Use of Alkali-Activated Fly Ashes for Soil Treatment

Enza Vitale[1(✉)], Giacomo Russo[1,2], and Dimitri Deneele[3,4]

[1] Department of Civil and Mechanical Engineering, University of Cassino and Southern Lazio, Via G. Di Biasio 43, 03043 Cassino, Italy
e.vitale@unicas.it

[2] Department of Earth Science, Environment and Resources, University of Naples "Federico II", Via Vicinale Cupa Cinthia 21, 80126 Naples, Italy

[3] LUNAM, IFSTTAR, Institut Français des Sciences et des Technologies des Transports, de l'Aménagement et des Réseaux, BP 4129, Route de Bouaye, 44332 Bouguenais, France

[4] Institut des Matériaux Jean Rouxel (IMN), Université de Nantes, CNRS, 2 rue de la Houssinière, BP 32229, 44322 Nantes Cedex 3, France

Abstract. The use of alkali-activated fly ashes (AAFA) to improve engineering properties of clayey soils is a novel solution, alternative to the widely diffused improvement based on the use of traditional binders such as lime and cement. An experimental investigation on chemo-physical evolution of alkali-activated binders and their use for soil improvement has been developed. Treated samples were prepared by mixing soil and fly ash with water and alkaline solution and dynamically compacted. Mechanical behaviour has been investigated by means of triaxial tests performed on treated samples compacted at optimum water content. Addition of alkali activated binder increased stiffness and shear strength of treated samples, whose extent depends on binder content and curing time.

Keywords: Soil improvement · Fly ash · Alkali-activated binders · Mechanical behaviour · Multi-scale analysis

1 Introduction

The use of novel and efficient binders for improving the engineering properties of natural soils not suitable for construction purposes is a promising issue for geotechnical applications in terms of sustainability since it reduces the carbon footprint and allows reusing secondary by-products such as artificial pozzolans. These by-products can be involved in soil improvement as cementing agents if properly activated, inducing a mechanical improvement of soils. Furthermore, recycling of waste materials such as by-products from industrial process to synthesize a new binder favors a closed loop of material use, which minimizes waste generation and reduces production costs.

It is widely accepted that use of traditional binders such as lime and cement has a strong impact on the physical and hydro-mechanical properties of soils as a result of reactions taking place after the treatment (Uddin et al. 1997; Miura et al. 2001; Boardman et al. 2001; Vitale et al. 2016a; Vitale et al. 2016b; Vitale et al. 2017a;

© Springer Nature Switzerland AG 2020
F. Calvetti et al. (Eds.): CNRIG 2019, LNCE 40, pp. 723–733, 2020.
https://doi.org/10.1007/978-3-030-21359-6_77

Guidobaldi et al. 2017; Guidobaldi et al. 2018; Vitale et al. 2019). A sustainable alternative to the use of ordinary stabilising agents for soil improvement is given by alkali activated binders (Wilkinson et al. 2010; Vitale et al. 2017b; Coudert et al. 2019). Alkali activated binders are formed by alkaline activation of an aluminosilicate source, containing precursor materials like artificial pozzolans (e.g., fly ash, silica fume, steel sludge), which react with an alkaline solution (i.e. sodium hydroxide, sodium silicate). The reaction mechanism is promoted by the alkaline solution, which enables the dissolution of the aluminosilicate source (precursor) and the subsequent precipitation of gel phases, which condense in a three-dimensional aluminosilicate network (Duxon et al. 2007; Provis and van Deventer 2014).

Experimental research about the use of alkali activated binders in soil improvement is still limited (Sargent 2014); nevertheless, recent studies highlight the relevant potential of novel binders for geotechnical purposes. Cristelo et al. (2011) researched the optimum fly ash—based alkaline activated binder for the improvement of soil to be used in rammed earth construction through a parametric analysis using laboratory tests. Rios et al. (2016) compared the mechanical behaviour of samples of sand improved by alkali activated fly ash binder and by cement, highlighting the effective increase of shear strength soil properties induced by alkali activated binders. In the present study, an insight into alkaline activation of a fluidal bed combustion fly ash and its effects on the improvement of mechanical behaviour of a clayey soil have been reported. Chemo-physical evolution of binder has been monitored at increasing curing times by means of X Ray Diffraction and Scanning Electron Microscopy (SEM). Microstructural features and mechanical behaviour have been investigated by means of MIP and triaxial tests performed on treated samples compacted at optimum water content. The effects of binder content and curing time on compaction characteristics and mechanical performance of treated samples have been considered.

