

Scientific paper

Influence of Alkali Cation on the Mechanical Properties and Durability of Fly Ash Based Geopolymers

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Abstract

This research has provided information about the influence of alkali cations (Na^+ and K^+) on the mechanical properties and durability of fly ash based geopolymers. The results have shown that alkali cations have a strong influence on the mechanical properties of fly ash based geopolymers. K-geopolymers generally reach a higher value of compressive strength in comparison to Na- geopolymers. On the other hand, microstructure and phase composition of fly ash based geopolymers are not influenced by the nature of alkali cations. The ratio of main gel structure forming elements is practically not affected by the nature of alkali cations. Durability of fly ash based geopolymers in different aquatic environments is greatly dependent on the choice of alkali cations. Na- geopolymers are generally more resistant in water and aggressive environments than the K-geopolymers. The best durability of fly ash based geopolymers was observed in sea water.

Keywords: Geopolymerization, fly ash, alkali metal, durability, compressive strength, hydrolytic stability

1. Introduction

It has been hypothesized that natural geological transformations which occur in aluminosilicate minerals may be the basis for the structure formation process in a cementitious binder.¹ Other authors² suggested that the reaction of aluminosilicate minerals with strong alkali silicate solutions be called geopolymerization and useful, environmentally friendly, materials produced in such a way - geopolymers (inorganic polymers). The geopolymer synthesis is very similar to the zeolite synthesis but quick hardening of geopolymer paste disables the formation of a well crystallized zeolite structure,³ while the structure of geopolymers is amorphous to semi-crystalline.²

The geopolymerization process includes dissolution of solid aluminosilicate materials in a strong alkaline solution. This is followed by the formation of Si and/or Si–Al oligo-

mers in the aqueous phase and the condensation of the oligomeric species. In such a way, the aluminosilicate network (inorganic polymeric material) is formed. The undissolved solid particles were incorporated in the final geopolymer structure.⁴ During the dissolution process, the breakage of Al–O–Si and Si–O–Si bonds and the liberation of Al and Si occur. Dissolved Al and Si in a strongly alkali solution create aluminate and silicate species, $[\text{Al}(\text{OH})_4]^-$, $[\text{SiO}(\text{OH})_3]^-$ and $[\text{SiO}_2(\text{OH})_2]^{2-}$.^{5,6} Simultaneously, with the dissolution process, the condensation between aluminate and silicate species occurs,⁷ leading to the formation of oligomers in an aqueous phase. Oligomers continue to condense which leads to the formation of large networks – the gel phase. After the rearrangement and reorganization, a three-dimensional aluminosilicate network known as the geopolymer was created.⁸

The aluminosilicate network consists of tetrahedrally coordinated Si^{4+} and Al^{3+} ions which form $[\text{SiO}_4]^{4-}$

and $[\text{AlO}_4]^{5-}$ groups linked by oxygen bridges. The negative charge of these groups is charge-balanced by alkali cations (Na^+ and/or K^+).^{8,9} Empirical formula of silicoaluminate based geopolymers, given in reference², is

$$M_n - [(\text{SiO}_2)_z \text{AlO}_2]_n \cdot w\text{H}_2\text{O} \quad (1)$$

where M is a cation, usually Na^+ , K^+ or Ca^{2+} , n is a degree of polycondensation, $w \leq 3$ and z is 1, 2 or 3.

Geopolymers were widely investigated in the past decade due to their desirable properties which depend on the type and concentration of the alkali silicate solution, the solid / liquid (S/L) ratio and curing conditions (time and temperature).

Although the geopolymerization process is widely investigated, limited research has been focused on the influence of Na^+ or K^+ alkali cations on the properties of fly ash based geopolymers. In this paper we have investigated the influence of alkali cations (Na^+ and K^+) on the mechanical properties and durability of fly ash based geopolymers in different aquatic environments.

2. Experimental

2.1. Materials

The fly ash (mean particles diameter $d_{50}=109 \mu\text{m}$) used for the synthesis of the geopolymer composites was

Table 1: Chemical composition of fly ash.

Content	%
SiO_2	49.45
Fe_2O_3	5.23
Al_2O_3	21.77
TiO_2	0.66
CaO	13.34
Na_2O	0.46
ZnO	4.510^{-3}
MgO	1.29
MnO	0.02
P_2O_5	0.24
K_2O	1.4
LOI*	4.35

*Loss on ignition

Table 2: Granulometric composition of fly ash.

