



Influence of anions on the geotechnical properties of soils treated with hydraulic binders: Individual and coupling effects



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HIGHLIGHTS

- A soil is submitted to an addition of SO_4^{2-} , Cl^- , NO_3^- or PO_4^{3-} before treatment.
- The influence of SO_4^{2-} alone or co-added with another anion is evaluated.
- Anions influence is estimated through swelling and indirect tensile strength.
- When co-added to other anions, disturbances appear at weaker SO_4^{2-} contents.
- Co-addition induces more important disturbances than single addition of SO_4^{2-} .

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ABSTRACT

The treatment of soils with hydraulic binders improves their physical and mechanical characteristics, allowing the soils to be reused in several geotechnical applications such as road construction. The aim of this research study is to assess the influence of sulfate, chloride, nitrate and phosphate ions, introduced as $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, NaCl , NH_4NO_3 and KH_2PO_4 , on the physical and mechanical properties of a treated soil under accelerated curing conditions. Then, the influence of sulfate ions is determined in the presence of another anion.

Regarding the influence of each anion on the soil considered, only phosphate ions have a deleterious effect on the stabilization at concentrations between 3 and 10 g kg^{-1} of dry soil. The sulfate ions induce an important volumetric swelling at 10 g kg^{-1} .

However, the combination of one anion with sulfate ion induces important disturbances on both volumetric swelling and indirect tensile strength: these interactions must be taken into account in the determination of the stabilization of disruptive parameters.

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1. Introduction

The use of soils *in situ* in road construction is possible with the application of a treatment with hydraulic binders. Cabane [1] indicated that, in France, in 2000, about two million tons of soil per year were recognized as unsuitable for the treatment due to an important volumetric swelling and/or weak mechanical strength.

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These disturbances can be associated with the presence in the soils of chemicals such as sulfate, chloride, nitrate and/or phosphate ions. The French technical guide for soil treatment (GTS) [2] suggests this risk without any indication of the chemical form and/or the concentration of the anion of interest.

GTS [3] indicates that sulfates have a harmful action at 10 g kg^{-1} . Several researchers [4–11] have recognized that sulfates are an important factor in altering soil stabilization, inducing considerable swelling by the development of ettringite ($3(\text{CaO}) \cdot (\text{Al}_2\text{O}_3) \cdot 3(\text{CaSO}_4) \cdot 32(\text{H}_2\text{O})$). Cabane [1] tried to generalize the threshold of concentration beyond which an important swelling is observed that compromises the structural stability. From the literature data, the swelling risk would appear from 0.03 g kg^{-1} . Multiple factors can explain this distinction on the risk level, such as the nature of the soil or the treatment formulation. The nature of the counterion associated with the sulfate ion can also be of great

importance. For example, Yilmaz and Civelekoglu [12] used gypsum (calcium sulfate) as an agent for solidification of soil and noted a beneficial action of Ca^{2+} . The sodium sulfate was recognized to have a negative effect on the development of mechanical strength of the treated soils [11,13,14]. The influence of potassium sulfate was not much discussed in the literature [8,15,16] and generally, the influence of potassium was not approached from the point of view of the mechanical strength. For Kinuthia et al. [15] the potassium sulfate would invert the effects of a lime treatment on plasticity and compaction parameters.

GTS indicates that chlorides can induce volumetric swelling. In the solution of a cement paste, the chloride ions are generally known to have a beneficial influence on the concrete durability. Calcium chloride (CaCl_2) is an accelerating admixture for cement. Several authors [17–21] have recognized this action, without explaining the mechanism. According to the observations of Xing et al. [14], the effect of chloride ions would be similar to the effect observed in cement-based materials [17,22–26], i.e., they react with C_3A to form Friedel's salt (calcium chloroaluminate) and delay the development of the calcium compounds (calcium aluminate hydrate (CAH) and calcium silicate hydrate (CSH)). Yuan et al. [27] prepared a literature review on the influence of chlorides on cementitious materials: they showed the positive influence of Na^+ by comparison to Ca^{2+} . Xu [28] and Delagrave et al. [29] indicated that the binding of CaCl_2 is more important than the binding of NaCl .

GTS indicates nitrates as retarders, even inhibitors, of setting. If various types of nitrate were studied to judge their stabilization in a cementitious matrix, the ammonium nitrate was targeted in the studies of soil treatment [30,31]. Cabane [1], by analogy with cementitious materials, proposed a scenario of disturbance of the solidification of soils in the presence of ammonium nitrate (NH_4NO_3). The reaction between the ammonium ion and the lime results in delays in setting and causes the release of ammonia and most likely a reduction of the pH of the environment until the disappearance of the ammonium ions. Then, the setting of the binder can occur. The nitrate ion ensures the stability in solution of the calcium ion at high pH (12.4) [1] and results in the formation of calcium nitroaluminate minerals and, later, in a fissuring of the treated material. Calcium nitroaluminate ($3(\text{CaO})\cdot(\text{Al}_2\text{O}_3)\cdot 3(\text{Ca}(\text{NO}_3)_2)\cdot X(\text{H}_2\text{O})$) was the mineral targeted by various authors [32–34] in the presence of nitrate in the cementitious environment.

