

Influence of sulfate and chloride ions on the geotechnical and microstructural properties of soils treated with hydraulic binders: Individual and coupling effects



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ARTICLE INFO

Article history:

Received 8 December 2013

Received in revised form 24 December 2014

Accepted 31 January 2015

Available online 12 February 2015

Keywords:

Soil stabilisation

Hydraulic binders

Disruptive anions

Co-addition

Volumetric swelling

Indirect tensile strength

SEM observations

ABSTRACT

The use of soils after treatment with hydraulic binders results in the improvement of their physical and mechanical characteristics for geotechnical applications. The influence of sulfate and chloride ions, introduced as $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and NaCl , on physical and mechanical properties of a treated soil is investigated.

For the considered soil, in the presence of one of these anions, in accelerated cure conditions, important volumetric swellings due to high concentrations in sulfate ions are the only disturbances observed. The co-addition of sulfate and chloride ions induces both important volumetric swellings and loss of indirect tensile strengths. Structural modifications are observed by scanning electron microscopy.

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1. Introduction

In situ stabilisation of soils with hydraulic binders improves their engineering properties for geotechnical applications. The French technical guide for soil treatment (GTS) (LCPC-SETRA, 2000b) points out the presence of some chemical compounds in the soil to explain the occurrence of important volumetric swelling and/or weak mechanical strengths: sulfate, chloride, nitrate and phosphate ions. Recent works (Cabane, 2004; Guichard, 2006; Le Borgne, 2010) try to determine the concentrations at which they could be potentially disruptive towards stabilisation, by macrostructural and microstructural tests.

In soil stabilisation studies, sulfate ions are recognised as the cause of ettringite development and volumetric swellings (from the most recent references Harris et al., 2004; Little et al., 2005; Rajasekaran, 2005; Beaucour and Raynaud, 2008 for example). From the literature data, the swelling risk due to the ettringite formation appears from 0.03 g of $\text{SO}_4^{2-} \cdot \text{kg}^{-1}$ of dry soil (Cabane, 2004; Saussaye, 2012).

In soil stabilisation studies, chloride ions are identified as an accelerator or retarder according to the soil nature or the treatment formulation tested (Xing et al., 2009; Le Borgne, 2010). Xing et al. (2009) notice that an increase of concentration in chloride ions reduces the proportion of CSH. The chloride ions react with the Ca^{2+} and Al^{3+} ions to form hydrocalumite $\text{Ca}_2 \cdot \text{Al}(\text{OH})_6 \cdot \text{Cl} \cdot (\text{H}_2\text{O})_2$. This mineral covers the clay surface, without improving the cohesion. Besides, in cementitious materials, chloride ions are known to react with C_3A to form Friedel's salt (calcium chloroaluminate) and delay the development of the calcic compounds (calcium aluminate hydrate CAH and calcium silicate hydrate CSH) (Barberon et al., 2005; Lubelli et al., 2006; Saikia et al., 2006; Elakneswaran et al., 2009). But this influence can be modulated by the presence of other ions such as sulfate (Neville, 2000).

Besides, it is also of major importance to note the influence of the associated counter ions. The sodium sulfate is recognised to have a negative action on the development of mechanical strengths of treated soils by Harris et al. (2004), Rajasekaran (2005) or Xing et al. (2009), whereas Yilmaz and Civelekoglu (2009) use the calcium sulfate as an agent of solidification of soil. Besides, in the case of chloride ions, Yuan et al. (2009), throughout a literature review, point out the positive influence of Na^+ compared to Ca^{2+} .

The present study determines the geotechnical behaviour of a treated soil spiked with sulfate and/or chloride ions in order to define the

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concentrations at which there is a risk of stabilisation disturbance. Then, when a disturbance is established, the mineralogical and microstructural parameters involved are investigated.

2. Experimental procedures

2.1. Soil characteristics

The effects of anions, by single or co-addition to the soil, are studied on one soil, from Loucelles, in Normandy region (France). The geotechnical classification of soils is realised according to the French technical guide for road embankments (GTR) (LCPC-SETRA, 2000a) and the NF P 11-300 standard. The organic matter content is low (loss of mass by ignition < 1%) as well as the clay content (clay fraction of 10%). A D_{max} of 50 mm, a fine fraction of 77% and a methylene blue value of 0.8 g/100 g of dry soil classified the soil as fine and silty soil A1 (or silt of low plasticity, sensitive to the variations of water of the environment, noted SL in the USCS).

The understanding of the mechanisms of stabilisation of the treated soils requires microscopic observations of the materials. The mineralogy of the soil is analysed by X-ray diffraction (XRD) (EN 13925-1 standard) on Bruker D5005 (anode Cu, in configuration $\theta/2\theta$, with an acceleration tension of 40 kV and a current of 30 mA). The selected soil is mainly composed of quartz SiO₂ and calcite CaCO₃. No halite, gypsum or some chlorinated or sulfur minerals are detected.