Particle size (μm)	%
<2	1.50
<5	1.90
<20	5.00
<30	7.60
>45	88.3
>60	80.90
>80	73.40
>100	58.10
>150	18.50

supplied from the coal fired power station – Pljevlja. The chemical composition of the fly ash and its granulometric composition are given in Tables 1 and 2, respectively.

2.2. Synthesis procedure

The fly ash was activated using Na or K based alkali silicate solutions. The Na and K based solutions were prepared by mixing Na_2SiO_3 or K_2SiO_3 and 10 M NaOH or 10 M KOH solutions at the mass ratio of 1.5, respectively. Previously it was reported that these parameters gave the best strength of fly ash based geopolymers.¹⁰

Commercial Na and K silicate solutions ($\text{Na}_2\text{O} = 8.5\%$, $\text{SiO}_2 = 28.5\%$, density of 1.39 kg/m^3 ; $\text{K}_2\text{O} = 13.18\%$, $\text{SiO}_2 = 26.38\%$, density of 1.39 kg/m^3) were used. The geopolymer paste was prepared by mixing fly ash with alkali solution at the solid to liquid (S/L) ratio of 1 at which moulding of the paste without additional pressure was possible. The Na/Si, Na/Al, K/Si and K/Al mass ratios in the initial geopolymer mixtures were 0.355, 0.95, 0.44 and 1.18, respectively. The initial M/Si and M/Al ratios (M is Na^+ or K^+ cation) are somewhat higher for K-geopolymer in comparison to the Na-geopolymer because of the somewhat higher content of M_2O in the commercial K silicate solutions in comparison to the Na-silicate solution.

The paste was casted in a closed plastic cylindrical mould, with dimensions of $28 \times 60 \text{ mm}$ and cured for 48 h at 65°C . After that time, the samples were allowed to cool, removed from moulds and left to stay for additional 28 days at ambient temperature before any testing was performed. Before testing, sample surfaces were polished flat and parallel.

2.3. Characterization of Geopolymers

The two control geopolymer samples of each series were tested for compressive strength according to the MEST EN 1354:2011 standard on the HP-400 hydraulic press at room temperature in air (standard test conditions). Before compressive strength testing sample surfaces were polished flat and parallel so that the ratio of height / diameter was approximately 2. The other samples were tested after the tests of chemical resistance in different aquatic environments. Dissolution tests of fly ash in NaOH and KOH were conducted by dissolution of 5 g of fly ash in 100 ml of 10M NaOH and KOH, respectively. A few ml of leachate was sampled at specified times (1–24 h), filtrated and analysed for Al and Si content by Inductively Coupled Plasma – Optical Emission Spectrometer, ICP – OES (Spectro Arcos).

The phase analyses of Na- and K-geopolymers were performed by X-ray powder diffraction (XRPD) technique. XRPD patterns were obtained on a Philips PHILIPS PW 1710 diffractometer using monochromatized $\text{CuK}\alpha$ radiation ($\lambda = 1.54178 \text{ \AA}$) and step-scan mode (2θ range was $4-90^\circ$, 2θ , step 0.02° , time 0.8 s).

Microstructural investigations of Na- and K-geopolymers were carried out using the FEI 235DB focused ion beam system at the National Center for Electron Microscopy at Berkeley, equipped with the EDAX Genesis energy dispersive spectrometer (EDS). The SEM images were recorded with a secondary electron detector (SED). Cross sectioning of Na-geopolymer was carried out using 1000 pA gallium ion beam and microstructural characterization was performed using electron beam at 5 kV operating voltage.

The pore size and the surface area were evaluated by N_2 adsorption/desorption isotherms using ASAP 2020 instrument. Before measuring, degassing of geopolymer samples was performed at 100 °C for 24 h. Surface areas were calculated using the Brunauer–Emmet–Teller (BET) method¹¹ and the pore size distribution and cumulative pore volumes were determined with the Barret–Joyner–Halenda (BJH) method.¹² The t-plot method was used to determine the mesopore surface area (external surface area) and micropore volume.

For the purpose of the durability test, Na- and K-geopolymer samples were immersed in distilled water, sea water, simulated acid rain ($H_2SO_4:HNO_3$ 60:40 wt.%, pH 3) and 1M HCl over the period of 14 weeks. The pH values of solutions were monitored weekly. Compressive strength, mass and dimensions of the samples were recorded before and after tests of chemical resistance.

