the swelling potential. In the present investigation, the phyllite clay had a measured swelling value of 3.6%, whereas the composites with 5–9 wt% cement additive did not experience any swelling under soakage (Table 2). The swelling behavior of phyllite clay is associated with its mineralogical composition (particularly that of the clay minerals), with the zero swelling potential for the composite materials most likely due to the pozzolanic reaction with the 5–9 wt% cement additive.

Compared with the phyllite clay, the required thickness E of the road pavement necessary to support vehicular traffic provoked by a linear work (Eq. 1) was significantly lower for the composite with 5 wt% cement (see Table 2). Further, based on the limited available data, a general trend of a modest reduction in the pavement thickness occurred with increasing cement content over the range 5 to 9 wt% cement investigated. Hence, the addition of cement to phyllite clays for road construction would allow considerable reductions in overall costs.

Table 3 lists the measured permeability coefficient values for the MP compacted test materials which were of the order of 10^{-10} to 10^{-11} m/s, indicating extremely low permeability. The permeability coefficient values of the composites with 5 and 7 wt% cement were approximately an order of magnitude greater than that measured for the phyllite clay and the composite with 9 wt% cement.

Figure 4a presents unconfined compressive stress against axial strain plots for the different test materials. The UCS increased approximately linearly in value with cement content (Fig. 4b), mobilizing 1.02 MPa for the composite with 9 wt% cement, approximately twice that for the phyllite clay (0.52 MPa). These strength values are in broad agreement with the range reported by Dal-Ré (1994) for expansive soils stabilized with cement for use in earth construction. The stiffness (Young's modulus) was also found to increase with increasing cement content (Fig. 4a); e.g. the composite with 9 wt% cement was three times stiffer than the phyllite clay. However, the test materials were quite brittle, with the axial specimen strain corresponding to the UCS reducing from 1.3% (phyllite clay) to 0.75% (composite with 9 wt% cement) (Fig. 4b).

The results of this experimental study indicate that a relatively low addition of cement can produce significantly higher UCS values (0.74 MPa for 5 wt% cement), compared with the raw phyllite clay (0.52 MPa). On this basis, 'green ceramic bodies' (e.g. bricks and tiles) can be produced at relatively low additional cost using ground phyllite clay with 5–9 wt% cement addition. This was demonstrated in the laboratory by depositing phyllite clay–cement mixtures into 330 x 330 x 16 mm (for bricks) and 280 x 280 x 14 mm (for tiles) molds, consolidating, curing for a 7 d period, and de-molding. Using a conventional laboratory press and moderate values of confining pressure, bricks and tiles of different shapes can be manufactured for ready-to-use applications (particularly as impermeabilization products having moderate compressive strength), without the need for firing. However, the PI range (Fig. 2a) is not sufficient for processing of these composite materials by extrusion techniques. Other potential material applications include for building construction, flexible pavements, and road sub-base and sub-grade construction.

4. Summary and conclusions

This study reported a new class of composite prepared using phyllite clay and cement additive at 5, 7 and 9 wt%, which has improved engineering properties over the raw phyllite clay. For 5 wt% cement, the composite material had a plasticity index of 10.5%, a maximum dry density of 2.17 Mg/m^3 and an optimum water content of 8% for MP compaction, an unconfined compression strength of 0.74 MPa, and very low permeability coefficient value of 7.4 x 10^{-11} m/s.

Potential material applications include for building construction, roofs, pavements, and road sub-base and sub-grade construction. For instance, bricks and tiles can be manufactured using ground phyllite clay and cement additive, conventional pressing and a curing period of 7 d, before use in-service. In such instances, pyllite clay–cement composites have the potential for use as a low-cost alternative when they are available locally, such as in the Andalusia region, Spain. Further research on the use of phyllite clays in the preparation of mortars and concrete (cement matrix composites) for specific material applications is underway and will be the subject of future reports.

