

MICROSTRUCTURE OF GEOPOLYMER MATERIALS BASED ON FLY ASH

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Geopolymer prepared by brown coal fly ash alkali activation contains mainly structures of the types $AlQ^+(4Si)$ and $SiQ^+(3Al)$ and $SiQ^+(2-3Al)$. Any presence of the temporary phase with different composition was not found between geopolymer and aggregate as it is at the concretes from Portland cement. Geopolymer composition is almost the same in the close nearness even in the geopolymer matrix. Elastic modulus evaluated for the mixed geopolymeric and C-S-H phase by means of nanoindentation was found to be $E = 36.1 \pm 5.1$ GPa. Such result is comparable (a little bit higher) to ordinary Portland cement pastes.

INTRODUCTION

When strongly alkali surrounding is effective to aluminosilicate substances like cement, marl, slag, fly ash or heat activated kaolin substances, new materials - geopolymers arise. Their base is two or three dimensioned structure Si-O-Al. Great attention are devoted to these new materials. Knowledge of these materials were published especially in symposiums [1-7]. In these works there are mentioned the study synthesis results of geopolymers, microstructures (mainly SEM) but also resulting possibilities how to apply them, mainly when using the waste inorganic materials (above all power station ash). Geopolymers represent new material types on the border among glass, ceramics materials and materials based on classical inorganic bonds (linkage). Geopolymers give the potential possibilities to prepare inorganic bonds and building materials from the waste as slag, fly ash, kaolinitic substances, etc. When geopolymers are prepared, the burning process at high temperatures is not needed as it is during production of cement or lime. Building material production from geopolymers does not in effect encumbrance the environment by CO₂ emissions and that is why these materials present the potential possibility of building material production without CO₂ emissions [1-7]. Out of the up-to-date knowledge of geopolymer qualities, some materials are obvious to have the same or even better qualities than those based on Portland cement. And above all, the materials based on geopolymers are evidently the parts of the ancient constructions aged several thousand years as the detailed search of these ancient constructions in Egypt and other countries shows [8,9].

In the until now published works [1-7] the attention is mainly devoted to geopolymer's polymer character (NMR in solid phase), mechanical characters, influence of aggressive environment, ability of leaching and others. In literature one can find only few facts about other material qualities of geopolymers like porosity, qualities on the border geopolymer - aggregate or micromechanical qualities. This study is devoted to these parameters.

EXPERIMENTAL

In this study power station fly ash (the Czech Republic, brown coal) with specific surface 210 m²/kg (Blaine), whose chemical composition is given in table 1, was used.

Pastes were prepared by mixing fly ash and other ingredients with solution of alkali activator. Mortars were prepared by mixing the pastes with siliceous sand (0.2-2 mm fraction) in proportion fly ash : aggregate 1:2 to 1:4. Concretes were prepared by mixing fly ash with the alkali activator and aggregate (0-16 mm fraction) in proportion fly ash : aggregate 1:3 to 1:5. Proportion SiO₂/Na₂O (modul Ms) in alkali activator was processed by extra NaOH to water glass at modul Ms = 2.5 to the values within Ms = 1 up 1.6. The total concentration of alkali activator was within 6 to 10 wt.% Na₂O from the fly ash mass. Water coefficient (proportion water mass/fly ash mass) w/s varies from 0.23 to 0.37. In some cases the substances containing Ca like ground blast furnace granulated slag were added.

After preparing the pastes, mortars resp. concretes the experimental bodies (in steel forms) were being put under thermal process on "dry conditions" in a dry room

at temperature 60-90°C (in the open air atmosphere) for 4-16 hours. After they were stored in the open air surrounding with 40-50 % R.H. until strength was determined. Compressive strength were determined after 2, 7 and 28 days resp. after 90, 360 days. Fragments, after determining the strength, were investigated with x-ray diffraction analysis, high-pressure Hg porosimetry, measuring by BET isotherm. Further on spectra FTIR and NMR MAS (²⁹Si, ²⁷Al) were measured. The microstructure ESEM was examined on the fragments' breaking flats after destructing solid tests where analysis ED was done on the chosen spots with spectrometer.

A micromechanical measurement (nanointendence) was done on the polished cement flats but only on the places of geopolymer, resp. C-S-H matrix. This technique is based on the direct measurement of the load-displacement relationship using a very sharp diamond tip pressed into the material. The Nanotest nanoindenter by Micromaterials, UK was used for all measurements. It was equipped with standard pyramidal Berkowich type. The depth of penetration starts from the level of nanometers and can reach several micrometers. In our case, the final depth was not set equal but final load 30 mN was common for all indents. The corresponding indentation depth was about 1µm for polymeric phase. First the samples were mechanically polished on emery papers to prepare sufficiently flat surface. Then, the sample was indented at the selected area which covered about 350×300 µm. The indenter imprints formed a rectangular matrix of about 100 indents. It was unavoidable to produce indents in all material phases (geopolymer, C-S-H, slag, fly ash or aggregate particles). These were subsequently separated into individual groups by ESEM.