For the purpose of hydrolytic stability tests, 2 g of Na- or K-geopolymers, granulation below 90 μm , were mixed with 100 ml of distilled water, sea water and acid rain and left in contact for 24 h. After the test, the leachate was separated by the filtration from the insoluble part and analyzed for Al and Si content by ICP–OES (Spectro Arcos).

3. Results and Discussion

3.1. Compressive Strength of Na- and K-geopolymers

The strength of fly ash based geopolymers is strongly dependent on the type of alkali solution. K-geopolymers show higher compressive strength (21.52 MPa) compared to the Na-geopolymers (15.43 MPa), prepared under the same conditions (Fig. 1). The obtained values of compressive strength are lower in comparison to the results reported in the literature because of the considerably higher fly ash particle size ($d_{50} = 109 \mu m$). Granulation of fly ash particles plays a crucial role in the strengthening of fly ash based geopolymers. The previous results have shown that in order to obtain the optimal strength of fly ash based geopolymers (60–66 MPa), 80–90% of fly ash particles must be with size lower than 45 μm .¹³ In this case, chemical aspect (dissolution of reactive phase) gets a great relevance and increases the strength of geopolymers. The results pre-

sented here are obtained using the fly ash which contains 88.3 % particles higher than 45 μm and thus the lower values of compressive strength were obtained.

The difference in strength of Na- and K-geopolymers is not attributed to the different dissolution of fly ash in NaOH and KOH solutions, respectively. According to the dissolution test results (Table 3), it would be expected that Na-geopolymers reach a higher value of compressive strength because more Al and Si is released from the fly ash in NaOH than in KOH solution. A similar effect was observed in a previous work.¹⁴ The difference in dissolution of Al and Si from the starting material in different alkali solutions is associated to the difference in ability of Na^+ and K^+ cations to stabilize silicate species (monomers and dimers) compared to K^+ cations and thus enhance the dissolution of Al and Si from the fly ash.¹⁵ On the other hand, the higher value of compressive strength for K containing geopolymers is attributed to the higher degree of polymerization in a system containing K^+ , compared to those containing Na^+ cations.¹⁶ The possible explanation lies in the difference of alkali cation sizes. Smaller Na^+ cations bind strongly with silicate species present in the alkali solution so that the pair is relatively inert to condensation with another silicate species¹⁷ which results in a lower degree of polymerization and lower compressive strength of the Na-geopolymer compared to the strength of K-geopolymer.

Moreover, K^+ cation accelerates condensation between $Al(OH)_4^-$ and large silicate oligomers and promotes the higher ratio of gel/(undissolved Al-Si surface area), compared to the Na^+ cation.¹⁸ The higher amount of gel in K-geopolymer compared to that containing Na^+ cation is attributed to the higher concentrations of free hydroxide and lower viscosity in K silicate solutions. This facilitates mass transfer to and from the reaction surface resulting in the higher amount of gel in K-geopolymers¹⁹ and a stronger structure.