Acknowledgements

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FOUR Figures

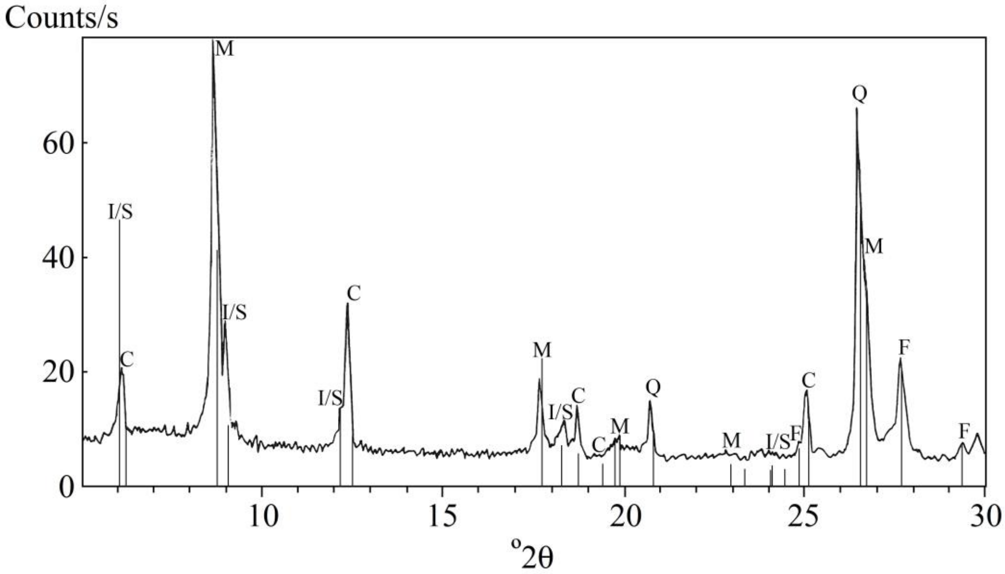

Figure 1. XRD diagram of clay phyllite sample. Note: C, chlorite; F, feldspar; I/S, interstratified illite/smectite phase; M, mica (illite); Q, quartz.

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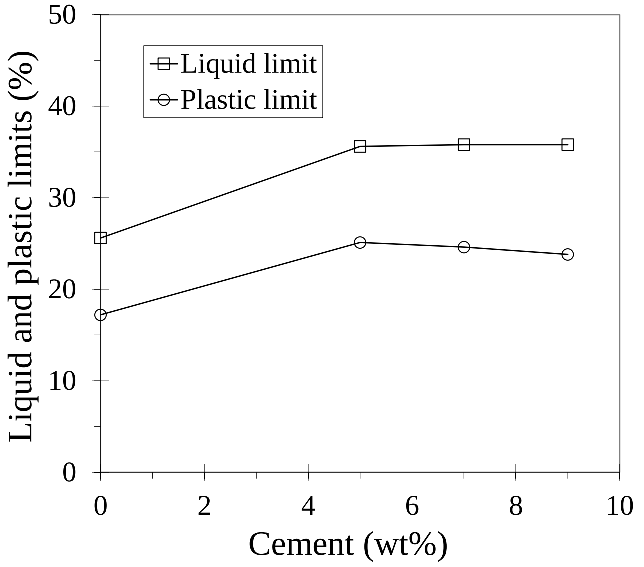
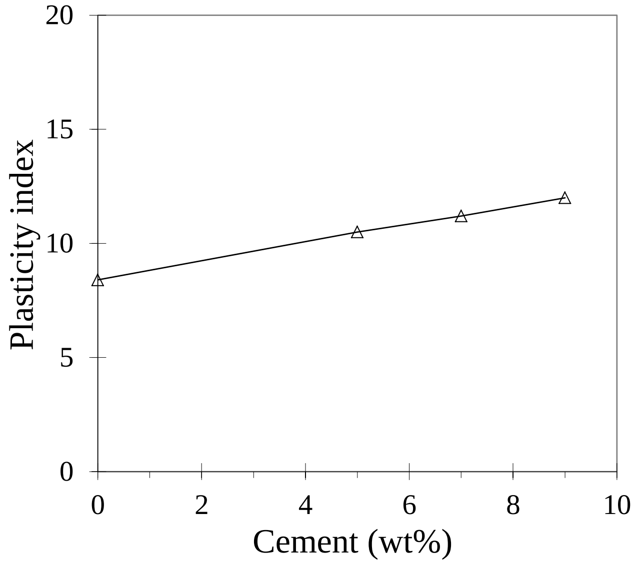


(a) Liquid and plastic limits.



