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# Alkali activated soil-ash mixtures Activation alcaline de mélanges sol-cendre

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ABSTRACT Mixtures of soil, fly ash and an alkaline solution made from sodium hydroxide and sodium silicate were artificially moulded using different sodium hydroxide concentrations and water contents. The chemical reactions involved in the alkaline activation of fly ash produce a geopolymeric gel that links the soil particles with cementitious bonds. Two different soils were tested, and the effect of the activator and water content on workability, stiffness and strength was carefully evaluated by means of unconfined compression strength tests, indirect tensile strength tests and seismic wave measurements. The results indicate that the addition of water has a negative effect on stiffness, in both soils, and in strength, for the coarse soil.

RÉSUMÉ Mélanges de sol, cendres et une solution alcaline composée par hydroxyde et silicate de sodium ont été moulés artificiellement en utilisant différentes concentrations d'hydroxyde de sodium et teneurs en eau. Les réactions chimiques impliquées dans l'activation alcaline des cendres volantes produisent un gel géopolymèrique qui lie les particules de sol comme en ciment. Deux sols différents ont été essayés et l'effet de la teneur en liquide et de l'eau sur la faisabilité, la rigidité et la résistance ont été soigneusement évalués par essais de compression simple, essais de traction indirecte et des mesures d'ondes sismiques. Les résultats indiquent que l'ajout d'eau a un effet négatif sur la rigidité des deux sols et aussi dans la résistance du sol grossier.

# 1 INTRODUCTION

The production of cement has severe environmental impacts, using vast amounts of fossil fuels and being responsible for the emission of around 5% of all the carbon dioxide worldwide (Worrell et al. 2005). In this paper an alternative to cement for soil improvement is proposed, based on the reuse of an industrial waste – coal burning fly ash. The alkaline activation of fly ash to create an alternative binder to Portland cement has been successfully applied to replace traditional Portland cement concrete (Palomo & Fernandez-Jimenez 2004, Turner & Collins 2013).

It consists in the reaction of a solid aluminosilicate (like fly ash) with a highly concentrated aqueous alkali hydroxide or silicate solution producing a synthetic alkali aluminosilicate material with comparable performance to traditional cementitious binders, but with significantly reduced greenhouse emissions (Duxson et al. 2007).

However, few studies have been carried out in soil improved by the alkaline activation of fly ash (Cristelo et al. 2013). In fact, adding this new binder to a soil changes the conventional soil-cement paradigm in terms of moulding parameters. Void ratio, compaction degree and water content, among others, which have a known effect on soil-cement mixtures (Consoli et al 2011, 2012), need to be validated for this new material.

In previous work Rios & Viana da Fonseca (2014) highlighted the potential of this material since strength and stiffness are highly improved when the alkaline solution and fly ash are added. In this paper, the influence of water is more deeply analysed distinguishing between water content and liquid content. While the water content is the ratio between the mass of water by the mass of dry soil, the liquid content is the mass of alkaline solution by the mass of dry soil. In this new material water affects not only compaction but also the concentration of the alkaline solution justifying the need of a careful analysis of these two effects.

# 2 MATERIALS AND METHODS

#### 2.1 Soils, fly ash and alkaline solution

The tested specimens were moulded with two different soils. This first, called herein IP, is a remoulded residual soil from granite, typical from the Porto region, collected near the Civil Engineering Department in a construction site for a new building of Porto University. It is a very well graded soil with around 30% of fines as presented in Rios and Viana da Fonseca (2014). The second soil, designed by SF, corresponds to the finer part of the previous soil, passing on the ASTM sieve n0. 200 (< 0.075 mm). It is mainly composed by kaolinite and silt, i.e. nonplastic.

Type F fly ash was obtained from a coal thermoelectric power plant in central Portugal.

The alkaline solution comprised sodium hydroxide (SH) and sodium silicate (SS). SH was in flake form with a specific gravity of 2.13 at 20°C and 95-99% purity was dissolved in water up to the desired concentration. The SS was already in solution form with a specific gravity of 1.5 and  $SiO_2/Na_2O$  ratio of 2 by mass.