2 Materials and Experimental Procedures

The artificial pozzolan used in the present work is a fluidal bed combustion fly ash supplied by a power plant located in Italy. Raw material is composed by vitreous and crystalline phases which include calcium-containing minerals such as anhydrite, calcite and other minerals like quartz and hematite.

Sodium silicate solution was supplied by Woellner (Germany) with a SiO_2/Na_2O mass ratio of 1.7, a density at 20° of 1,55 g/cm^3, a pH value of 12,5 and viscosity at 20° of 450 mPas.

Speswhite kaolin from deposits in the South West of England was the soil considered for the experimental investigation. The specific gravity G_s is 2.6 and its surface area, determined by BET, is 14 m^2/g. The pH value is 4.6 and plastic limit and liquid limit are respectively 32% and 70%, with a plasticity index IP equal to 38%. Soil is manly composed of kaolinite clay minerals with small amounts of quartz and muscovite.

Particle size distributions of Speswhite kaolin and fly ash, obtained from laser particle size analysis, are shown in Fig. 1. Chemical composition of soil and fly ash is reported in Table 1.

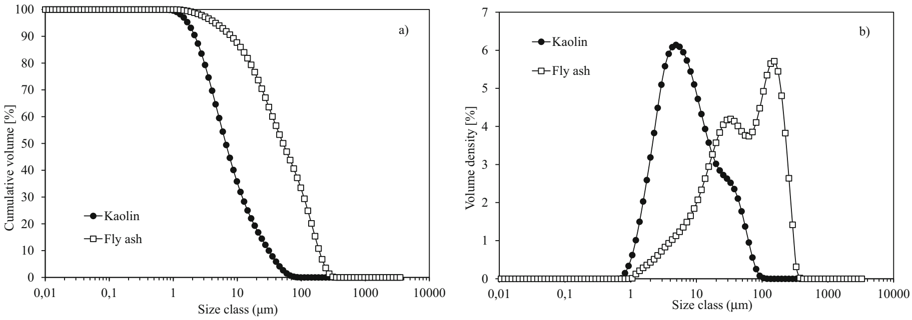

Fig. 1. Cumulative and density particle size distribution of Speswhite kaolin and fly ash

Table 1. Chemical composition of fly ash and Speswhite kaolin.

Chemical composition	Spwt kaolin	Fly ash
SiO_2	53.80	19.80
Al_2O_3	43.75	39.40
CaO	0.02	5.20
K_2O	1.45	1.8

Alkali activated binder (FA100% in the following) was prepared by mixing fly ash, alkaline solution and water in fixed proportions. Alkaline solution/fly ash mass ratio was kept constant and equal to 0.5. Additional deionized water was provided to the system in order to guarantee an effective mixing and a sufficient liquid phase amount for the dissolution of aluminosilicate source. The deionised water/solid ratio, by mass, was selected equal to 0.5. Soil samples treated with alkali-activated binder (KA-FA) were prepared by considering increasing fly ash percentages equal to 10% (sample KFA10%), 15% (sample KFA15%) and 20% (sample KFA20%) by dry weight of solids (soil + fly ash) and alkaline solution/fly ash mass ratio equal to 0.5. Compaction characteristics of not treated sample, clay-fly ash mixtures (sample KFA) and alkali-activated fly ash treated samples (KA-FA) were determined according to ASTM D698-91 using a Standard Proctor test. With reference to compaction curves, optimum initial moisture content was selected for both not treated and treated samples. Treated samples were then sealed in plastic bags and cured at increasing curing times of 24 h, 3, 10 and 60 days before performing microstructural and mechanical tests.

