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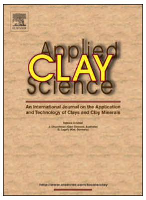

Research paper

Sulfate soils stabilization with magnesium-based binders

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ABSTRACT

Sulfate soils' stabilization is a very interesting subject with technical, economic and environmental implications. The difficulty of their stabilization is due to the fact that the usual stabilizer additives are based on calcium. In these soils, sulfate combines with the calcium from the additive and the aluminum from the clay, resulting in a highly hydrated expansive mineral named ettringite. This provokes the swelling of the treated material and even its destruction. This study analyzes the result of the substitution of the calcium based additives by one alternative additive based on magnesium, an industrial byproduct named PC-8, in the stabilization of five different sulfate soils. From a mechanical point of view soils treated with PC-8 reached similar resistance values to the lime treated ones, of about 2–3 MPa for 4% dosage and 2–5 MPa for 8% dosage, being usually better with the PC-8 results than with the lime ones. When PC-8 was combined with GGBS the resistance values increased up to 11–13 MPa and the lime-GGBS reached the 6–7 MPa. The natural swelling of the soils treated with PC-8 decreased substantially and maintained constant even for immersion at long-term. In the case of the soils treated with lime, long-term swelling increased up to very high values even in the case of soils without natural swelling. XRD analysis of these samples demonstrated the existence of ettringite in 4 of the 5 soils when they were treated with lime and there was not expansive minerals in the PC-8 treated soils, agreeing with the swelling observed behavior of the soils when treated with both additives.

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1. Introduction

Soils' stabilization based on lime or cement is a widely used technique to improve the soils' properties as a construction material. The use of these additives is particularly suitable in clayey soils, which usually have inadequate properties like high plasticity, bad workability as well as low bearing capacity (Guney et al., 2007; Lin et al., 2007; Göktepe et al., 2008). Clayey soils consist in aluminosilicate mineral layers with negative surface charge that, by electrostatic repulsion, maintain an open structure. With the addition of lime, Ca^{2+} ions fixation takes place, electrostatic charges of the clay layers are balanced and electrochemical repulsion forces between them are reduced. This causes the adhesion of the clay particles in flocs where the clay layers are now linked by Ca^{2+} ions, resulting in a soil with improved properties: a more granular structure, higher permeability and lower plasticity (Kinuthia et al., 1999; Lin et al., 2007). In addition, the lime's hydration releases OH^{-} ions that increase the pH up to about 12.4. Under these conditions pozzolanic reactions take place in the soil: Aluminum (A) and silicon (S) from the clay matrix are solubilized and combine with the available calcium (C) generating hydrated (H) cementitious compounds schematically named CSH, CAH and CSAH (Nalbantoglu, 2004; Guney et al., 2007; Yong and Ouhadi, 2007; Chen and Lin, 2009). These compounds are responsible of the improvement of the mechanical properties of the stabilized soil upon curing time, as pozzolanic reactions develop. The kinetics of these reactions depend on the amount and availability of the concerned oxides and they can last even years (Wild et al., 1998). The clay matrix usually has enough aluminum and silicon, which allows the use of lime instead of cement, being a cheaper and effective treatment (Chen and Lin, 2009). In those soils where enough silicon and aluminum are not available, they have to be added with the additive. In these cases it is recommended the use of products like Ordinary Portland Cement (OPC), rich in available SiO_2, Al_2O_3 and CaO, that provides the needed oxides, shortening also the reaction time (Wild et al., 1998; Degirmenci et al., 2007). The improvement of the obtained properties will depend on the quantity and richness of the additive, the soil's mineralogy, its particles' size and shape and the curing conditions (Misra et al., 2005; Yarbasi et al., 2007; Göktepe et al., 2008).

Despite the demonstrated technical, economic and environmental suitability of the use of calcium based additives as stabilizers for soils, this type of treatment may cause adverse effects in some soils and even lead to the total destruction of the stabilized material.

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One of the most common causes of this type of failure is the presence in the stabilized soil of sulfate (SO_4^{2-}). Sulfate is a very common oxide as well as in many natural soils around the world as in industrial wastes. Other times it may stem from the water used for compaction, or arise from infiltration from the surrounding terrain. Whatever its origin, the reaction of the calcium supplied by the lime or cement, aluminum, silicon and the sulfate all together in the presence of water, causes the formation of expansive minerals, the most common being a highly hydrated and expansive crystalline mineral named ettringite [$Ca_6Al_2(SO_4)_3(OH)_{12}·26H_2O$] (Crammond, 2002; Nobst and Stark, 2003; Ciliberto et al., 2008; Norman et al., 2013). The mechanics of the formation of ettringite are not well established (Mohamed, 2000), although the conditions for its formation are known (Ouhadi and Yong, 2003), which are: (1) high pH, (2) presence of soluble Al, (3) presence of soluble Ca, (4) presence of soluble sulfate and (5) availability of water. It is also known that the rate of ettringite formation is accelerated by high temperatures (Rajasekaran, 2005). Talero (2005) and Rahhal and Talero (2014) pointed out different formation rates, shape and size of the ettringite's crystals based on different reactive aluminum compounds from OPC (C_3A) and pozzolanic additions ($Al_2O_3^{r-}$). Given the right conditions, ettringite formation is the dominant reaction, even appearing during the mixing of the materials. Mohamed (2000) determined the total time of formation of ettringite in a natural marly soil within 48 h. In their experiments Ouhadi and Yong (2003, 2008) established the formation of this mineral over one month in one case, and between the mixture of the soil with lime and 48 h in another experiment. From a chemical point of view, the reactions involved in the formation of ettringite are (Ouhadi and Yong, 2008):

