GEOPOLYMERS: INFLUENCE OF ALKALINE ACTIVATOR CATIONS ON EFFLORESCENCE

Novotná M.[1,2], Polonská A.[1], Šídlová M.[1], Perná I.[2]

novotna@irsm.cas.cz

[1]Department of Glass and Ceramics, University of Chemistry and Technology Prague, Technická 5, 166 28 Prague 6.
[2]Department of Material Structure and Properties, Institute of Rock Structure and Mechanics, The Czech Academy of Sciences, V Holešovičkách 41, 182 09 Prague 8.

INTRODUCTION

- Geopolymers are highly heat-resistant, insoluble in water, they can inhibit hazardous materials, and simultaneously achieve high strength.
- **Efflorescence** arises from the reaction of free alkali metal or alkaline earth cations with the surrounding environment.
- Efflorescence is not only unaesthetic but it can also have a negative impact on the structure and properties of materials.
- The formation of efflorescence influences the possibility of using geopolymer in common practice.

STUDY AIM

- Examination of the effect of efflorescence on geopolymer materials
- Measurement of setting time, mechanical properties, and pore diameter
- Determination of intensity and mineralogical composition of efflorescence

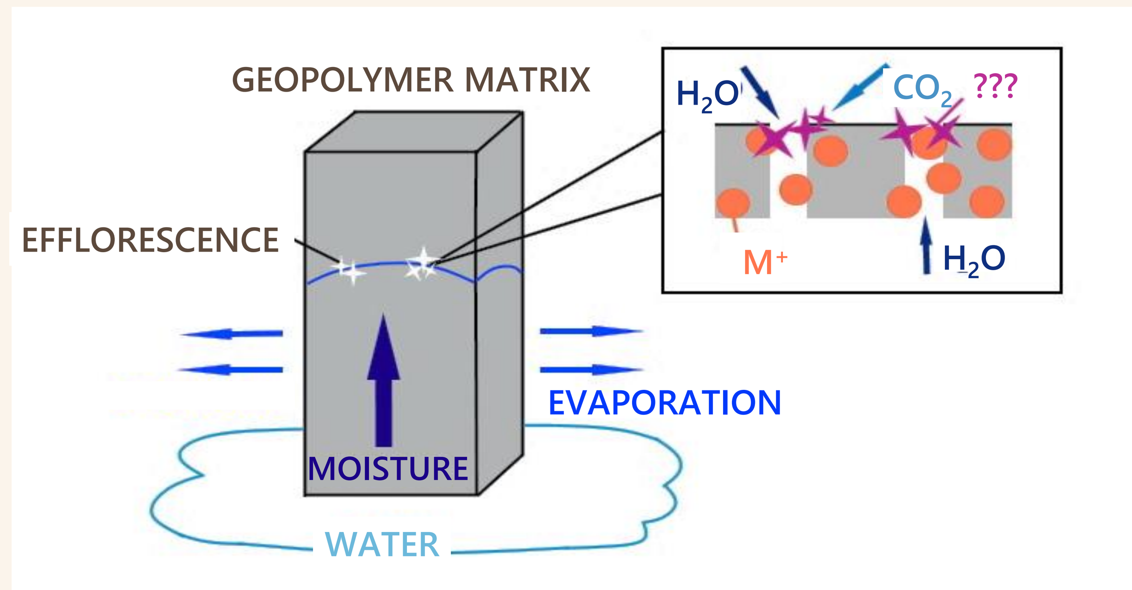
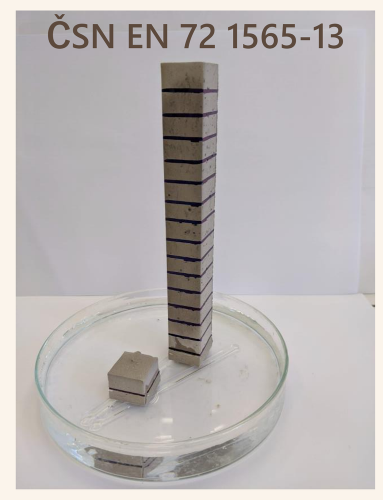