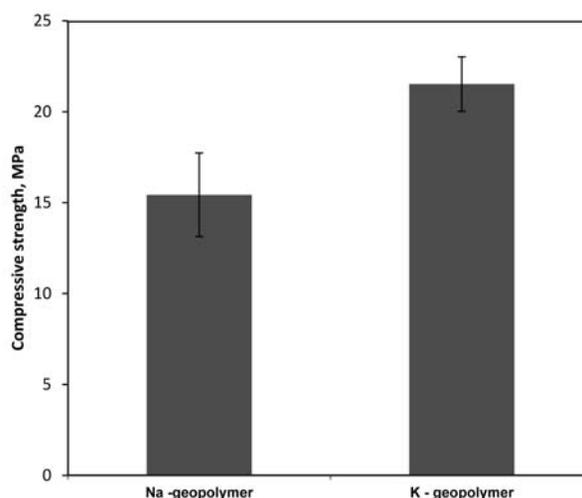


Figure 1: The compressive strength of Na- and K-geopolymers

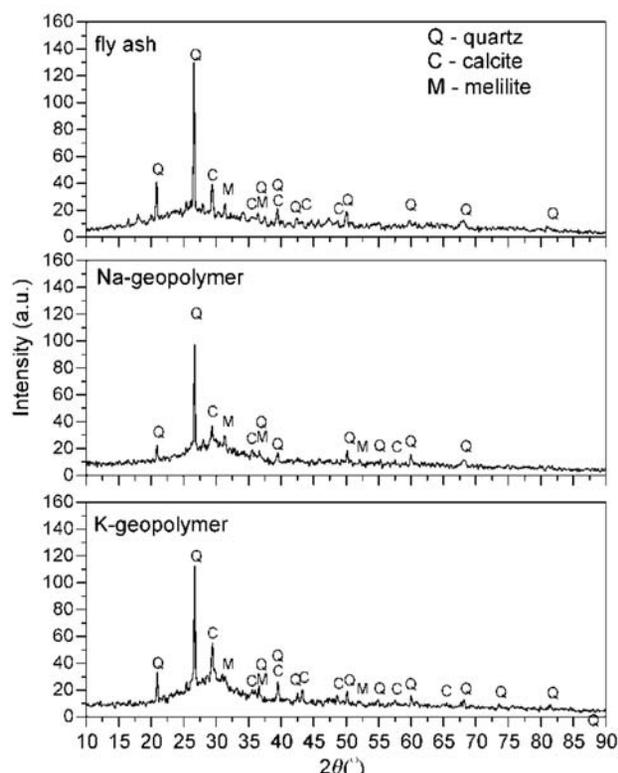
Table 3: The dissolution of Al and Si in 10 M NaOH and 10 M KOH

Time (h)	C(Al), mg L ⁻¹		C(Si), mg L ⁻¹	
	10M NaOH	10 M KOH	10M NaOH	10 M KOH
1	3.25	1.85	6.75	3.28
2	5.9	2.92	14.73	7.15
3	10.28	4.79	23.13	11.10
4	13.53	5.93	36.20	16.38
5	20.61	6.35	43.68	20.12
10	26.45	15.68	55.16	48.72
15	29.3	23.8	63.81	60.21
20	33.89	26.25	71.31	79.31
24	49.50	38.21	85.25	88.98

3. 2. X-ray powder diffraction analysis

X-ray powder diffraction patterns of the fly ash and fly ash based Na- and K-geopolymers are shown in Fig. 2.

The XRDP patterns of fly ash based Na- and K-geopolymers prepared at $S/L=1$, $C(MOH) = 10$ M and $M_2SiO_3/MOH = 1.5$, (M is Na⁺ or K⁺ cation) show that they are mainly X-ray amorphous, as was expected. Some undissolved components such as quartz, calcite and melilite remained from unreacted fly ash. There were no new crystalline phases formed as a result of geopolymerization, no matter which alkali cation was included in the reaction.

**Figure 2:** XRPD patterns of the fly ash, Na-geopolymer and K-geopolymer

3. 3. SEM-EDS Microanalysis

Results of SEM – EDS analysis of Na- and K-geopolymers are presented in Fig. 3 (a) and 3 (b), respectively. Microstructures of both, Na- and K-geopolymers are characterized by the presence of amorphous aluminosilicate gel phase and unreacted fly ash particles embedded in the geopolymeric structure. Results of EDS analyses of amorphous gel phases of Na- and K- geopolymers have shown that the dominant elements present in the gel phases are Si, Al, O. Moreover, Na is present in the amorphous gel phase of Na –geopolymer while K is a dominant alkali metal in the case of K-geopolymer. A certain quantity of Ca, originated from the fly ash, is present in both Na- and K –geopolymers.

Previous investigations have shown that the strength of geopolymers is determined by the ratios of Si/Al and alkali metal to Si or Al.^{20,21}

The content of the main structure forming elements in the gel phases of the both Na- and K-geopolymers and their ratios are summarized in the in Table 4. The results have shown that the alkali metal cation has no significant influence on the, M/Al and M/Si ratios (M is alkali metal: Na or K). These ratios are slightly higher in the K-geopolymers than in the Na-geopolymer. This is in accordance with the lower dissolution of Al and Si from fly ash in the KOH compared to the NaOH (table 3). Moreover, the ratio of Si/Al is slightly higher in the K-geopolymer compared to the Na-geopolymer which is in agreement with previous observations where the increase of the Si/Al ratio leads to a higher compressive strength.²¹

Table 4: The elements content and their ratios (wt%) in the gel phase of Na- and K-geopolymers

Ratio	Na – geopolymer	K – geopolymer
Na/Al	1.2	–
K/Al	–	1.52
Si/Al	2.8	3.0
Na/Si	0.42	–
K/Si	–	0.51