$$CaO + H_2O \rightarrow Ca(OH)_2$$

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2(OH)^-$$

$$Al_2Si_4O_{10}(OH)_2 \cdot nH_2O + 2(OH)^- + 10H_2O \rightarrow 2\{2Al(OH)_4^- + 4H_4SiO_4\} + nH_2O$$

$$M_xSO_4 \cdot nH_2O \rightarrow XM^y + SO_4 + nH_2O$$

where

$x = 1, \quad y = 2$

or

$x = 2, \quad y = 1$

$$6Ca^{2+} + 2Al(OH)_4^- + 4OH^- + 3\left(SO_4^{2-}\right) + 26H_2O \rightarrow Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$$

A second way for the ettringite formation has been observed in OPC mortars and concretes where, after long curing periods in wet conditions, sulfate attacks the CAH, generating what is commonly referred to as "delayed ettringite", which provokes the same undesirable effects in the cementitious materials (Lee et al., 2005; Escadeillas et al., 2007; Ciliberto et al., 2008; Chen and Jianj, 2009; Pavoine et al., 2012; Nguyen et al., 2013). The swelling capacity of the soil in which ettringite develops, and therefore its undesirable effects, are produced during the formation of this mineral. Once ettringite is present in the soil, it is stable and even contributes to improve bearing capacity (Kinuthia et al., 1999; Rajasekaran, 2005; Min et al., 2008).

One way to reduce the formation of ettringite in a stabilized sulfate soil is the partial substitution of lime or cement by ground granulated blastfurnace slag (GGBS). GGBS contributes with a large amount of quickly available aluminum and silicon both of which react with the available calcium, expending it for the cementitious gels formation, avoiding the formation of ettringite. This protective effect against sulfate attack is especially intense for high replacement ratios of lime with GGBS (about 83%). Furthermore, GGBS also produces a denser cementitious matrix, reduces the permeability and hence the water availability, increasing the durability against the internal and external sulfates' attack (Wild et al., 1998; Tasong et al., 1999; Obuzor et al., 2012). However this way of limiting the sulfate attack must to be used carefully because all the oxides needed for the ettringite formation remain in the treated material (Celik and Nalbantoglu, 2013). Another probable way to stabilize the sulfate soils can be the replacement of calcium based additives by other metal cations, such as magnesium. Xeidakis (1996a,b) demonstrated that the $Mg(OH)_2$ fixing in the expansive clay layers is more rapid than that $Ca(OH)_2$, resulting also in the clay's flocculation. He also proved the $Mg(OH)_2$ capacity to increase the soil's pH and the theoretical possibility to generate Mg based cementitious gels.

The lack of guaranties about the sulfate soils stabilization with calcium additives, the magnesium capacity to flocculate clays and its eventually cementing properties, both make the magnesium-based additives a priority research subject.

This study analyzes the possibility to stabilize sulfate-containing soils with magnesium-based additives instead the calcium-based ones, taking into account the capacity to create cementitious gels as well as the reduced potential for the formation of expansive minerals due to the stabilization of both additives.

2. Materials and methods

2.1. Materials

Three natural soils (I, II and III) and two based in waste artificial soils (IV and V) were considered in this study. Table 1 shows the characterization of the analyzed soils from a soil mechanics and chemical points of view. All the laboratory test were carried out in accordance with Spanish and European Standards, mineralogical compositions were estimated by X-ray diffraction (XRD) analysis based on the chart proposed by Al-Rawas (1999) and the soils composition, expressed as their most significant oxides, were based on X-ray fluorescence (XRF) analysis.

Soils I, II and III were obtained from different geological formations across the Ebro's Valley in the North of Spain. They are expansive clayey soils from different geological tertiary structures with different sulfate contents.

Soil IV comes from the fraction smaller than 5 mm, obtained from crushing of old concrete structures in a building site in the city of Pamplona (Spain). This recycled material contains a 5% of sulfate, mainly because of the plasterboards remains crushed together with the concrete. Soil V consists of a mix of a 70% slag from a biomass plant that burns cereal straw, with a 30% of a local marly soil to get adequate workability properties for the soils mechanical tests.

The second kind of materials considered are the following additives: (1) PC-8. This material is a by-product rich in MgO calcined during magnesite production by means of the calcination of natural $MgCO_3$ rocks up to 1100 °C. This process is carried out in a rotatory kiln with crosscurrent air circulation, which pulls dust particles along the whole kiln. So that, this dust contains $MgCO_3$ (inert), calcined MgO (reactive) and vitrified MgO (inert) particles and it is recovered by means of air cyclones. The sample used in this study was supplied by Magnesitas de Navarra S.A. Company. (2) Commercial hydrated lime CL-90-S. Rich in $Ca(OH)_2$, it has been used like reference calcium based additive for the soils treatment, and (3) GGBS. This by-product of the steel production has been used combined with PC-8 and lime as activators. The sample used was supplied by Hanson Cement Company.

Table 2 shows the additives' properties and their XRF obtained oxide composition.

In the case of the soils the only pre-treatment made consisted on grinding and sieving to get particles sizes smaller than 2 mm to allow a better contact soil-additive and thus, a more efficient treatment. In the case of the additives no pre-treatment was necessary.

2.2. Samples' preparation

Two kind of samples were prepared during the experimental investigation: The first category are the samples to establish the mechanical properties of the tested combinations. The mixing of soils and additives

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