#### 2.2 Experimental program

The experimental program described herein comprises unconfined compression, and indirect tensile strength tests and seismic wave measurements on different specimens, moulded specifically for this study. Mixtures of soil, ash and alkaline solution were all compacted to the same dry unit weight of 17.41  $kN/m^3$ . This value corresponds to the Normal Proctor optimum dry density of IP soil plus 10% of fly ash (Figure 1). The amount of fly ash was kept constant at 10% of the mass of solids (soil and fly ash) as well as the ratio of sodium silicate over sodium hydroxide (SS/SH=0.5). The SH concentration varied from 4, 6, 8 and 10 molal.



Figure 1. Normal Proctor curve of the IP soil mixed with 10% of fly ash

Proctor tests are performed in mixtures without curing in order to evaluate the compaction properties. Consequently, the addition of the alkaline solution is considered to have marginal influence of the optimum compaction point. However, the moisture content associated to a soil+ash+water mixture is only due to water while in an alkaline activated mixture the liquid content is not only composed by water. In addition, as the alkaline solution triggers the chemical reactions that will produce the geopolymeric gel that links the soil particles, the evaluation of the water content becomes much more complicated. In this sense, it is difficult to understand whether the optimum point observed in the Proctor curve should be interpreted as the liquid or water content of the mixtures.

To evaluate the effect of water on workability, strength and stiffness of the mixtures, four different types of mixtures were prepared as follows:

- T0) Mixtures of soil, fly ash and water at a water content of 13.6% (no alkaline solution)
- T1) Mixtures of soil, fly ash and alkaline solution considering a liquid content of 13.6%
- T2) Mixtures of soil, fly ash and alkaline solution considering a water content of 13.6%

The liquid and water contents are calculated as a percentage of the solids (ash and soil). This means that for T1, the concentration of the sodium hydroxide changes the water content while for T2 different concentrations result in distinct liquid contents. Table 1 presents the values of liquid and water contents for the two types of mixtures depending on the concentration of sodium hydroxide.

The name of the specimens presented in the next section includes the type of soil (IP or SF), the type of mixture (T0, T1 or T2) and SH concentration. For example, SF\_T0 means the mixture of SF soil with 10% of fly ash, while IP\_T1\_4 means IP soil with 10% of fly and 13.6% of alkaline solution with 4 molal SH concentration.

 
 Table 1. Liquid content and water content depending on the concentration and on type of mixture

	SH concentration	Liquid content	Water content
	(molal)	(%)	(%)
T1	4	13.60	10.57
	6	13.60	10.06
	8	13.60	9.62
	10	13.60	9.23
T2	4	17.50	13.60
	6	18.38	13.60
	8	19.23	13.60
	10	20.05	13.60

#### 2.3 Testing procedures and equipment

Seismic wave velocities measurements were performed on the specimens during curing at the following periods: 0, 3, 7, 14, 21 and 28 days. Compression and shear waves were measured by means of P and S wave ultrasonic transducers as described by Amaral et al. (2013). These transducers were linked to a signal generation and data acquisition Pundit lab unit from Proceq connected to a laptop computer for display and data storage. To improve coupling between the transducer and the specimen, contact gel for ultrasound testing was used which highly improves the signal quality without damaging the specimen. In fact, one of the main advantages of these transducers is the easiness of application avoiding holes in the specimen which could prevent the subsequent wave analysis or the unconfined compression tests performed at the end of curing periods. Several fixed frequencies were used ranging from 24 to 500 kHz. Depending on the stiffness some specimens led to clearer signals than others, but the propagation time was not sensitive to frequency, i.e, it was mostly constant with it.

At the end of the curing period (28 days) the specimens were tested in unconfined compression (UCS) and indirect tensile strength (CD) performed according to the corresponding European standards (CEN, 2003a and b). An automatic loading machine of 100 kN of capacity together with a load cell of the same capacity and resolution of 0.006 kN was used. The UCS test speed was adjusted to 0.05 mm/min in order to enable the execution of small unload-reload cycles at different compression stresses. Local deformation transducers – LDTs (Goto et al., 1991) and Hall-Effect transducers (Viana da Fonseca et al., 2013) were used for accurate strain measurement during the test.

#### 3 RESULTS AND DISCUSSION

#### 3.1 Compression and tensile tests

Figure 2 presents the stress-strain curves of T0 mixtures without alkaline activation of fly ash. It is assumed that in these mixtures there are no chemical reactions and therefore, the strength is solely due to the soil and ash mechanical strength without any cementation. Consequently, the differences between the two curves are due to the different characteristics of the soils. The finer soil (SF) presents much higher strength (almost 10 times higher) than the other soil (IP).