RESULTS AND DISCUSSION

Strength

From our experiments, strength of geopolymer's pastes, mortars and concretes based on fly ash are obvious to have increasing character within 2-360 days according to preparing conditions and composition. The highest geopolymer strength was reached within 100-160 MPa in pressure after 28 days (fly ash and blast furnace slag) [10,11].

The basic material of the geopolymer based on fly ash is of prevalingly amorphous character only seldom containing needle-shaped minority crystals. XRD pat-

terns (figure 1) show that the geopolymer materials are prevalingly of X-ray amorphous character where the unique diffraction (apart from those of the original materials, mullite, quartz) is that at *d* = 0.304 nm, solely occurring in systems with a high content of slag [10]. The line obviously corresponds to the C-S-H phase formed. There is obvious difference among original fly ash and geopolymer (paste) on spectra FTIR (figure 2).

Table 2. Compressive strength of geopolymer materials (MPa).

Geopolymer material	days			
	2	28	180	360
Mortar	32	44	53	63
Concrete	no exam.	55	58	60
Paste (fly ash + ground slag)	95	138	152	164

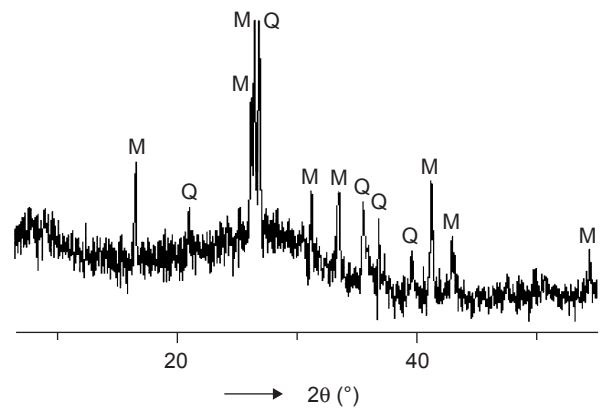


Figure 1. XRD pattern of fly ash based geopolymer (M - mullite, Q - quartz).

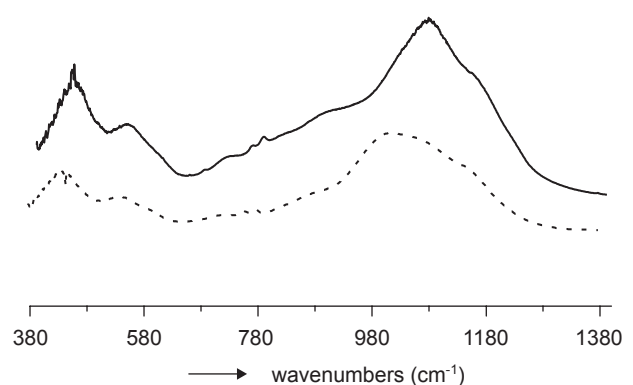


Figure 2. IR spectrum of fly ash and fly ash based geopolymer (paste *w* = 0.29).

Table 1. Composition of fly ash (wt.%).

Oxide (wt.%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	TiO ₂	P ₂ O ₅
Fly ash	53.79	32.97	5.51	1.84	0.92	0.46	1.76	0.37	2.1	0.15

Zone corresponding to Si–O and Al–O vibrations in original fly ash is 1080–1090 cm^{-1} , while it is shifted to lower values in geopolymers. This shift is interpreted as result of penetration into original skeletons structure Si–O–Si as it was found out at the zeolites. The more considerable the shift was, the higher penetration degree Al from the glassy parts of fly ash into $[\text{SiO}_4]^{4-}$ net [12,13].

A chemical shift equal to -107 ppm attributed to the tetrahedral $[\text{SiO}_4]^{4-}$ coordination of $\text{SiQ}^4(0\text{Al})$ and identified in ^{29}Si NMR MAS spectra of the original fly ash is regarded as the most important one [14,15]; also the shifts equal to -95, 87 and 79 ppm corresponding to the $\text{SiQ}^4(2-3\text{Al})$ coordination could be found. The latter shift belongs probably to the mullite phase. The shifts equal to 64.3, 51.9, 18.4 and 3.5 ppm corresponding to the $\text{AlQ}^4(4\text{Si})$ and $\text{AlQ}^2(2\text{Si})$ and $\text{AlQ}^3(3\text{Si})$ coordination were found in the ^{27}Al NMR spectra.