3. 4. Porosity of Na- and K-geopolymers

Adsorption/desorption isotherms of Na- and K-geopolymers obtained by nitrogen adsorption are presented in Fig. 4 (a) and 4 (b), respectively. According to the IUPAC (International Union of Pure and Applied Chemistry) classification²² both, the Na- and K-geopolymer samples show type IV isotherms. The existence of hysteresis loops on adsorption/desorption isotherms of Na- and K-geopolymers indicate that both are mesoporous materials. The hysteresis loop originates from the difference in adsorption and desorption processes, which is attributed to the capillary condensation taking place in mesopores. The isotherms for Na- and K-geopolymers have the same sha-

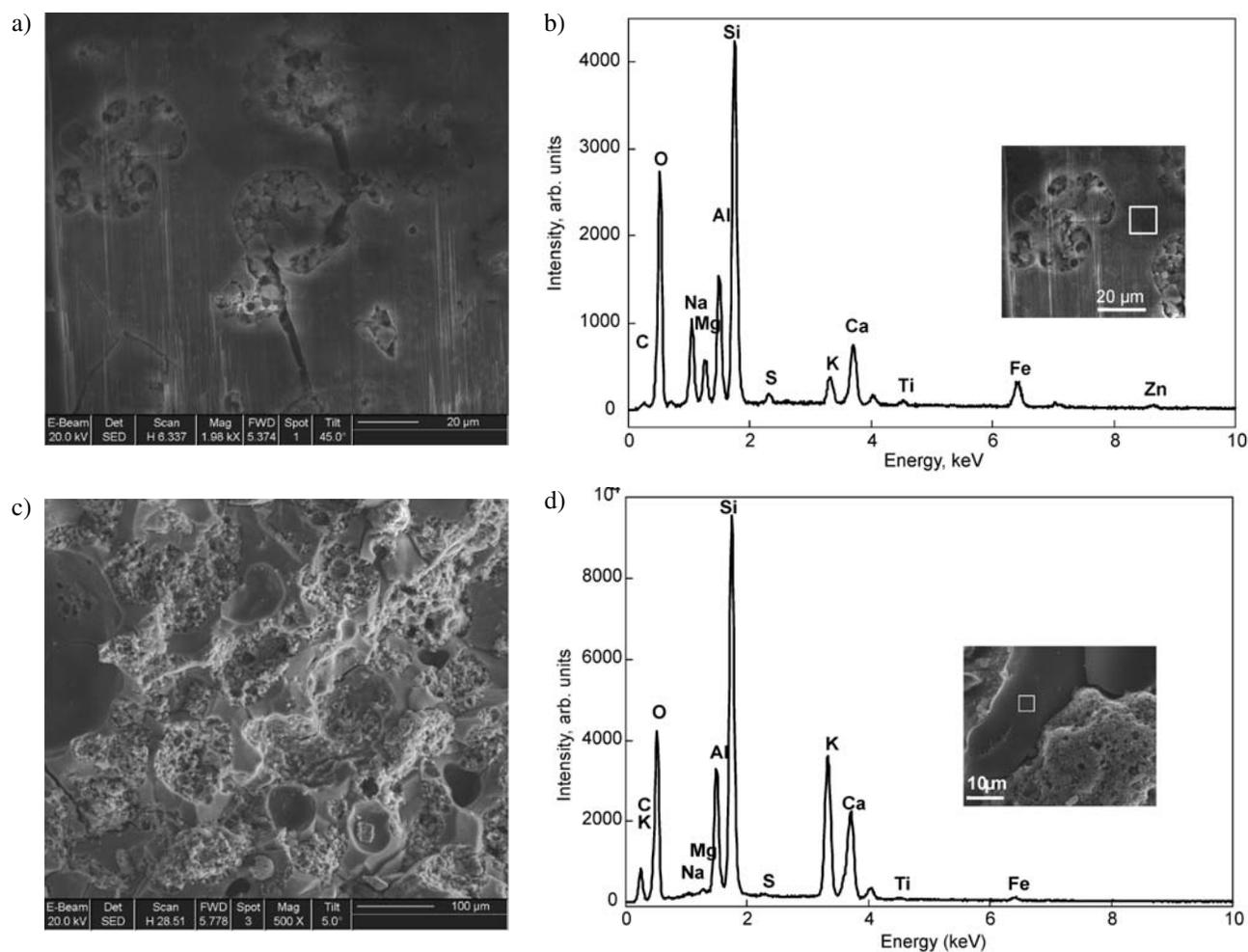


Figure 3: SEM-EDS of (a,b) Na- and (c,d) K-geopolymers

pes and almost the same closure points of hysteresis loops ($P/P_0 = 0.446$ for Na- geopolymers and $P/P_0 = 0.449$ for K-geopolymers). Both closure points are above the relative

pressure of $P/P_0 = 0.4$ which is also associated with capillary condensation in mesopores.²³ The results of porosity investigations are given in Table 5. It is evident that