Figure 2. Stress-strain curves of both soils mixed with fly ash and no alkaline solution

The effect of the alkaline activation and the SH concentration is presented in Figure 3 and Figure 4 comparing T1 and T2 approaches, having T0 as reference. For that purpose, T0 strength is presented in a dashed horizontal line since it is not affected by SH concentration.

In IP soil the additional water content associated to T2 approach seems to have a negative effect on strength comparing with T1 approach. T1 mixtures show higher strength values which increase with SH concentration. The significant decrease in strength for the 10 molal SH concentration (in T2 specimen) was not expected so further tests are needed to confirm this. On the contrary, in the finer soil (SF) extra water seems to have a positive effect on strength which might be associated to a higher workability. Being a very fine soil, it is expected that higher water content is needed to achieve the same workability and compaction degree.



Figure 3. Unconfined compression strength (UCS) of IP mixtures with SH concentration



Figure 4. Unconfined compression strength (UCS) of SF mixtures with SH concentration

Indirect tensile tests were also performed on some specimens to evaluate the ratio between tensile and compressive strength. Figure 5 shows the results obtained for SF\_T2 mixtures. It is interesting to notice that the ratio between indirect tensile strength and unconfined compression strength is very high when compared to what is generally observed in cemented soils. In fact, ratios above 30% were registered in alkaline activated mixtures while Rios & Viana da Fonseca (2013) obtained 10% in soil-cement specimens.



Figure 5. Comparison between unconfined compression strength (UCS) with indirect tensile strength (CD) of SF\_T2 mixtures

#### 3.2 Dynamic stiffness evolution with curing time

The dynamic stiffness was evaluated by S and P wave measurements using time domain analysis for signal interpretation according to Viana da Fonseca et al. (2009). Having obtained the seismic wave propagation time the corresponding velocities were derived dividing the distance between transducers (i.e., the specimen height) by the time. The dynamic modulus ( $E_{din}$ ) was calculated through the elasticity theory using equations 1, 2 and 3. Figures 6, 7, 8 and 9 show the evolution of  $E_{din}$  with curing time.

$$G = \rho V_{\rm S}^{2} \tag{1}$$

$$\upsilon = \frac{\left(\frac{V_P}{V_S}\right)^2 - 2}{2\left(\frac{V_P}{V_S}\right)^2 - 2}$$
(2)

$$E_{din} = 2G(1+\nu) \tag{3}$$



**Figure 6.** Dynamic modulus evolution with curing time for IP\_T1 mixtures at various SH concentrations



Figure 7. Dynamic modulus evolution with curing time for SF\_T1 mixtures at various SH concentrations



**Figure 8.** Dynamic modulus evolution with curing time for IP\_T2 mixtures at various SH concentrations



Figure 9. Dynamic modulus evolution with curing time for SF\_T2 mixtures at various SH concentrations

The analysis of these graphs shows very interesting results distinct from what was observed in the strength trends. In fact, conversely to UCS values, IP mixtures showed higher stiffness than SF specimens. While IP mixtures have small stiffness at the beginning of the curing process and then show a significant increase with time, SF start from considerable stiffness values. This is in agreement with the stressstrain curves presented in Figure 2 where a greater initial tangent stiffness modulus is observed in SF\_T0 rather than in IP\_T0.

Comparing T1 and T2 approaches, T1 seems to show higher stiffness especially at higher SH concentrations. Since higher concentration means less water for the same liquid content (in T1), it can be concluded that less water resulted in higher stiffness. This is valid for both soils including the finer soil which has showed an increase in strength with water.

# CONCLUSIONS

Strength and stiffness analysis of two soils mixed with alkali activated fly ash were performed by unconfined compression tests, indirect tensile tests and seismic wave measurements. The evolution with sodium hydroxide (SH) concentration was observed together with the effect of the quantity of water in the mixture. The results indicate that the addition of water has a negative effect on stiffness for both soils and also in strength for the coarse soil. This was especially evident for higher SH concentrations where the quantity of water is reduced.

The ratio between indirect tensile strength and unconfined compression strength was especially high (between 28% and 43%) revealing that these mixtures might be more resistant to tensile stresses than other cemented materials.

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