A different situation was detected in the geopolymers (pastes). The main shift - the $\text{AlQ}^4(4\text{Si})$ coordination - equal to -89 ppm and also the shift equal to -97 ppm corresponding to the $\text{SiQ}^4(2-3\text{Al})$ were found in the ^{29}Si NMR MAS spectra. The shift equal to -107 ppm corresponding to the $\text{Si}(0\text{Al})$ coordination was less represented, which points to the Al penetration into the $[\text{SiO}_4]^{4-}$ skeleton. There was a dominant shift equal to

-55 ppm in the ^{27}Al NMR MAS spectra as well as a weak shift equal to 3 ppm. The $\text{AlQ}^4(4\text{Si})$ coordination corresponds to these shifts [15, 16, 25, 26]. Weak shift 3 ppm probably belongs the mullite phase in unreacted fly ash [28], see figure 3.

It is obvious from these results that the structure of product after alkali fly ash activation (geopolymerization) is completely different from the products obtained with alkali activation of Portland clinker (low porosity gypsum-free Portland cement) [11].

Geopolymer's point analysis (SEM + ED) based on fly ash shows the composition of geopolymer within $\text{Na}_2\text{O}/\text{SiO}_2$ 0.1–0.3, $\text{Al}_2\text{O}_3/\text{SiO}_2$ 0.25–0.35. Analysis of the geopolymer based on fly ash and ground slag shows local spots containing higher proportion of Ca beside Si, Al and Na. Those spots obviously correspond with the phase C–S–H enriched by Al, which coexist with geopolymer phase. Geopolymer based on fly ash has very similar building units as the mineral analcim $\text{Na}_{16}[(\text{AlO}_2)_{10}(\text{SiO}_2)_{26}] \cdot 2\text{H}_2\text{O}$.

Process of geopolymerization (alkali activation of fly ash in water surrounding at $\text{pH} > 12$), causing hardening the material, is different from the processes of hydration the inorganic binders, for example Portland cement. This process (obviously) mainly runs by mechanism "through the solution", when parts of fly ash are firstly

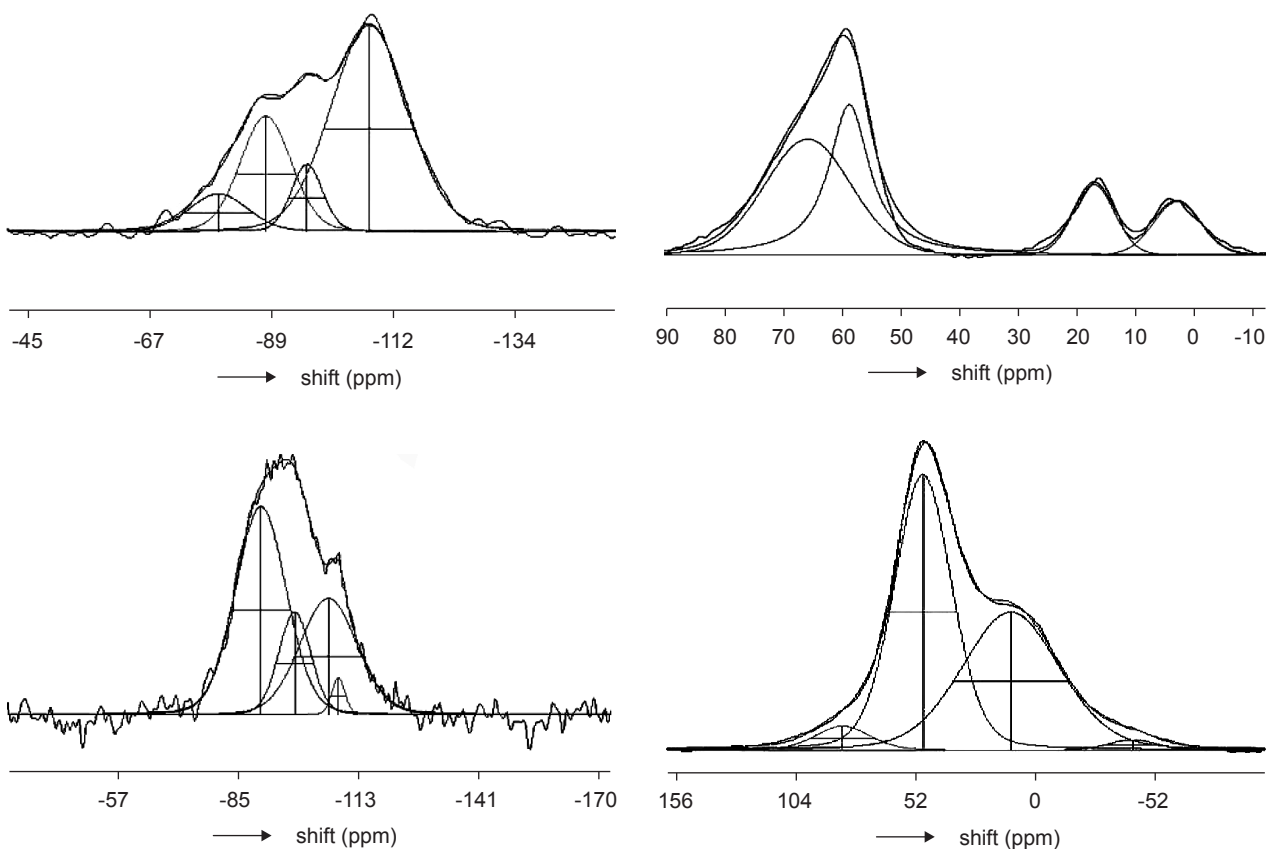


Figure 3. NMR MAS ^{29}Si , ^{27}Al spectra of fly ash and geopolymer (paste $w = 0.29$).

