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The influence of partial saturation on the engineering properties of a metakaolin-improved sand

L'influence de la saturation partielle sur les propriétés techniques d'un sable amélioré au métakaolin

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ABSTRACT: Ground improvement by injection of sustainable binders and geopolymers is generating much interest. The use of these products in foundations, landslides, excavations, retaining structures and tunnel applications is being progressively considered in geotechnical standards. This fact would require consideration of material evolution under mechanical or environmental action, including changes in the hydraulic state of the ground. An extensive laboratory investigation was conducted on sand specimens treated with a metakaolin-based geopolymer to evaluate the effect of different saturation conditions on the main properties required for ultimate and serviceability limit states for ground improvement works. Curing the treated samples under partially saturated conditions dramatically affected the stiffness characteristics at low strains, the shear strength under unconfined and axisymmetric conditions, and the response to cyclic loading. In any case, the geopolymer satisfactorily avoided liquefaction initiation within the number of cycles investigated. A decrease in water permeability was observed as a function of the degree of filling of the voids with the binder and an increase in retention capacity and air-entry value compared to the untreated material. Mercury intrusion porosimetry and micro-CT provided microstructural insight into these phenomenological observations.

RÉSUMÉ: L'amélioration des sols par injection de liants durables et de géopolymères suscite un grand intérêt. L'utilisation de ces produits dans les fondations, les glissements de terrain, les excavations, les structures de soutènement et les tunnels est progressivement prise en compte dans les normes géotechniques. Cela nécessiterait de prendre en compte l'évolution des matériaux sous l'action mécanique ou environnementale, y compris les changements dans l'état hydraulique du sol. Une étude approfondie en laboratoire a été menée sur des échantillons de sable traités avec un géopolymère à base de métakaolin afin d'évaluer l'effet de différentes conditions de saturation sur les principales propriétés requises pour les états limites ultimes et de service pour les travaux d'amélioration du sol. Le durcissement des échantillons traités dans des conditions partiellement saturées a considérablement affecté les caractéristiques de rigidité à faibles déformations, la résistance au cisaillement dans des conditions non confinées et axisymétriques, ainsi que la réponse aux chargements cycliques. Dans tous les cas, le géopolymère a évité de manière satisfaisante l'initiation de la liquéfaction dans le nombre de cycles étudiés. Une diminution de la perméabilité à l'eau a été observée en fonction du degré de remplissage des vides par le liant et une augmentation de la capacité de rétention et de la valeur d'entrée d'air par rapport au matériau non traité. La porosimétrie par intrusion de mercure et la micro-CT ont fourni un aperçu microstructural de ces observations phénoménologiques.

Keywords: Sand improvement; metakaolin; partial saturation; geotechnical properties.

1 INTRODUCTION

Ground improvement aims to increase the mechanical properties of soils and rocks while decreasing permeability in engineering applications.

The traditional Ordinary Portland Cement (OPC) is used for ground improvement. However, other materials, known as non-conventional binders, are used as an alternative to OPC for ground improvement purposes, such as colloidal silica, resins, micro-fine cement, and geopolymers (e.g., Anagnostopoulos and Sapidis, 2017; Salvatore et al., 2020; Spagnoli et al., 2022; Fraccica et al., 2022).

It is critical to analyse the hydro-mechanical behaviour of non-conventional binders that could be used as an alternative to OPC in sustainable engineering practices. When exposed to acid solutions, geopolymer materials outperform OPC (e.g., Bakharev, 2005).

This work has focused on Soil Deep Mixing (SDM)-adaptable binder (metakaolin-based geopolymer - MK). To the authors' knowledge, no commercial applications of MK-based binders for ground improvement are known. Most laboratory studies on geopolymer-treated soils focus on volcanic ash, fly ash, or ground blast-furnace slag (e.g. Verdolotti et al., 2008; Rios et al., 2017). Metakaolin-based geopolymers have received little attention for soil treatment and have typically been used with other geopolymers or OPC (e.g. Kolovos et al., 2013). Furthermore, in many laboratory investigations, soil and geopolymer precursor powders are combined dry before the activator is applied, and the samples are compacted (e.g. Zhang et al., 2013). However, this does not reflect how SDM is often performed in situ because already-activated slurries are combined with wet soils and no compaction is performed (Yaghoubi et al., 2018). Consoli et al. (2011) and Spagnoli et al. (2022) have demonstrated that the soil structure obtained after compaction (final porosity and amount of binder) has a significant impact on the strength and stiffness of the treated material.

Finally, few studies have been conducted to investigate the combined impact of extreme dry curing conditions and lengthy curing times on soil binder performance (e.g. Rios et al., 2017).