Fig. 1: Illustration of the arising of the efflorescence

METHODS



- X-ray powder diffraction method XRD (Bruker AXS D8 θ-θ).
- X-ray fluorescence analysis XRF (Spectro IQ).
- Setting time – VICAT apparatus (EN UNI 196-3).
- Flexural and compressive strength after 28, 90, 180, 365 days (16 x 2 x 2 cm).
- Porosity – mercury intrusion porosimetry MIP (Autopore 9500, Micromeritics).
- ČSN EN 72 1565-13 – the susceptibility to the formation of efflorescence

MATERIALS

Geopolymer materials were prepared by a standard procedure (mix for 10 min; 7 days covered by a plastic sheet, 21 days air drying).

- **Precursor:** based on metakaolinite
- **Activators:** differ in the type of cations (Li^+, Na^+, K^+), and in the ratio of SiO_2/M_2O.
- **Filler:** chamotte grit

RESULTS

X-ray powder diffraction analysis of the raw materials

- Chamotte contains as **major phases:** mullite and quartz; **minor phase:** cristobalite and **traces:** rutile, anatase and hematite.
- Precursor incorporates about 80 % of the amorphous phase and **minor phases:** quartz, illite, kaolinite, calcite, and mica.

X-ray fluorescence analysis of the raw materials

Sample	Al_2O_3	SiO_2	CaO	Na_2O	K_2O	MgO	Fe_2O_3	SO_3	LOI
Chamotte	38.65	56.00	0.28	0.11	0.72	0.02	1.86	0.24	0.02
Precursor	28.68	44.76	17.31	0.11	0.42	2.96	0.73	0.76	2.71

Tab. 1: Results of the XRF analysis.

Sample preparation

Mix designation	Content [g]			SiO_2/Al_2O_3	M_2O/Al_2O_3	H_2O/M_2O	Filling [%]	W/S
	Precursor	Activator	Chamotte					
Na 1.9	100.0	80.0	180.0	3.7	0.6	18.5	58.5	0.2
K 1.7	100.0	90.0	190.0	3.7	0.6	19.2	59.0	0.2
Li 1.8	100.0	80.0	180.0	3.3	0.4	34.8	61.2	0.2
Li 2.6	100.0	80.0	180.0	3.5	0.4	34.1	60.4	0.2

Tab. 2: Preparation of the geopolymer mixtures.

Mechanical properties (flexural and compressive strength)

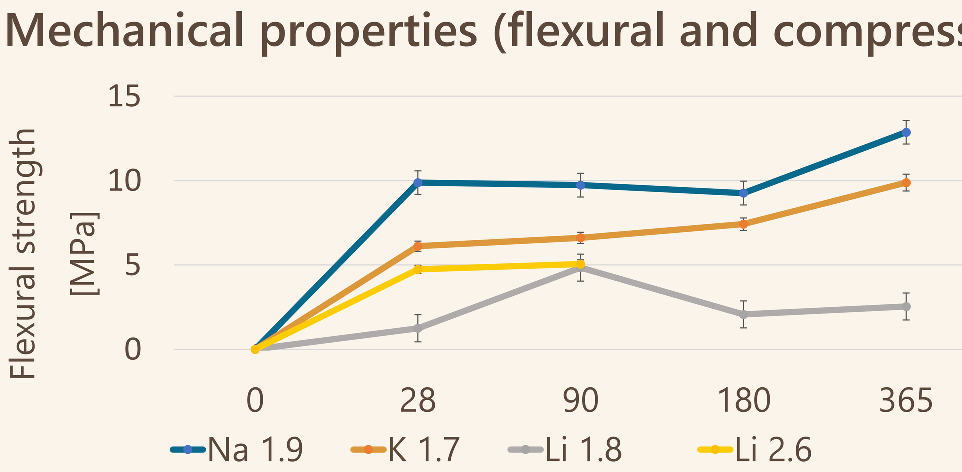


Fig. 2: Flexural strength after 28, 90, 180 and 365 days.