Mineralogical composition of samples was investigated by X Ray diffraction analysis performed on randomly oriented powder using a Brucker AXS D8 Advance Diffractometer with CuKα (λ = 0.154 nm) radiation and a step size of 0.021°. Samples were dehydrated before testing by freeze-drying technique.

Surface state modifications of samples due to alkaline activation process have been examined through Scanning Electron Microscopy by using Hitachi SU5000 microscope. Raw and treated samples have been dehydrated by freeze-drying technique. A pre-treatment gold coating has been applied for SEM observations.

MIP tests were performed by a double chamber Micromeritics Autopore III apparatus. In the filling apparatus (dilatometer) samples were outgassed under vacuum and then filled by mercury allowing the increase of absolute pressure up to the ambient one. Using the same unit, the intrusion pressure was than raised up to approximately 200 kPa by means of compressed air. The detected entrance pore diameters range between 134 μm and 7.3 μm (approximately 0.01 MPa–0.2 MPa for a mercury contact angle of 139°). After depressurisation to ambient pressure, samples were transferred to high-pressure unit, where mercury pressure was increased up to 205 MPa following a previously set intrusion program. MIP tests were performed on alkali-activated fly ash treated samples at different binder contents (KA-FA10% and KA-FA20%) and curing times (namely 24 h, 7 and 28 days).

Drained triaxial compression tests have been performed on saturated samples by means of a controlled stress-path triaxial cell. Saturated samples have been isotropically compressed at mean effective stress of 50 kPa and 300 kPa. Deviatoric stages in drained conditions have been then performed at deformation rate of 0.5%/h up to failure. Treated samples have been tested at increasing curing times (namely 24 h, 3, 10 and 60 days) and increasing binder contents (i.e. KA-FA10%, KA-FA15% and KA-FA20%).

3 Results

X Ray diffraction patterns of raw and alkali activated fly ash binder (FA100%) are shown in Fig. 2. Mineralogical composition of fly ashes is modified by alkaline activation process. Consumption of crystalline phases such as anhydrite ($CaSO_4$) and aluminate phases ($MgAl_2O_4$) is evidenced by disappearance of their characteristic peaks after 60 days of curing. Precipitation of new mineralogical phases such as thernadite (Na_2SO_4) seems to be consistent with the release of sulphate from anhydrite dissolution and its subsequent reaction with sodium provided by silicate solution. A broad reflection between 25° and 35° corresponding to new poorly crystalline compounds resulting from alkaline-activation reactions was observed in alkali activated fly ash after 60 days of curing. SEM observations on raw and alkali activated fly ash after 60 days of curing are shown in Figs. 3 and 4. Raw sample is characterised by particles of different shape and size. The vitreous phase is made of some bigger grains (Fig. 3a, b-2, c), spherical particles (Fig. 3b-1) and aggregate of small particles (Fig. 3b-3). A coating of gel hydrates on particles surface is observed at 60 days of curing (Fig. 4a, b). After the treatment, surface state of the sample is clearly modified. Alteration of glassy/vitreous phases induced by the alkaline environment is clearly evidenced by SEM on raw and alkali activated samples at higher magnifications (Figs. 3d and 4d).

Compaction curves of samples treated with increasing fly ash (KFA) and alkali activated fly ash (KA-FA) contents are reported in Fig. 5. For increasing fly ash percentage, compaction characteristics of clay-fly ash mixture are approximately the same at the optimum water content (Fig. 5a). The maximum dry density of not treated and fly ash treated soil is almost constant. A slight dispersion of data is observed for the dry and wet side of optimum (Fig. 5a). Changes of grain size distribution of treated