The current paper focuses on the permeability, static and cyclic strength of sandy soils treated with a metakaolin-based geopolymer after curing in dry and wet conditions at a relative humidity RH = 50% and 100%, respectively.

2 MATERIALS AND METHODS

The base material used in this study is a medium quartz sand (Holcim 0.2–0.6 mm), while the binder is an alkali-activated metakaolin. The metakaolin is activated with a water solution of potassium silicate. Potassium silicate solution, water and metakaolin are mixed in equal masses, leading to a geopolymer with a fluid/metakaolin mass ratio of 2:1 and a mass ratio between dry metakaolin and dry sand of 5.2% and 13% for initial slurry volumetric void filling F_{MK} values of 40% and 100%, respectively. Specimens were prepared by pouring the pure sand or the sand+geopolymer mixture into moulds. Specimens for Unconfined Compression (UC) tests are 70 mm in diameter and 164 mm in height. In contrast, those for cyclic and monotonic triaxial undrained compressions (CTX, STX) are 38 mm in diameter and 96 mm in height. After an initial 3-day common stage in the casting mould (at 20°C and RH = 50%), some specimens were cured at 20°C in dry conditions and some in wet conditions, at a relative humidity RH = 50% and 100%, respectively. The last condition was achieved by submerging the samples in water after three days of curing. The following nomenclature identifies the specimens: S, sand; MK, geopolymer; SMK, geopolymer+sand. The F_{MK} achieved is indicated between parenthesis; the curing condition is indicated by a letter (W: water immersion, D: curing at 50% RH) and a number indicating the curing time in days. For example, SMK(100)D7 indicates sand+geopolymer, with a F_{MK} = 100%, dry-cured for 7 days. D or W is not indicated for specimens at 3 days curing as they were submitted to a common preparation stage.

3 HYDRO-MECHANICAL TESTS

3.1 Microstructural observations

Treated soil's microstructure was investigated using mercury intrusion porosimetry (MIP), X-ray tomography, Field Emission Scanning Electron Microscopy (FESEM) and paraffin tests (Spagnoli et al., 2022; Fraccica et al., 2022). An increase in macro-pores was observed in the dry-cured specimens by MIP, resulting from micro-cracks formation in the geopolymer and detachment at the grain/geopolymer contact (FESEM). X-ray and paraffin tests revealed a decrease in dry density with dry curing (void ratio $e \approx 0.42$ after three days and $e \approx 0.47$ after 100 days).

3.2 Hydro-mechanical properties

After evaluating the bar wave velocity v_b in dry- and wet-cured samples through an ultrasonic pulse velocity tester (Proceq Pundit Lab Lab+, see Spagnoli et al., 2022), the small-strain Young's modulus was calculated as follows:

$$E_0 = \rho(t_c) v_b^2 \tag{1}$$

where $\rho(t_c)$ is the bulk density at a given curing time. At similar curing times, lower stiffness values were observed on dry-cured samples than on wet-cured ones (Table 1).

UC tests were conducted at a 0.5%/min axial deformation rate on specimens with a height/diameter ratio between 2 and 2.5. Lower Unconfined Compression Strengths (UCS) were observed in dry-cured samples, with a slight decreasing trend with curing time (Table 1).

Isotropically consolidated undrained triaxial compression (TXCIU) tests were performed under monotonic and cyclic conditions. After saturation, the water permeability was evaluated under steady-state conditions by imposing a back pressure of 200 kPa at the bottom and 10 kPa at the top cap. The treatment progressively contributed to decreased soil water permeability as the F_{MK} and the curing time increased due to void clogging and the solidification of geopolymers (Figure 1).

Table 1. Mechanical properties of treated sand under dry and wet curing.

Specimen	E_0 (GPa)	UCS (MPa)
SMK(100)3	-	1.44
SMK(100)3	-	2.07
SMK(100)D7	0.59	1.18
SMK(100)W7	3.44	2.05
SMK(100)D28	0.71	1.10
SMK(100)W28	5.40	1.51

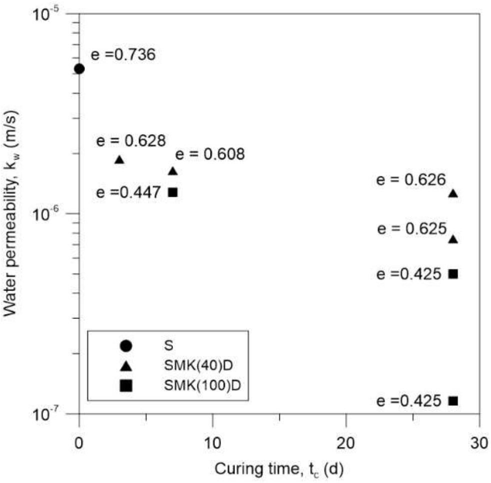

Figure 1. Water permeability of geopolymer-treated sand at different curing times. Void ratio as labels.