dissolved in strong alkali surrounding solution and then a new geopolymer structure is developed in this solution (see figures 4, 5). An important role, apart from the conditions of preparing, is also played by presence of atoms

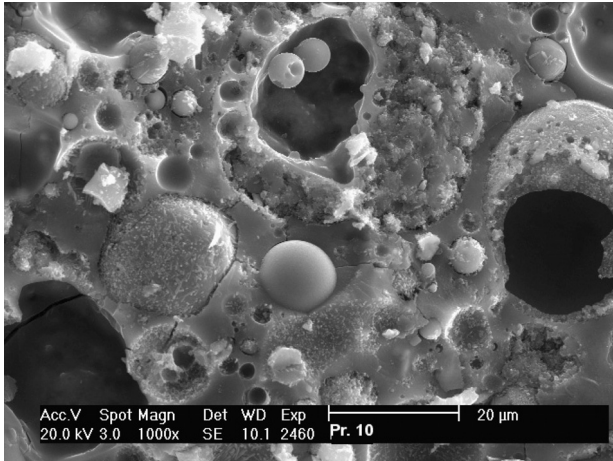


Figure 4. Typical appearance of the geopolymer from fly ash (fractured flat, paste $w = 0.27$).

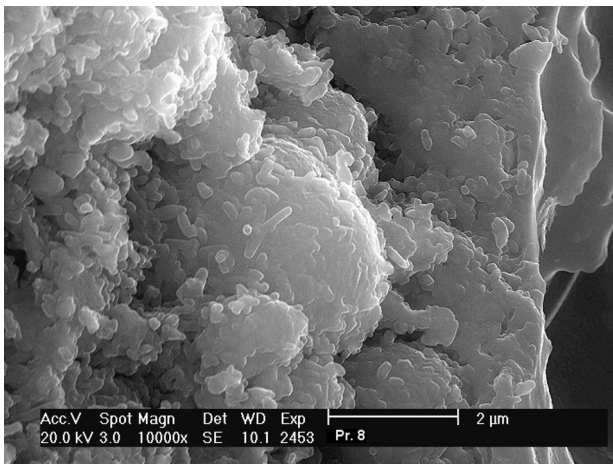


Figure 5. Detailed character of the geopolymers.

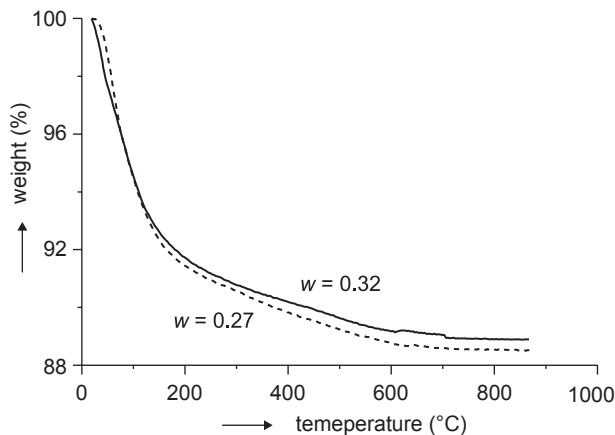


Figure 6. GTA of geopolymer (paste $w = 0.27$, resp. 0.32).

Ca which enter the Si–O–Al–O skeleton and in which they compensate the loading on Al atoms. These loadings are usually compensated by Na^+ , Ca^{2+} ions. However, they can connect the individual chain Si–O–Al–O through and it makes the structure with a higher strength at presence of the substances containing Ca. The significant role of Ca atoms (arising of strength, reduction of leaching) in geopolymer's skeleton is also shown in results from works [17-20]. The presence of water was proved by thermal analysis, figure 6. Water is obviously present in the form of "free water", further the water molecules are inside the structure and further the groups OH are present. When heating to 150-200°C, the main part of water disappears.

Geopolymer is possible to characterize as two up three-dimensioned inorganic polymer (geopolymer) of common pattern $\text{M}_n[-(\text{Si-O})_2-\text{Al-O}]_n \cdot w \text{H}_2\text{O}$.