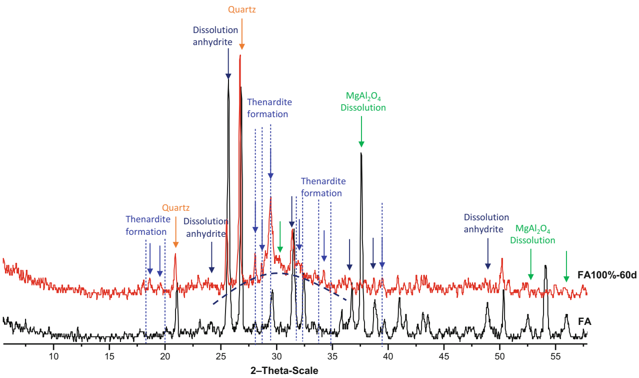

Fig. 2. X Ray diffraction patterns of raw and alkali activated fly ash (FA100%) at 60 days of curing.

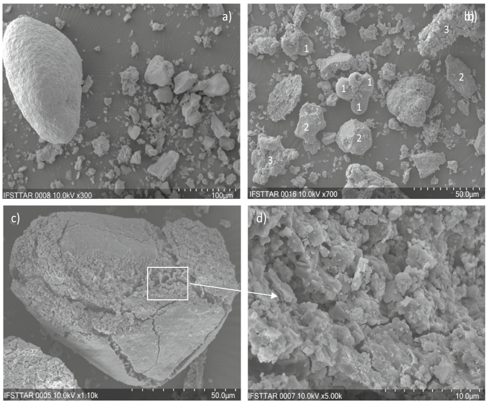

Fig. 3. SEM observations of raw fly ash.

samples induced by addition of increasing fly ash contents (up to 20%) does not relevantly affects compaction efficiency at the optimum. Soil matrix is ruled by fine grained particles; the space between coarser particles is filled with the clay fraction that form a relatively uniform matrix.

A different behavior is observed for samples treated with increasing alkali-activated fly ash contents (Fig. 5b). Compaction curves show a reduction of optimum water content and a relevant increase of maximum dry density as binder content increases. The viscosity of alkaline solution plays a key role in improving compaction characteristics of treated soil, acting as a lubricant among soil particles and favoring therefore a higher compaction efficiency.

Microstructural features of samples treated with alkali-activated fly ash compacted at optimum water content have been investigated by means of MIP. Cumulative intruded void ratios and pore size distributions of samples treated with 10% and 20% of binder (KA-FA10% and KA-FA20%) at 24 h of curing are shown in Fig. 6. A decrease

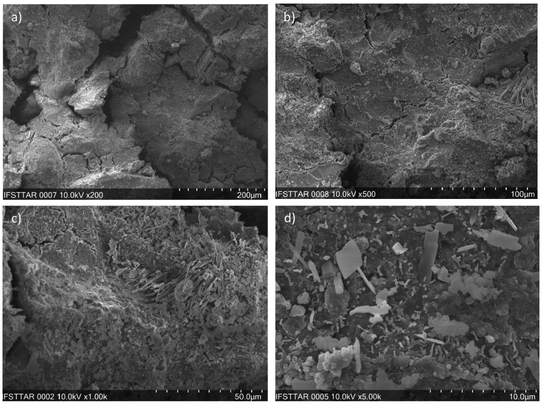

Fig. 4. SEM observations of alkali activated fly ash at 60 days of curing.

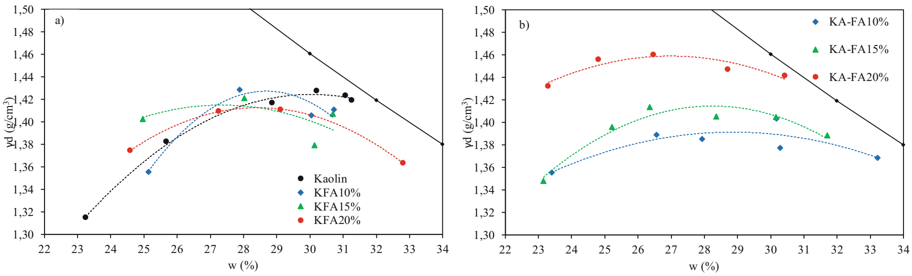

Fig. 5. a) Compaction curves of soil treated with increasing fly ash contents; b) Compaction curves of soil treated with alkali activated fly ash at increasing binder contents

of cumulative intruded void ratio is observed as the binder content increases, which is consistent with a reduction of porosity of the sample. Pore size distributions are characterised by modal diameter pore size ranging between 0.04 μm–0.4 μm. Addition of alkali-activated fly ash does not modify the modal pore size of treated samples and the frequency of pores in the small diameters. Whereas, a decrease of frequency in the largest pore range (i.e. entrance pore diameter >0.5 μm) is observed for higher binder content.