(b) Plasticity index.

Figure 2. Consistency limits for phyllite clay and composites with cement.

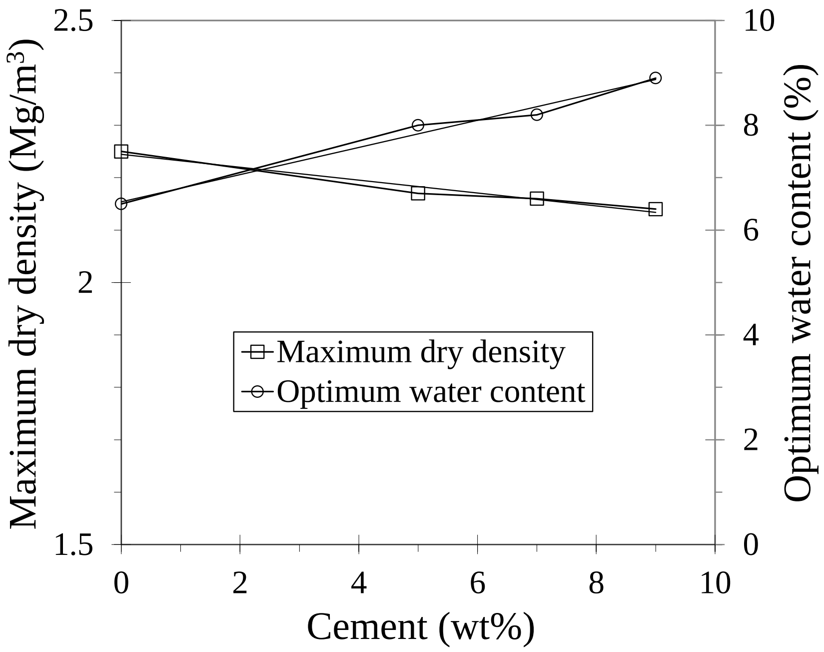
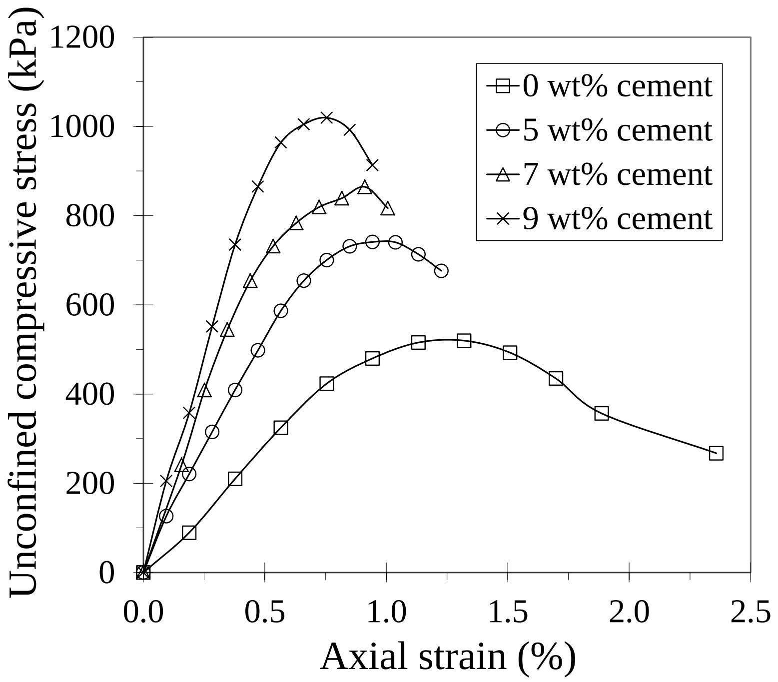
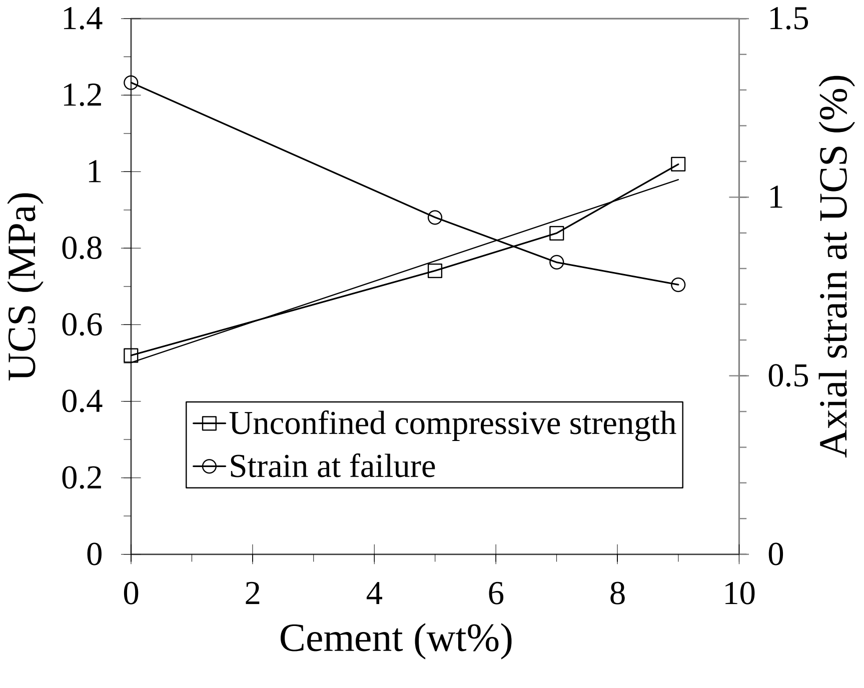