Porosity of geopolymer

Fly ash based geopolymer's basic mass is of amorphous glass character, where needle minority configurations are present only seldom. There are the rests of origin ash parts present in the geopolymer's mass, where the influence of gradual dissolution is obvious on. At those geopolymers the relatively high porosity was found out (up 50 %) without any regards to character of preparing conditions. There are closed ball-shaped pores here, which were made by dissolving the origin fly ash parts, resp. bringing the air at preparing. The influence of preparing conditions, it means Na_2O contain, resp. proportion of $\text{SiO}_2/\text{Na}_2\text{O}$, is displayed on the approached geopolymers' strength but also on pore size distribution, figure 7. The geopolymer porosity is also evidently influenced by the value of the water-to-fly ash ratio, figure 8. Evidently lower porosity is at the geopolymers, prepared from the mixture of fly ash and slag, where the total porosity was 2-10 % and where the shift to lower pore's dimension was recorded. These substances reached the strength 100-160 MPa in pressure after 28 days.

High pressure Hg porosimetry, however, gives only limited information on the character of the pores in nanometric area. Other information is possible to get from measurement with the BET, where, figure 9, distribution of the pore sizes of geopolymers prepared on different conditions is seen. These obvious results, that the geopolymers have relatively similar character in nanometric area, which is not too dependent on preparing conditions. Only geopolymer patterns differ from each other where, apart from fly ash, slag was used. There the porosity drops in these materials, because of geopolymer coexistence and C–S–H phase, figure 10.

Interface geopolymer - aggregate

Transited layers between aggregate corn and hard cement exist at the common cement based on standard Portland cement [21, 22]. This layer is 20-100 μm thick and it has different microstructure and composition from the hardened cement paste [21, 22]. It is richer in $\text{Ca}(\text{OH})_2$ and ettringite content. There are often orientated $\text{Ca}(\text{OH})_2$ parts along the aggregate parts. Porosity of

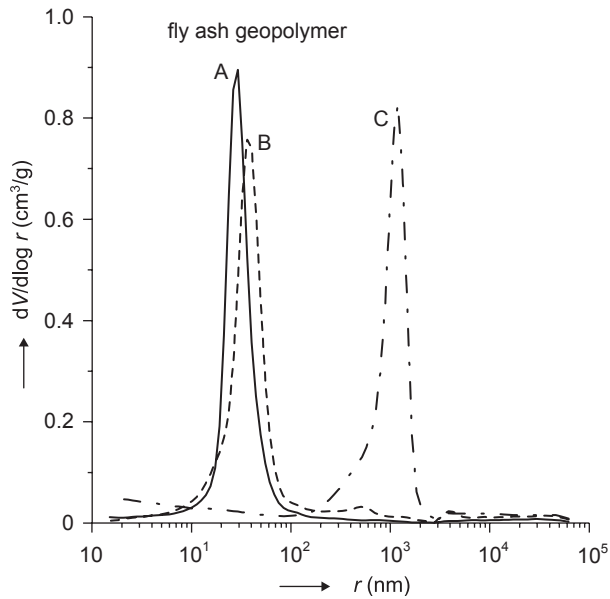


Figure 7. Influence of the alkali activation conditions on the pore sizes distribution (Hg porosimetry) in geopolymer, paste $w = 0.27$ (A - 60 MPa 9 % Na_2O , B - 40 MPa 8 % Na_2O , C - 18 MPa 5 % Na_2O).

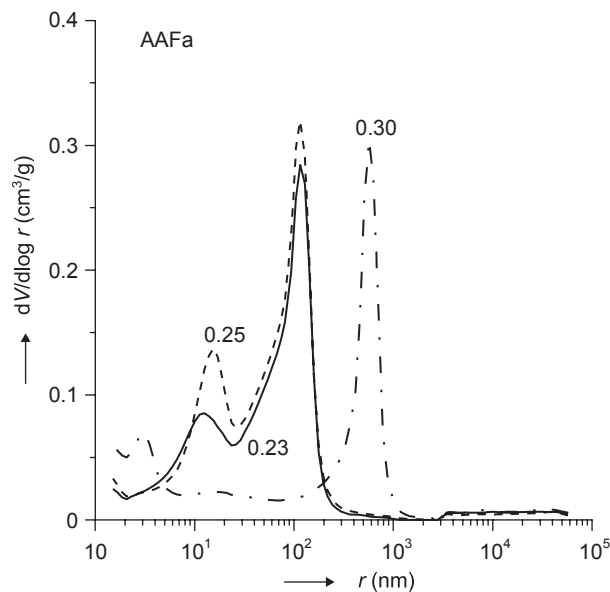


Figure 8. Influence of the water coefficient to the pore size distribution in the geopolymer, paste with water coefficient 0.23-0.30.

the transited layer is bigger than the one of the hardened cement paste and porosity gradient was found when it drops in the direction of the aggregate part [21,22].