The total concentrations in the soil of some major elements are inductively coupled plasma atomic emission spectroscopy (ICP-AES) (NF ISO 22036 standard) after acid mineralisation (EN 1356 standard). However, as Al and Si are poorly recovered after the acid mineralisation used, their concentrations are not reported here. The water soluble part of these elements is also measured after a water extraction of the soil. The chloride and sulfate concentrations of the water soluble fraction are analysed by ionic chromatography. The detailed analytical procedures are given elsewhere (Saussaye, 2012). The values determined from 25 measurements on the soil sample, as presented in Fig. 1, are reported in Table 1. The average values of the total concentrations values are similar to those reported for the earth crust by Allègre and Michard (1973).

$R_{W/T}$, the ratio of mean water soluble fraction concentrations (C_W) on total concentrations (C_T), is also reported in Table 1, and calculated according to Eq. (1):

$$R_{W/T} = 100 \times C_W / C_T \quad (1)$$

$R_{W/T} \leq 1\%$ means that a low part of the elements could react with the hydraulic binders during the soil treatment.

The SO₄²⁻ and Cl⁻ contents in the water soluble fraction are similar to those reported by Calvet (2003) in the soil interstitial solution, from 13 to 182 mg of SO₄²⁻·kg⁻¹ and from 16 to 142 mg of Cl⁻·kg⁻¹ of dry soil.

Table 1
Chemical characteristics of the soil.

	Major element concentrations (mg·kg ⁻¹ of dry soil) (mean value (standard deviation))		$R_{W/T}$ (%)
	C_T	C_W	
Ca	19606 (6179)	208 (28)	1.06
Fe	19966 (1912)	< detection limit	< 5.52·10 ⁻⁴
K	3466 (1790)	17 (2)	0.48
Mg	2860 (497)	4.2 (0.2)	0.15
S	137 (24)	27 (8)	19.42
SO ₄ ²⁻	Not measured	98 (25)	–
Cl ⁻	Not measured	92 (38)	–

C_T : total concentration in the soil.

C_W : concentration in the water soluble fraction.

2.2. Chemical addition

In order to be largely superior to the initial natural concentration values and to evaluate disturbance levels, four values are chosen for the concentration of each anion: 3, 5, 7 and 10 g of anion·kg⁻¹. The anions are added respectively as CaSO₄·2H₂O and NaCl. The sulfate and chloride ions are noted respectively as S and C hereafter. The treated soil is subjected to suitability tests to treatment to detect the failure risk (see Section 2.3.1). To evaluate the co-addition effect, the two chemical products are added simultaneously. The protocol of additions of the chemical compound(s) is developed by Saussaye (2012).

The association of the NaCl and CaSO₄·2H₂O salts in solution obviously induces new ionic interactions. The influence of Na⁺–SO₄²⁻ interaction is evaluated by the addition of 7 and 10 g of SO₄²⁻·kg⁻¹ as Na₂SO₄·10H₂O (mirabilite) to the soil before the HRB treatment. The addition of sodium sulfate is noted (Na)S hereafter. However, these compounds do not really exist in the soil: only the released ions interact during the hydration. As the aim of our study is to define the influence of sulfate ions in the presence of chloride ions, the interaction Ca²⁺–Cl⁻ is not investigated.

2.3. Treatment and disruption evaluation

2.3.1. Geotechnical evaluation

According to the methylene blue value measured and the French standards (Abdo, 2008), a formulation of 1% of quicklime (CaO) and 6% of HRB is applied. The binders meet the EN 459-1 and NF P15-108 standards criteria. The treatment methodology is defined by the NF P94-093 standard and is detailed in Saussaye (2012).

The compaction references, the specimen preparation and the test procedures were carried out according to the GTS (LCPC-SETRA, 2000b, 2007) and the EN 13286 standards. In this paper, only results of the suitability tests are reported (NF P94-100, EN 13286-42 and EN 13286-49 standards). This is an accelerated procedure which allows quick detection of the failure risk (results obtained in 7 days). The criteria of suitability to the treatment are as follows:

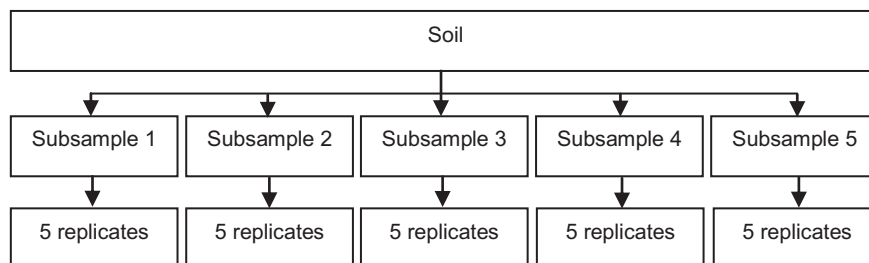


Fig. 1. Subdivision of the sampling soil for physicochemical characterisations.

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