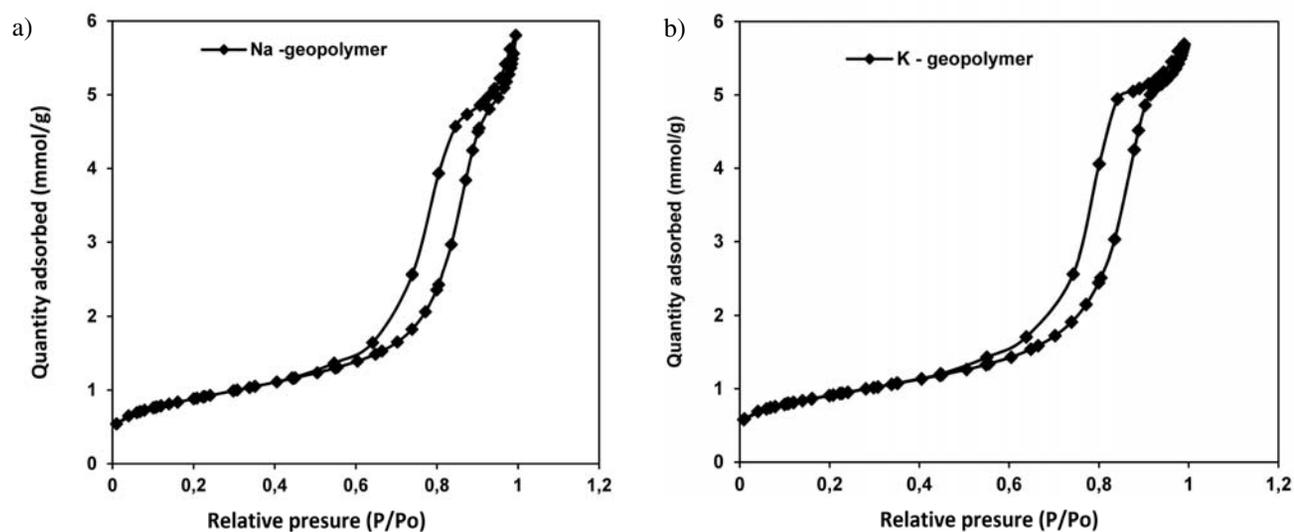


Figure 4: Adsorption/desorption isotherms of Na-geopolymers (a) and K-geopolymers (b)

Na- and K- geopolymers show almost the same porosity, which indicates that the alkali cation has no influence on the porosity of fly ash based geopolymers. Practically there is no difference between Na- and K-geopolymers in cumulative pore volume (V_{cum}), while there is little difference in average pore width (D_p), BET surface (S_{BET}) and mesopore surface (S_{mes}). Therefore, the difference in compressive strength of Na- and K-geopolymers is not influenced by their porosity.

Table 5: Porosity characteristics for Na- and K-geopolymers

Type of geopolymer	S_{BET} $m^2 g^{-1}$	S_{mes} $m^2 g^{-1}$	V_{cum} $cm^3 g^{-1}$	D_p nm
Na-geopolymer	70.89	62.38	0.1952	9.03
K-geopolymer	72.42	60.35	0.1949	8.63

3. 5. Durability of Na- and K-geopolymers in Different Aquatic Environments

Durability of Na- and K-geopolymers is strongly influenced by the nature of the alkali cation. Immersing either Na- or K-geopolymers in distilled water and acid rain, immediately boosts the pH of the highly alkaline solution to 11.4 and 11.7 respectively, due to the mobility of alkali cations in solution. The excess of Na and K ions in the geopolymer mixture remains unreacted after the geopolymerization process and is deposited in geopolymer pores.^{19,24} While in contact with an aquatic environment, the ion exchange between Na^+ or K^+ and H_3O^+ occurs, contributing to the increase of pH.²⁵ The pH values of the sea water and 1M HCl solution after the immersion of geopolymers were 9.3 and 2.6, respectively. The pH values of solutions were monitored weekly and there were no significant changes.