Border character of geopolymer and aggregate is completely different. The transmitted layer was not found either morphologically nor by direct measurement of geopolymer composition in the aggregate surroundings, figures 11, 12. Proportions of $\text{Na}_2\text{O}/\text{SiO}_2$ to $\text{Al}_2\text{O}_3/\text{SiO}_2$, in dependence of the distance from the aggregate part, are in the figures 13, 14. In the frame of experimental errors (heterogeneous geopolymer character) the considerable changes of geopolymer composition in the close nearness are not possible to happen as it is at Portland cement. Completely analogical character is of border steel- reinforcement geopolymer [11].

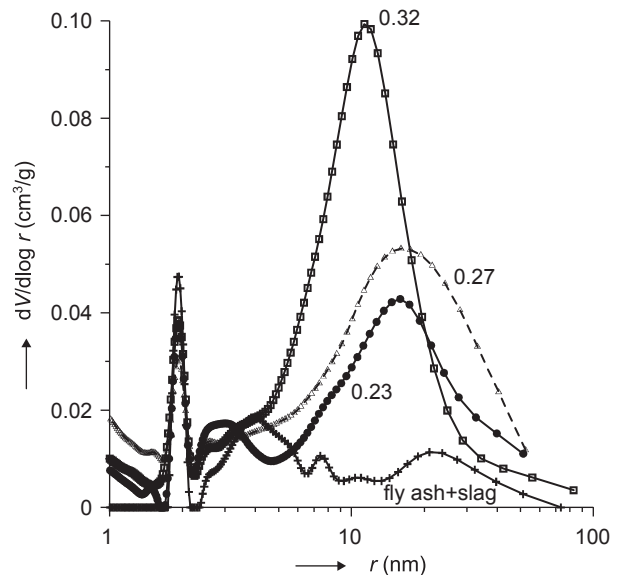


Figure 9. Pore size distribution of pores (BET) of geopolymers in dependence on water coefficient within 0.23-0.30 (paste), fly ash + slag geopolymer prepared from 60 % of fly ash and 40 % of ground slag.

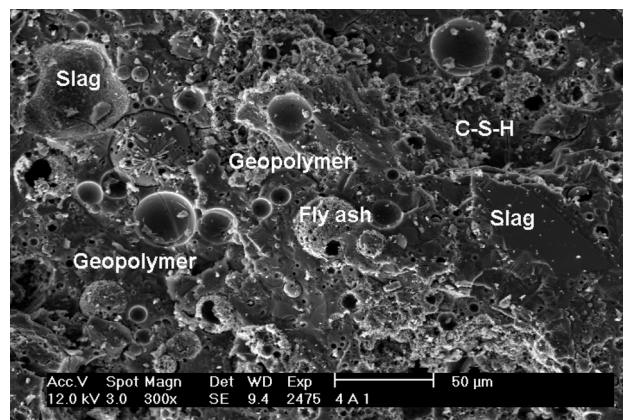


Figure 10. Geopolymer coexistence and C-S-H phase in geopolymer (paste, 60 % of fly ash and 40 % of ground slag), compressive strength after 28 days 120 MPa, fractured flat.

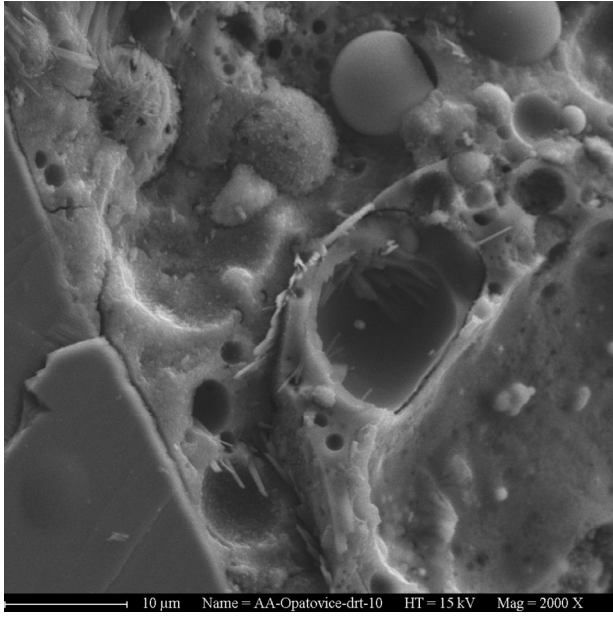


Figure 11. Interface between aggregate and geopolymer (mortar, fly ash:aggregate = 1:4.7, $w = 0.37$), fractured flat, 28 days.



Figure 12. Interface between aggregate and geopolymer (concrete, fly ash:aggregate = 1:4.7, $w = 0.37$, fractured flat, 28 days).