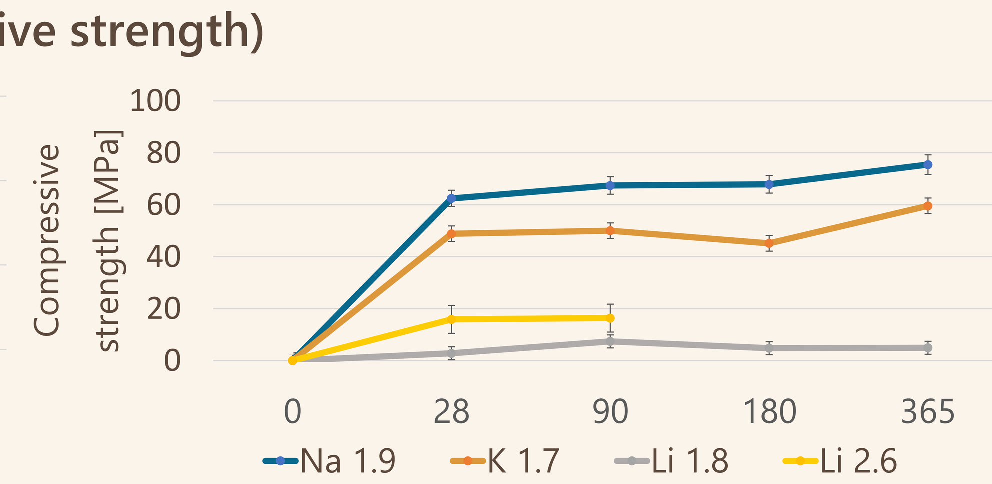


Fig. 3: Compressive strength after 28, 90, 180 and 365 days.

Pore structure

Porosity: Na 1.9 = 18.8 %, K 1.7 = 19.3 %, Li 1.8 = 25.4 %, Li 2.6 = 25.4 %

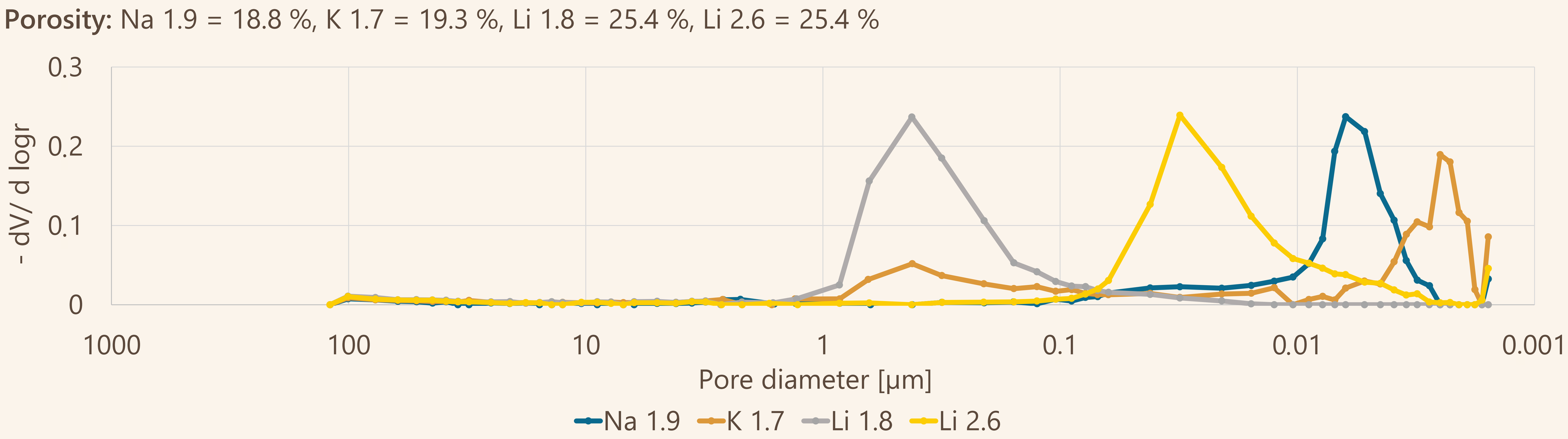


Fig. 4: Pore diameter measured by MIP.