MIP results on alkali-activated fly ash treated samples (KA-FA20%) as function of curing time are shown in Fig. 7. Cumulative intruded void ratio decreases as curing time increases, highlighting a gradual overall reduction of porosity over time (Fig. 7a). Pore size distribution showed a decrease of frequency of main pore entrance diameters and an increase of frequency of pores entrance diameters in the small range (between 0.008 μm–0.02 μm). Modal pore size is shifted towards smaller sizes as a consequence of formation of a well-connected network of gel hydrates over time, which increases small pores frequency and contributes to the progressive decrease of main pore entrance. Frequency of large pores (i.e. pore entrance diameter 1 μm–80 μm) is not affected by curing condition.

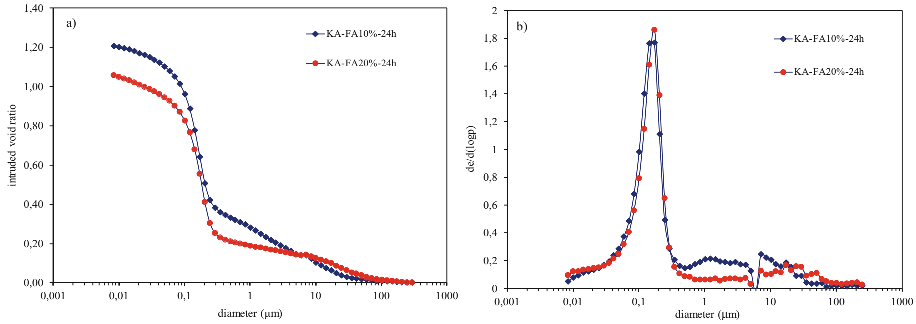

Fig. 6. a) Cumulative intruded volumes and b) Pore size distributions of samples treated with 10% and 20% of binder at 24 h of curing

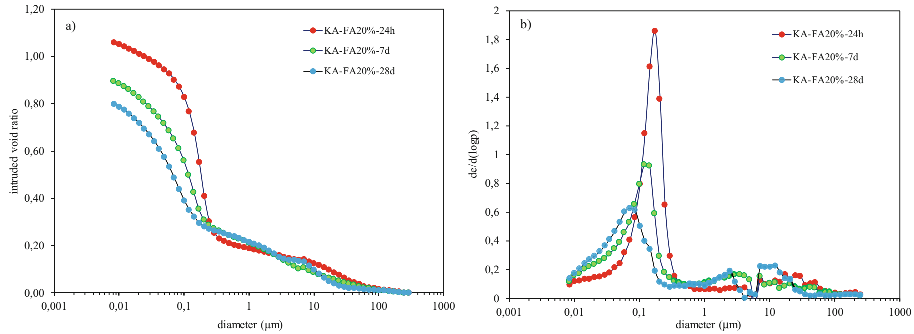

Fig. 7. a) Cumulative intruded volumes and b) Pore size distributions of samples treated with 20% of binder at increasing curing time

Results of triaxial drained compression tests on raw kaolin and alkali activated binder treated samples (KA-FA20%) at increasing curing times (namely 24 h, 3, 10 and 60 days) are reported in Fig. 8a.