Micromechanical properties

Elastic properties of geopolymeric phase were evaluated according to standard Oliver-Pharr procedure [24] from unloading branches of P-h curves (figure 15). Since polymer is a time-dependent creeping material it was necessary to use long dwell period (200 s) in order to minimize effect of creep on the evaluation of elastic constants. Its time-dependent behavior is similar to cementitious composites. The scatter of results given by the different composition of indented places is relatively high. Therefore statistical evaluation of results from large number of indents is needed. The total num-

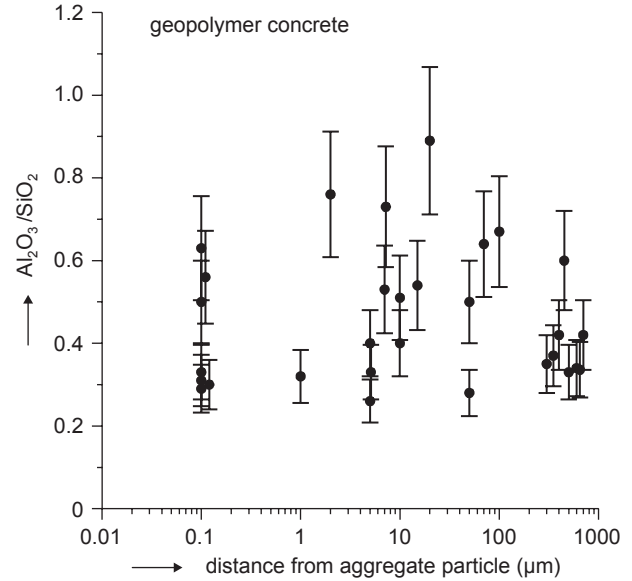


Figure 13. Dependence of the ratio $\text{Al}_2\text{O}_3/\text{SiO}_2$ in the geopolymer phase on the distance from aggregate in geopolymer concrete.

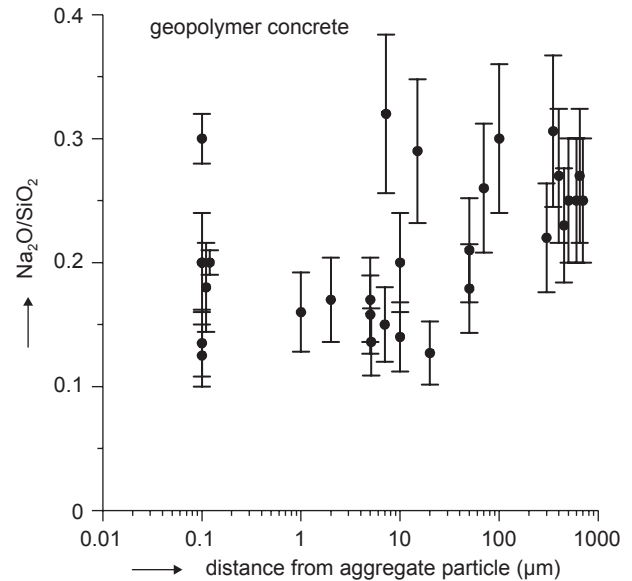


Figure 14. Dependence of the ratio $\text{Na}_2\text{O}/\text{SiO}_2$ in the geopolymer phase on the distance from aggregate.

ber of evaluated indents was 45. The rest of indents lied in remaining phases. Such evaluated elastic modulus for considered phase was $E = 36.1 \pm 5.1$ GPa. Hardness measured as the ratio of maximum indentation force and projected contact area of indenter was 1.11 ± 0.3 GPa. The course of indentation creep is shown in figure 16.

It is worth noting that the results are quantitatively close to results obtained on standard Portland cement pastes in case of elastic modulus. The elastic modulus evaluated only for hydrated cement phases (C-S-H and Portlandite) can be roughly estimated by the value of

25 ± 5 GPa (as taken from other studies). In case of hardness, the situation is different. Normal Portland cement pastes have hardness around 0.5 GPa. It points out the fact that geopolymeric matrices are less deformable and it could also indicate larger brittleness of geopolymer compared to ordinary Portland cement. However, such statement needs to be investigated in more details and further fracture tests need to be carried out.

CONCLUSION

1. Geopolymer's pastes, mortars and concretes based on fly ash are obvious to have increasing character within 2-360 days.
2. Geopolymer based on fly ash is the porous material. In the area of nanopores the geopolymer porosity is very similar without any regards to preparing conditions. The way how to prepare geopolymers (water coefficient, composition of alkali activator) influences the geopolymer porosity in the area (102 nm

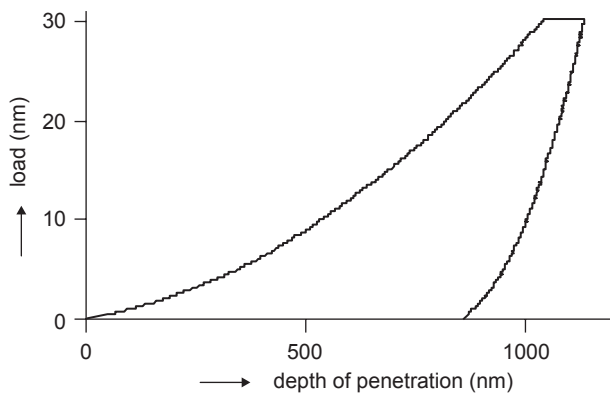


Figure 15. Example of experimental indentation curve measured on the geopolymer phase.