The analyzed samples which were immersed in a 1 M HCl solution have shown the a dramatic deterioration and obvious mass loss during the first week. In the next few weeks, the presence of gelatinous phase in solution was observed, but no additional deterioration was discovered. The formation of a gelatinous phase is a result of the acid attack on the aluminosilicate network and depolymerization process in which the Si–O–Al and Si–O–Si bonds break. Consequently the dealumination and an increase in the amount of silicic acid ions in solution occur.²⁶ Besides, the exchange of H^+ or H_3O^+ ions with alkali cations and neutralization of alkali hydroxide take place, leading to the depolymerization process.²⁷ Both, Na- and K-geopolymers have shown mass losses of 16.7 % and 19.4 %, respectively. Because of obvious deterioration, geopolymer samples after immersion in HCl were not tested for compressive strength.

The results obtained after exposure of Na- and K-geopolymers to distilled water, sea water and acid rain over

a period of 14 weeks indicate strength lose (Fig. 5). Na-geopolymers loss 35.7, 30.3 and 44.6 % strength in distilled water, sea water and acid rain, while the K- geopolymers lose 50.1, 43.5 and 54.0 % of strength, respectively. It is evident that K-geopolymers are characterized by higher strength loss compared to Na-geopolymers, and the best resistance in both cases was achieved in sea water. The satisfactory resistance of fly ash based geopolymers in sea water was also observed by Zhang *et al.*^{28,29} According to the strength loss after immersing of Na- and K-geopolymers in a different aquatic environment they can be arranged as follow: acid rain > distilled water > sea water.

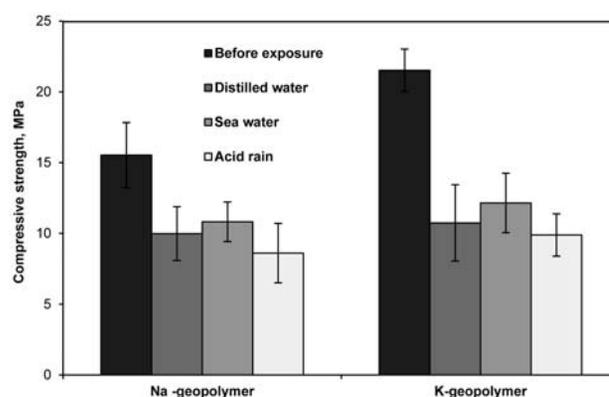


Figure 5: The compressive strength of Na- and K-geopolymers before and after exposure to different aquatic environments

In addition, the mass loss was observed in both Na- and K-geopolymers, after immersion in distilled water and acid rain, while in the case of sea water, mass increase was observed (Table 6). There were no changes in the dimensions of geopolymer samples, no matter which aquatic environment they were exposed to. Almost the same mass increase (1.93% for Na- geopolymers and 2.02% for K-geopolymers) was observed in the case of exposure to sea water. A new layer on the geopolymer surface is probably created, but this statement needs to be further investigated.

The differences between Na- and K-geopolymers are in durability, when they are exposed to different aquatic and acid environments, and these differences are in agreement with the hydrolytic stability of geopolymers. The hydrolytic stability of geopolymers is reflected in the

Table 6: The change in mass of fly ash based geopolymers before and after they were exposed to different aquatic environments (“–“ refers to the decrease of mass, “+” refers to the increase of mass).

	Change in mass (%)	
	Na-geopolymers	K-geopolymers
Distilled water	–2.5	–4.32
Sea water	+1.93	+2.02
Acid rain	–4.14	–6.03