A ductile and contractile behaviour is observed for kaolin, whereas treated samples exhibit an overall increase of stiffness and shear strength. The improvement of the mechanical behaviour of treated sample is due to bonding effects induced by precipitation of gel phases resulting from development of alkaline activation process. As shown in Fig. 8b, both peak resistance (q_{peak}) and end-of-test resistance (q_{eot}) are increased as curing time increases. Precipitation of cementitious compounds, whose amount increases over time, is responsible of brittle and dilative behaviour upon shearing. Effects of binder content on mechanical response of treated samples are highlighted by stress-strain curves after 10 days of curing reported in Fig. 9. As showed in Fig. 9a, increasing binder content results in a progressive increase of stiffness and shear strength, coupled with a transition from contractile to dilative behaviour of samples treated with higher binder content. FA-KA20% treated sample showed a stress-strain curve typical of cemented materials (Rios et al. 2014, 2016), with a peak stress attained at low strain levels, followed by an abrupt strain softening due to bonds

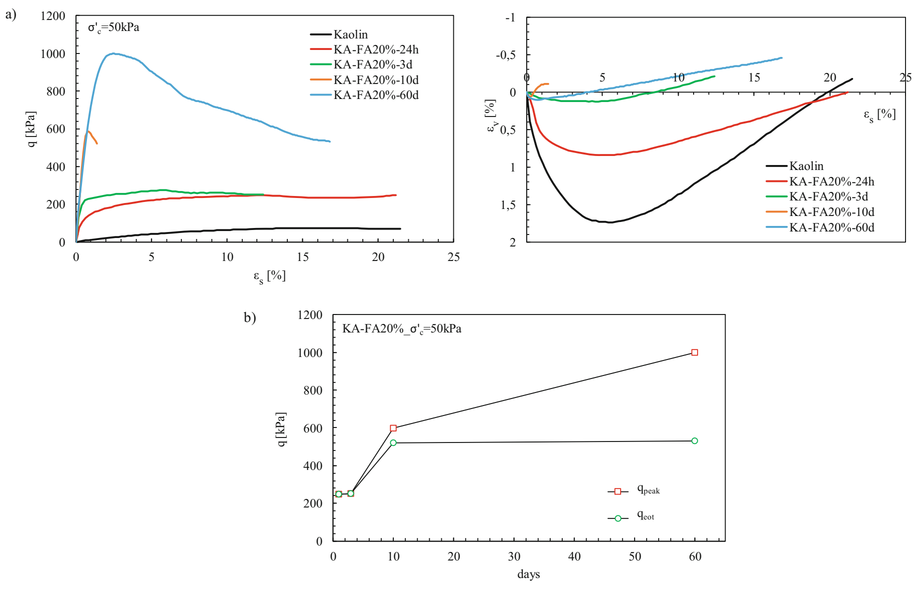

Fig. 8. a) Triaxial results: stress-strain curves of raw and 20% treated samples (KA-FA20%) at increasing curing time b) Evolution of peak resistance (q_{peak}) and end-of-test resistance (q_{eot}) of 20% treated samples (KA-FA20%) as a function of curing time