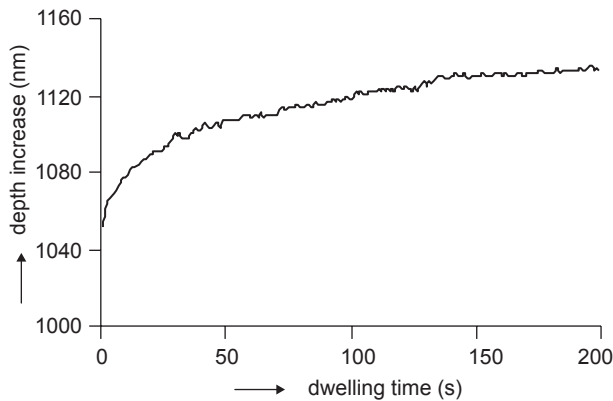


Figure 16. Example of indentation creep during 200 s dwell period.

- and more), which develop themselves by bringing the air to the geopolymers or these are partly reacted (hollow) fly ash parts. These pores have closed character.
3. Geopolymer prepared by fly ash alkali activation contains mainly structures of the types $AlQ^+(4Si)$. The main shift-coordination $SiQ^+(3Al)$ and $SiQ^+(2-3Al)$ was found in spectra ^{29}Si NMR MAS. Coordination $Si(0Al)$ has the minority representation, which penetration Al into the net $(SiO_4)^+$ proves. Products structure at alkali fly ash activation (geopolymerization) is completely different from the products developed at the alkali activation of Portland cement clinker (gypsum-free Portland cement).
4. Water is obviously present in the form of "free water", further the water molecules are inside the structure and further the groups OH^- are present.
5. Geopolymer is possible to characterize as two up three-dimensional inorganic polymer (geopolymer) of common pattern $M_n[-(Si-O)_2-Al-O]_n \cdot w H_2O$.
6. In the presence of Ca contained ingredients (slag) as a result of geopolymer coexistence and C-S-H phase and maximal geopolymer strength was reached.
7. Any presence of the temporary phase with different composition was not found between geopolymer and aggregate as it is at the concretes from portland cement. Geopolymer composition is almost the same in the close nearness even in the geopolymer mass.
8. Elastic modulus evaluated for the geopolymeric phase by means of nanoindentation was found to be $E = 36.1 \pm 5.1$ GPa. Such result is comparable (a little bit higher) to ordinary Portland cement pastes (OPC). The hardness of geopolymer is approximately twice higher than for OPC that could indicate less deformability and higher brittleness. Therefore, more studies on fracture properties of geopolymers is needed in this context.

Acknowledgement

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MIKROSTRUKTURA GEOPOLYMERNÍCH
MATERIÁLŮ NA BÁZI POPÍLKŮ

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Geopolymer připravený alkalickou aktivací popílku obsahuje převážně struktury typu $AlQ^+(4Si)$. Ve spektrech ^{29}Si NMR MAS byl nalezen jako hlavní posun - koordinace $SiQ^+(3Al)$ a $SiQ^+(2-3Al)$. Koordinace $Si(0Al)$ má minoritní zastoupení, což prokazuje průnik Al do sítě $[SiO_4]^+$. Struktura produktů při alkalické aktivaci popílků (geopolymeraci) je zcela odlišná od produktů vzniklých při alkalické aktivaci křemičitanového slínku. Geopolymer na bázi popílků je porézní materiál. V oblasti nanopórů je pórovitost geopolymerů velmi podobná bez ohledu na podmínky přípravy. Způsob přípravy geopolymerů (vodní součinitel, složení alkalického aktivátoru) ovlivňuje pórovitost geopolymerů v oblasti 102 nm a více. Pevnost geopolymerů výrazně ovlivňují makropóry (103 nm a více), které vznikají zavlečením vzduchu do geopolymerů nebo jsou to částečně zreagované (duté) částice popílku. Tyto póry mají uzavřený charakter. Za přítomnosti přísad obsahujících Ca (struska) se pórovitost značně snižuje v důsledku koexistence geopolymerní a C-S-H fáze a bylo dosaženo maximálních pevností geopolymerů. Mezi geopolymerem a kamenivem nebyla nalezena přítomnost přechodné fáze s odlišným složením, jako je tomu u betonů z portlandského cementu. Složení geopolymerů je prakticky stejné v těsné blízkosti kameniva i v hmotě geopolymerů. Modul pružnosti pro geopolymerní fázi byl naměřen $E = 36.1 \pm 5.1$ GPa. Tyto hodnoty jsou srovnatelné s hodnotami pro portlandský cement.