GTS indicates phosphates as retarders, even inhibitors, of setting. Blot [35] noted that phosphate ions are known to have a dispersive capacity in a soil suspension. This property is not favorable to the flocculation of clays during the lime pretreatment. Guichard [30] indicated that, at pH equal to 12.4, the phosphate ions (amphoteric ions) are present as PO_4^{3-} and react with the calcium ions to form insoluble calcium phosphate (hydroxyapatite), delaying the setting. According to Cabane [1], the mechanism of delay of the hydraulic setting is the formation of calcium phosphate (hydroxyapatite), until total consumption of the soluble phosphate ions. Once the soluble phosphate is eliminated, the system evolves towards the formation of the hydrated calcium aluminates (CAH). Several researchers [36–40] reported the formation of hydroxyapatite in combination with CSH and/or $\text{Ca}_3(\text{PO}_4)_2$ during the hydration of a cement in the presence of sodium phosphate, potassium phosphate or ammonium phosphate. Crannell et al. [41], during the stabilization of incineration waste by soluble phosphates, indicated the formation of $\text{Ca}_3(\text{PO}_4)_2$ and of $\text{Ca}_5(\text{PO}_4)_3\text{OH}$, with a stabilization of approximately 50% of the water soluble calcium. The stabilization of the phosphate ions would thus take place before the treatment of the soil.

Among the four anions recognized by the GTS as being potentially disruptive, the sulfate ions are the most widely studied. However, the influence of this anion depends on the ionic interactions within the interstitial solution of the soil that cannot be neglected.

For example, the chloride–sulfate interactions were often studied in efforts dealing with the influence of the sea water on cementitious materials [28,29,42–54]. The internal sulfate reaction was supposed to be moderated by the presence of the chloride ions, in competition for the hydration reactions of the C_3A . However, Revertegat et al. [55] indicated that the chloride ions also lead to the ettringite formation by favoring the alumina–sulfate reaction and increase the volumetric swelling. Jallad et al. [38] observed the formation of hydroxyapatite when they were studying thaumasite ($\text{CaCO}_3\cdot\text{CaSO}_4\cdot\text{CaSiO}_3\cdot 15\text{H}_2\text{O}$). With an immersion in a solution of KH_2PO_4 and Na_2HPO_4 , at pH 6, they noted that the surface of the thaumasite was completely covered with hydroxyapatite. However, for higher values of pH, between 7 and 12, the mineral is no longer detected. Only calcium carbonate (CaCO_3) is detected. Kordlaghari and Rowell [56] showed that, in a soil, the presence of gypsum can increase the absorption of the phosphate ions, added as KH_2PO_4 . The microstructural analyses, SEM and XRD, enabled us to observe a disappearance of the gypsum and a formation of calcium phosphate. This transformation would thus take place before the treatment, releasing the sulfate ions in the interstitial solution of the soil.

Sulfate, chloride, nitrate and phosphate ions have an influence on the properties of soils treated with hydraulic binders. However, no specific chemical forms or threshold concentrations have been defined until now to predict the disturbances in stabilization. The influence of each anion depends on the presence of the others.

In this paper, the results are associated with one soil treated with quicklime and hydraulic road binder (HRB) and subjected to geotechnical tests under accelerated curing conditions, as recommended by the GTS to quickly detect chemical solidification disturbances and performed by French embankment companies. First, concentration thresholds for each disruptive element beyond which the soil stabilization could be altered are searched. Then, the concentration threshold for sulfate ions is redefined in the presence of another anion. The knowledge of these thresholds should allow us to optimize the treatment formulations and the rate of reuse of soil on site for road construction.

2. Experimental procedures

2.1. Soil characterization

The effects of anions, by single or co-addition to the soil, are studied on one soil, sampled from Loucelles, in the Normandy region (France). Loucelles is located on a Jurassic calcareous horizon. The geotechnical classification of soils is performed according to the French technical guide for road embankments (GTR) [57] and the NF P 11-300 standard: the soil is classified as fine and silty soil A1 (Dmax of 50 mm; fine fraction of 77%; methylene blue value of 0.8 g/100 g of dry soil). The soil is low in organic content (loss of mass by ignition <1%) and low in clay content (clay fraction of 10%).

The physicochemical characterization is performed on the 500 kg of soil sampled, the amount necessary for the whole study. The characterization consists of the determination of the total element concentration as well as the soluble water element and anionic concentrations. The methodology of microwave-assisted acid mineralization, water soluble fraction extraction and analysis are described by Saussaye [58]. The mineralization method does not allow the correct measurement of the total content of Al and Si. As indicated in Table 1, the collected soil is fractionated into 5 samples and each sample, after homogenization, is divided into 5 subsamples (replicates). The mean value for the concentration of each element is calculated from 25 measurements. The results are in accordance with the mean world values of the earth crust [59]. The anionic concentrations of the water soluble fraction range from 81 to 136 mg of $\text{SO}_4^{2-}\cdot\text{kg}^{-1}$ of dry soil, from 16 to 115 mg of $\text{Cl}^{-}\cdot\text{kg}^{-1}$, from 41 to 86 mg of $\text{NO}_3^{-}\cdot\text{kg}^{-1}$, whereas the phosphate ions are present in concentrations below to 0.1 mg kg^{-1} . These minimum and maximum anionic concentrations detected in the soil interstitial solution remain in the range of the most frequent world values [60], even if important range values are noticed. Table 1 shows that standard deviations are often weaker for a sample (5 replicates on a homogenized sample) than for the soil (25 replicates). The soil heterogeneity combined with the numerous subsamples analyzed might explain the wide ranges of concentrations values observed. The physicochemical heterogeneity of the soil most likely induces different behavior towards the hydraulic binders treatment.

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