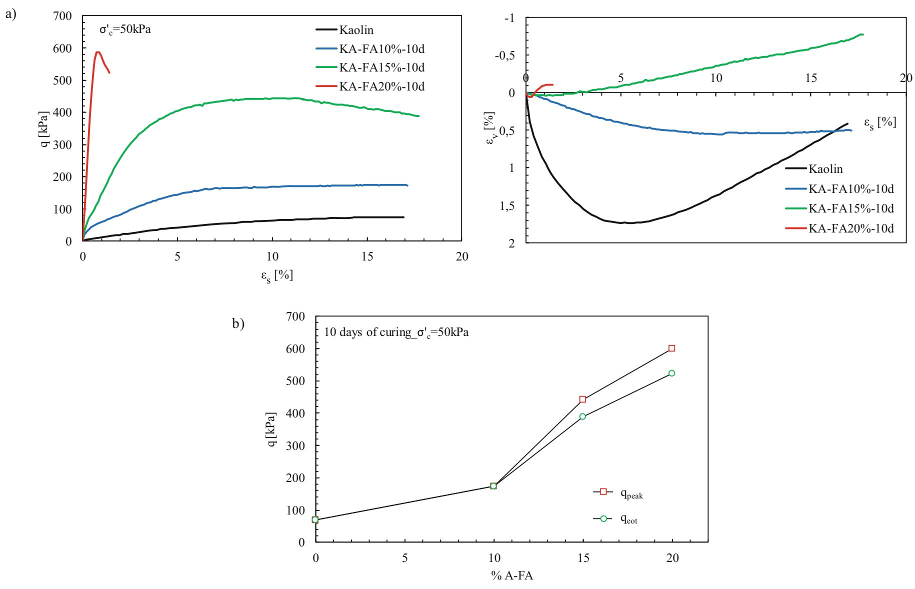

Fig. 9. a) Triaxial results: stress-strain curves of raw and binder treated samples at 10 days of curing b) Evolution of peak resistance (q_{peak}) and end-of-test resistance (q_{eot}) as function of binder content after 10 days of curing.

degradation. The evolution of peak resistance (q_{peak}) and end-of-test resistance (q_{eot}) as function of binder content is reported in Fig. 9b. The amount of new mineralogical phases formed in the system, proportional to the amount of added binder, affects the mechanical response of treated samples. The strength increase resulting from treatment with alkali-activated fly ash can be quantified in terms of Mohr-Coulomb failure criterion, namely from the resulting strength parameters ϕ' (friction angle) and c' (cohesion). Mohr semi-circles of failure at different effective confining stresses and failure envelopes of not treated and 20% treated samples at 10 days of curing are shown in Fig. 10. Addition of binder leads to a relevant increase of cohesion of treated soil. The values of cohesion range from 3,11 kPa for raw soil to 181,7 kPa for treated samples after 10 days of curing. Conversely, no evolution of the friction angle is detected after the treatment (φ' = 22,9° for not treated soil and φ' = 21,2° for treated soil).

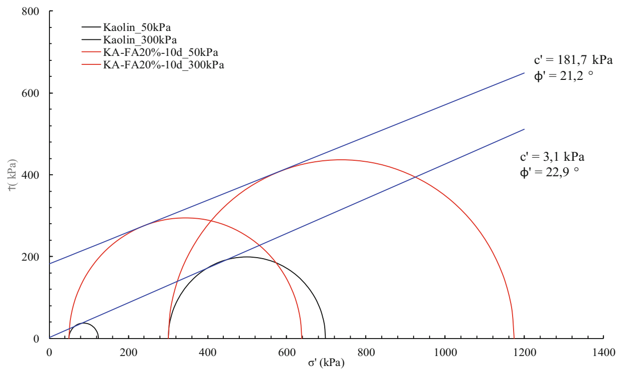

Fig. 10. Mohr circles of not treated and 20% treated soil (KA-FA20%) at 10 days of curing

4 Conclusions

In the present study, an experimental multiscale investigation on the effects induced by alkali activated binders on microstructural features and mechanical behavior of a clayey soil has been developed. Test results on chemo-physical evolution of alkali activated fly ash binder highlighted a high reactivity of binder to promote dissolution of ash particles and formation of alumino-silicate gel with cementitious properties. The addition of alkali activated fly ash to a clay soil improves compaction characteristics of treated samples leading to higher dry unit weight as binder content increases as a consequence of the lubricant effect induced by the high viscosity of alkaline solution. From a microstructural point of view, the progressive increase of maximum dry density of samples treated with higher binder contents resulted in reduced size of larger pores and total porosity, as detected by MIP results. At increasing curing time, the progressive dissolution of ash particles and corresponding precipitation of network of gel phases due to alkaline activation process resulted in a reduction of porosity of treated samples over time with an increase of small pores frequency and a progressive decrease of main pore entrance. At volume scale of the sample, triaxial tests highlighted the effectiveness of the binder to promote the improvement of mechanical behavior of treated soil as a result of bonding effect induced by new cementitious phases formed. An increase of stiffness and shear strength was observed since the very short term, whose extent depends on curing time and binder content. The interpretation of results in terms of shear strength parameters evidenced a relevant increase of cohesion of treated sample with no significant effects on the friction angle value.