Consolidation was induced under isotropic stresses p'_0 = 200 and 600 kPa. The shearing phase was carried out with a back-pressure of 500 kPa under stress-controlled conditions (47 kPa/min). Increasing F_{MK} resulted in more dilatant, stiffer and stronger specimens (Fraccica et al., 2022). Similar to what was observed for UC tests, samples with longer dry curing periods presented slightly lower shear strength starting from similar isotropic consolidation pressures (Figure 2). The different treatments of F_{MK} increased soil cohesion with no changes in the friction angle.

Cyclic undrained triaxial compressions (CTXCU) were performed; consolidation was carried out under K_0 = 0.5 until reaching σ'_1 = 250 kPa. Cyclic shearing was imposed through 600 cycles (1 Hz) at a cyclic stress ratio CSR = 0.25 and a back pressure of 400 kPa (Fraccica et al., 2022). The untreated sand underwent large strains (i.e. higher than 5%) after the first ten cycles with a fast increase in r_u (Figure 3) and liquefaction. The treatment reduced strains (i.e. lower than 0.07%), cyclic stiffness degradation and excess pore pressures.

Small treated soil subsamples (~300 mm^3) were extracted from dry-cured specimens to evaluate their water retention properties. A dew-point psychrometer measured total suction while gravimetric water content was assessed by oven drying at 105°C. Results from MIP tests were converted into Soil Water Retention Curves (SWRC) through the procedure explained by Romero (1999). The main drying curve of the treated sand was evaluated starting after 28 days of dry curing (Figure 4).

The soil's air-entry value rose after the treatment. The soil's ability to retain water decreased with greater total suctions, as seen by a sharp decline in the degree of saturation S_r.

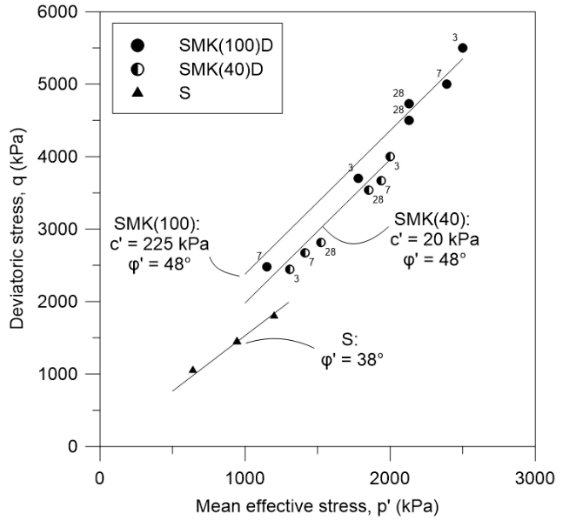

Figure 2. Shear strength envelopes for untreated and treated sand. Curing days as labels.

4 DISCUSSION AND CONCLUSIONS

Dry curing impacted the microstructure and hydro-mechanical behaviour of the metakaolin-treated sand.

Compared to wet-cured samples, microcracks and grain-binder detachments led to a decrease in stiffness and strength over time and an increase in the void ratio. Unlike untreated conditions, void clogging, filling ratio, and binder solidification helped reduce permeability and boost shear strength.

The treated soil's higher stiffness contributed to lower strains and excess pore pressures in the CTXCU, avoiding liquefaction. The almost saturated material showed a very high air-entry value due to the higher void filling with fine clayey material; this behaviour was likely caused by the microstructural changes previously noted. The S_r value then sharply decayed. Therefore, when assessing the effects of such a ground improvement technology on geotechnical parameters in partially or saturated soils, consideration should be given to slurry void filling, curing duration, and *RH* conditions.

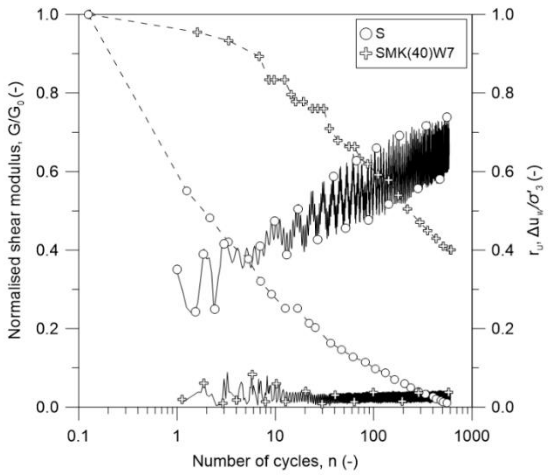

Figure 3. Cyclic stiffness degradation (dotted lines) and normalised excess pore pressure (continuous lines).