Setting time

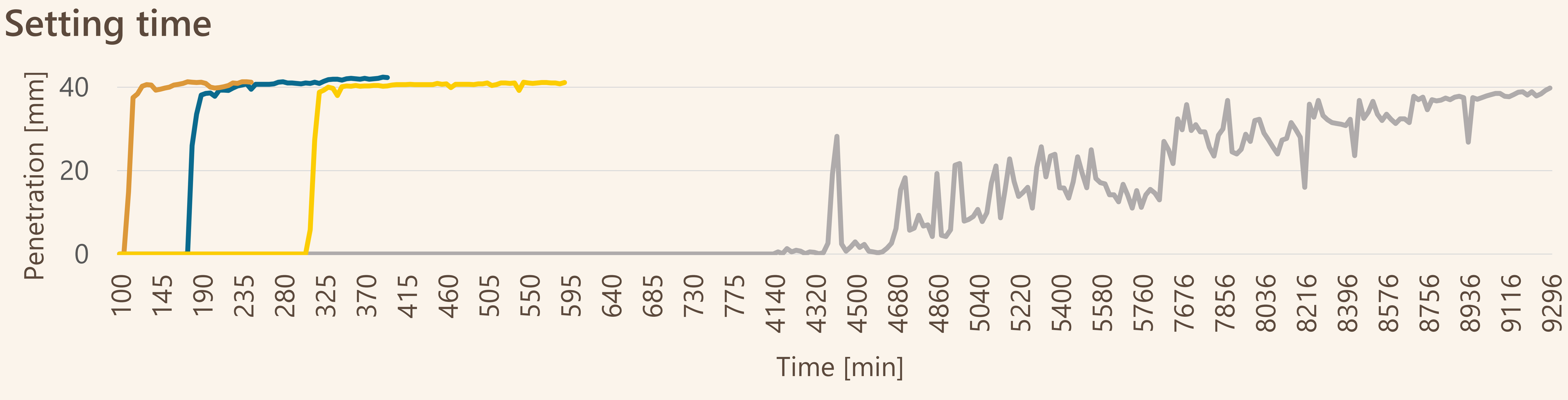


Fig. 5: Vicat setting time.

Visible efflorescence after 28 days in distilled water

Sample	After 7 days in distilled water			After 28 days in distilled water		
	Intensity	Colour	Location	Intensity	Colour	Location
Na 1.9	—	—	—	weak	white	surface
K 1.7	weak	white	surface, edge	weak	white	surface, edge
Li 1.8	medium	white, yellow	surface, edge	medium	white, yellow	surface, edge
Li 2.6	—	—	—	—	—	—

Tab. 3: Evaluation of visible efflorescence after 7 and 28 days in distilled water.

Na 1.9 K 1.7 Li 1.8 Li 2.6

Fig. 6: Photos of efflorescence after 28 days in distilled water.

Na 1.9 K 1.7 Li 1.8 Li 2.6

Fig. 7: Detailed photos of efflorescence taken by microscopy method.

X-ray powder diffraction analysis of the efflorescence

- Li 1.8; Li 2.6 → Li_2CO_3 (zabuyelite)
- Na 1.9 → Na_2CO_3 (natrite)
- K 1.7 → K_2SO_4 (arcanite)

CONCLUSION

- Alkaline activator cations have an obvious impact on the type of efflorescence.
- **Intensity of efflorescence:** Li 2.6 < Na 1.9 < K 1.7 < Li 1.8
- **Mechanical properties (28 days):** Na 1.9 > K 1.7 > Li 2.6 > Li 1.8
- Specimens with lithium cations have the largest porosity, pore radius and the longest setting time.
- It would be appropriate to further investigate the influence of lithium ions on geopolymer materials and to optimize these mixtures.

REFERENCES AND ACKNOWLEDGEMENT

[1] Davidovits J.: Geopolymer Chemistry and Applications, 4th. ed. Institut Géopolymère, Saint-Quentin 2008.

[2] Allahverdi, A. et al.: Methods to control efflorescence in alkali-activated cement-based materials. Handbook of alkali-activated cements, mortars and concretes, 463-483. Woodhead Publishing 2015.

This work was supported by the projects: the grant of Specific university research – grant No. A1_FCHT_2021_006 and the StrategyAV21, activity of the Czech Academy of Sciences, the research program VP23 –City as a Laboratory of Change: Historical Heritage and Place for Safe and Quality Life.