References

ASTM D698-91 1997 Test method for laboratory compaction characteristics of soil using standard effort (12,400ft-lbf/ft3 (600kN-m/m^3). Annual Book of ASTM Standard, 04.08. Soil and Rock (II), ASTM International West Conshohocken, 77–84

Boardman DI, Glendinning S, Rogers CD (2001) Development of stabilization and solidification in lime-clay mixes. Géotechnique 50:533–543

Coudert E, Paris M, Deneele D, Russo G, Tarantino A (2019) Use of alkali activated high-calcium fly ash binder for kaolin clay soil stabilisation: physicochemical evolution. Constr Build Mater 201:539–552

Cristelo N, Glendinning S, Teixeira Pinto A (2011) Deep soft soil improvement by alkaline activation. Proc Inst Civ Eng – Ground Improv 164:73–82

Duxon P, Fernàndez-Jiménez A, Provis JL, Lukey GC, Palomo A, van Deventer JSJ (2007) Geopolymer technology: the current state of the art. J. Mat. Sci. 42:2917–2933

Guidobaldi G, Cambi C, Cecconi C, Deneele D, Paris M, Russo G, Vitale E (2017) Multi-scale analysis of the mechanical improvement induced by lime addition on a pyroclastic soil. Eng Geol 221:193–201

Guidobaldi G, Cambi C, Cecconi C, Comodi P, Deneele D, Paris M, Russo G, Vitale E, Zucchini A (2018) Chemo-mineralogical evolution and microstructural modifications of a lime treated pyroclastic soil. Eng Geol 245:333–343

Miura N, Horpibulsuk S, Nagagaj TS (2001) Engineering behaviour of cement stabilized clay at high water content. Soils Found 41(5):33–45

Provis JL, van Deventer JSJ (2014) Alkali activated materials: state of the art reports, RILEM TC 224- AAM. Springer, Dordrecht, The Netherlands

Rios S, da Fonseca AV, Baudet B (2014) On the shearing behavior of an artificially cemented soil. Acta Geotech 9(2):215–226

Rios S, Cristelo N, da Fonseca AV, Ferreira C (2016) Structural performance of alkali activated soil ash versus soil cement. J Mater Civ Eng 28(2):04015125

Sargent P (2014) The development of alkali-activated mixtures for soil stabilisation. In: Pacheco-Torgal F, Labrincha J, Leonelli C, Palomo A, Chindaprasit, P (eds.) Handbook of alkali-activated cements, mortars and concretes. Elsevier, Cambridge

Uddin K, Balasubramanian AS, Bergado DT (1997) Engineering behaviour of cement treated Bangkok Soft Clay. Geotech Eng 28(1):89–119

Wilkinson A, Haque A, Kodikara J (2010) Stabilisation of clayey soils with industrial by-products: part A. Proc Inst Civ Eng - Ground Improvement 163(3):149–163

Vitale E, Deneele D, Russo G, Ouvrard G (2016a) Short-term effects on physical properties of lime treated kaolin. Appl Clay Sci 132–133:223–231

Vitale E, Cecconi M, Croce P, Deneele D, Pane V, Russo G, Vecchietti A (2016b) Influence of pore water chemistry on hydraulic conductivity of kaolinite suspensions. VI Italian Conference of Researchers in Geotechnical Engineering – CNRIG2016, Procedia Engineering 2016 158:81 – 86

Vitale E, Deneele D, Paris M, Russo G (2017a) Multi-scale analysis and time evolution of pozzolanic activity of lime treated clays. Appl Clay Sci 141:36–45

Vitale E, Russo G, Dell'Agli G, Ferone C, Bartolomeo C (2017b) Mechanical behaviour of soil improved by alkali activated binders. Environ 4(4):80

Vitale E, Deneele D, Russo G, De Sarno D, Nicotera MV, Papa r, Urciuoli G (2019) Chemo-mechanical behavior of lightweight cemented soils. Acta Geotech. https://doi.org/10.1007/s11440-019-00797-8