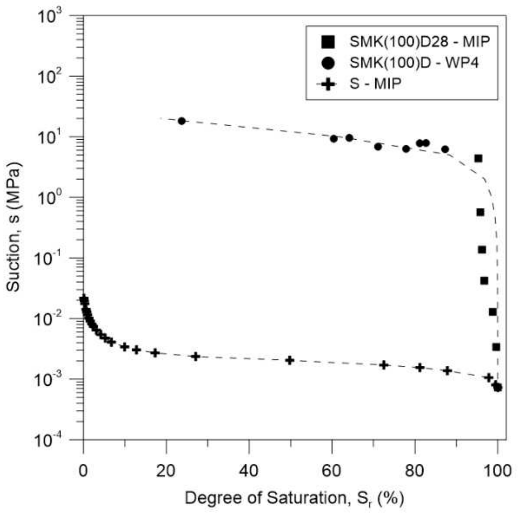

Figure 4. SWRC of treated and untreated sand.

REFERENCES

Anagnostopoulos, C. A. and Sapidis, G. (2017). Mechanical behaviour of epoxy resin-grouted sand under monotonic or cyclic loading. *Géotechnique Letters*, 7(4): 298-303. https://doi.org/10.1680/jgele.17.00063.

Bakharev, T. (2004). Resistance of geopolymer materials to acid attack. *Cement and Concrete Research*, 35(4): 658-670. https://doi.org/10.1016/j.cemconres.2004.06.005.

Consoli, N. C., Cruz, R. C., and Floss, M. F. (2011). Variables controlling strength of artificially cemented sand: influence of curing time. *Journal of Materials in Civil Engineering*, 23(5): 692-696. https://doi.org/10.1061/(ASCE)MT.1943-5533.0000205.

Fraccica, A., Spagnoli, G., Romero E., Arroyo, M., and Gómez, R. (2022). Exploring the mechanical response of low-carbon soil improvement mixtures. *Canadian Geotechnical Journal*, 59(5): 726-742. https://doi.org/10.1139/cgj-2021-0087.

Kolovos, K.G., Asteris, P.G., Cotsovos, D.M., Badogiannis E., and Tsivilis, S. (2013). Mechanical properties of soilcrete mixtures modified with metakaolin. *Construction and Building Materials*, 47: 1026–1036. https://doi.org/10.1016/j.conbuildmat.2013.06.008

Rios, S., Ramos, C., Viana Da Fonseca, A., Cruz, N., and Rodrigues, C. (2017). Mechanical and durability properties of a soil stabilised with an alkali-activated cement. *European Journal of Environmental and Civil Engineering*, 23(2): 245-267. https://doi.org/10.1080/19648189.2016.1275987.

Romero, E. (1999). *Characterisation and thermo-hydro-mechanical behaviour of unsaturated Boom clay: an experimental study*. PhD thesis. Universitat Politècnica de Catalunya (Spain).

Salvatore, E., Modoni, G., Mascolo, M.C., Grassi, D. and Spagnoli, G. (2020). Experimental evidences on the effectiveness and applicability of colloidal nanosilica grouting for liquefaction mitigation. *Journal of Geotechnical and Geoenvironmental Engineering*, 146(10):04020108. https://doi.org/10.1061/(ASCE)GT.1943-5606.0002346.

Spagnoli, G., Romero E., Fraccica, A., Arroyo, M., and Gómez, R. (2022). The effect of curing conditions on the hydro-mechanical properties of a metakaolin-based soilcrete. *Géotechnique*, 72 (5): 455–469. https://doi.org/10.1680/jgeot.20.P.259.

Verdolotti, L., Iannace, S., Lavorgna, M. and Lamanna R (2008). Geopolymerization reaction to consolidate incoherent pozzolanic soil. *Journal of Materials Science*, 43: 865–873. https://doi.org/10.1007/s10853-007-2201-x.

Yaghoubi, M., Arulrajah, A., Disfani, M.M., Horpibulsuk, S., Bo, M.W. and Darmawan, S. (2018). Effects of industrial by-product based geopolymers on the strength development of a soft soil. *Soils and Foundations*, 58: 716-728. https://doi.org/10.1016/j.sandf.2018.03.005.

Zhang, M., Guo, H., El-Korchi, T., Zhang, G., and Tao, M. (2013) Experimental feasibility study of geopolymer as the next-generation soil stabiliser. *Construction and Building Materials*, 47: 1468–1478. https://doi.org/10.1016/j.conbuildmat.2013.06.017.