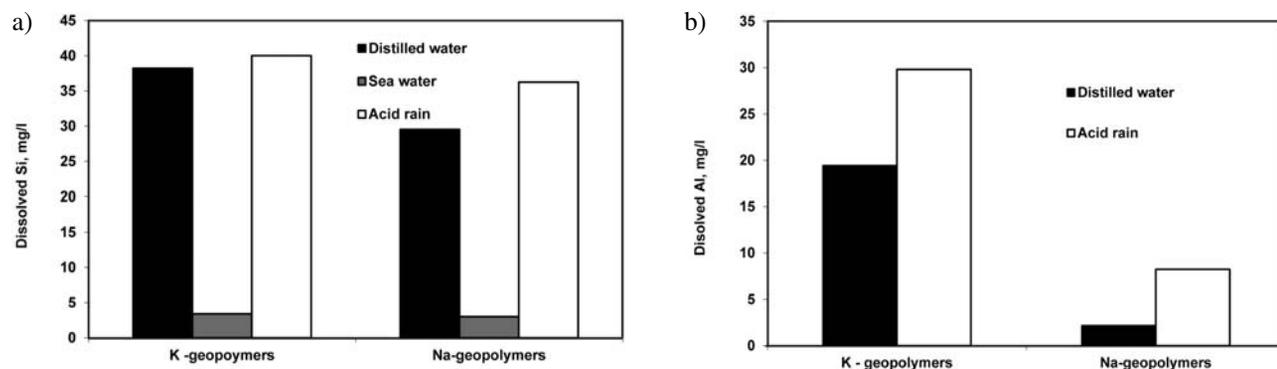


Figure 6: The dissolution of Si (a) and Al (b) from Na - and K - geopolymers in different aquatic environments

degradation of geopolymer structure in aquatic environments^{30,31} and leaching out of constituents of geopolymer structure.

As more Al and Si leach out from the geopolymer matrix, the hydrolytic stability decreases. The results of investigation of hydrolytic stability of Na- and K-geopolymers are given in Fig. 6 (a) and (b), respectively. Both, Na- and K-geopolymers show the highest hydrolytic stability in sea water, i.e. the lowest release of Al and Si was observed in this case. On the other hand, the Na- and K-geopolymers have shown the lowest hydrolytic stability in the acid rain. Moreover, the Si leaches more easily than Al, and the content of Al in sea water was below the detection limit. Generally, K-geopolymers leach more Si and Al in an aquatic and acid environment than Na-geopolymers, indicating a higher destruction of the aluminosilicate network in K-geopolymers, and thus higher strength loss.

4. Conclusions

The results of investigation of the influence of alkali metal cations on the mechanical properties and durability of fly ash based geopolymers in different aquatic environments provide the next conclusions:

- K-geopolymers reach higher compressive strength compared to the Na-geopolymers synthesized under the same conditions. The difference between Na- and K-geopolymers in mechanical properties should not be considered from a standpoint of difference in porosity because it is not influenced by it. Both, Na and K-geopolymers are considered as mesoporous materials.
- The alkali cation has no influence on the microstructure and phase composition of fly ash based geopolymers. Results of X-ray powder diffraction show that Na- and K-geopolymers are mainly X-ray amorphous and therefore only a small amount of undissolved components (quartz, calcite and melilite), remaining from unreacted fly ash, were identified. Their microstructures are characterized

by the presence of amorphous aluminosilicate gel phase and unreacted fly ash particles embedded in the gel phase. The alkali cation has no significant influence on the ratios of the main gel structure forming elements.

- Although K-geopolymers reach higher compressive strength than their Na counterpart, they are characterized by lower durability in different aquatic environments. Both, Na- and K-geopolymers have shown strength loss after immersion in distilled water, acid rain and sea water but generally K-geopolymers show higher strength loss than Na-geopolymers. Moreover, both types of geopolymers are characterized by mass loss after immersion in distilled water and acid rain. On the other hand, they have shown almost the same mass increase after exposure to sea water.
- Alkali metal cations also have an influence on the hydrolytic stability of fly ash based geopolymers in different aquatic environments. The fly ash based geopolymers have shown the best hydrolytic stability, i.e. the lowest dealumination and desilication of aluminosilicate network in the sea water, while the poorest stability was observed in the acid rain.

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Povzetek

Avtorji so raziskali vpliv alkalijskih kationov (Na^+ in K^+) na mehanske lastnosti in obstojnost geopolimerov na osnovi elektrofiltrskega pepela. Rezultati so pokazali, da imajo alkalijski kationi velik vpliv na mehanske lastnosti geopolimerov. K-geopolimeri imajo večjo tlačno trdnost kot Na-geopolimeri. Mikrostruktura in fazna sestava geopolimerov nista odvisni od vrste alkalijskega iona. Razmerje elementov, ki tvorijo gelsko mrežo, je skoraj neodvisno od vrste alkalijskega iona. Obstojnost geopolimerov na osnovi elektrofiltrskega pepela v različnih vodnih medijih je izrazito odvisna od vrste alkalijskega iona. Na-geopolimeri so obstojnejši v vodi in nekaterih agresivnih medijih kot K-geopolimeri. Geopolimeri na osnovi elektrofiltrskega pepela so izkazali največjo obstojnost v morski vodi.