Figure 3. Modified Proctor compaction test results.



(a) Unconfined compressive stress (in kPa) against axial strain.



(b) Unconfined compressive strength (UCS, in MPa) and corresponding axial strain for different cement content.

Figure 4. Unconfined compressive strength testing of specimens cured for 7 day period.

THREE Tables

Table 1. Chemical analysis by X-ray fluorescence. Note: $P_2O_5 < 0.1\%$; $MnO < 0.08\%$.

	Weight (%)		
	Sample 1	*Sample 2*	*Sample 3*
SiO_2	45.66	49.70	48.33
Al_2O_3	24.36	23.40	22.04
Fe_2O_3	9.41	8.51	8.35
TiO_2	1.30	1.01	1.15
CaO	3.06	1.68	4.43
MgO	2.81	2.95	3.43
Na_2O	2.33	2.45	1.84
K_2O	3.91	3.84	3.32

Table 2. Results of CBR and swelling tests and calculated road pavement thickness (refer to Eq. 1). Note: E_1 and E_2, thicknesses of the road pavement required based on measured CBR values for 100% and 95% of MP maximum dry density, respectively, determined in accordance with ASTM (2014).

Test material	**CBR at 100% MP (%)**	**CBR at 95% MP (%)**	**Swelling (%)**	E_1 **(cm)**	E_2 **(cm)**
Phyllite clay	2.5	1.7	3.6	48.0	53.7
Phyllite clay with 5 wt% cement	43	15	0	8	18
Phyllite clay with 7 wt% cement	50	28	0	7	11
Phyllite clay with 9 wt% cement	36	32	0	9	10

Garzón *et al*. (2015) Phyllite clay–cement composites having improved engineering properties and material applications. *Applied Clay Sciences* 114(September 2015): 229–233.

Table 3. Evolution of permeability coefficient for MP compacted phyllite clay samples with addition of cement.

Material	**Permeability coefficient (m/s)**
Phyllite clay	1.8×10^{-11}
Phyllite clay with 5 wt% cement	7.4×10^{-11}
Phyllite clay with 7 wt% cement	4.0×10^{-10}
Phyllite clay with 9 wt% cement	1.4×10^{-11}

END