

# ALKALI ACTIVATED MATERIAL – GEOPOLYMER

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**Abstracts.** Overview of the state-of-the-art on the alkali activated materials since 1973 in Department of Glass and Ceramics in Prague. Geopolymer or alkali activated materials? Properties of geopolymer based on brown coal fly ash. Properties of alkali activated materials in comparison with standard Portland cement.

**Keyword.** *Geopolymer, alkali activated materials, microstructure, fly ash*

## 1 Introduction

Alkalis (Na, K) bring about problems in the technology of Portland cement and concrete. These troubles can be characterized as problems in the clinker production and the formation of undesirable silicate and aluminous phases of the type  $KC_{12}S_{23}$ ,  $NC_8A_3$ . However, the alkali-silica reaction in the concrete that is accompanied by expansion represents the major source of problems. Such problems call for a strict limitation of

the alkali content in the cement to values amounting to 0.6 percent and less (expressed as  $Na_2O$ ). On the other hand, however, soluble alkali compounds speed up the hydration process of hydraulic and latently hydraulic substances thus facilitating the formation of new hydration phases as this was shown by Purdon [1] in his work from 1940. In 1959 Gluchovskij [2] demonstrated in his book “Gruntosilikaty” the possibility of preparing new materials by means of the reaction of alumino-silicate raw materials (slags, fly ashes, clay materials) with alkaline compounds (carbonates, hydroxides, silicates).



Fig. 1. V.D.Gluchovskij: Book Gruntosilikaty, Kijev 1959.



The possibilities resulting from the use of the alkali activation of alumino-silicate materials were later investigated by a variety of workers. At present, the data base in Chemical Abstracts includes more than 1,800 original papers devoted to this topic. Furthermore, a series of congresses dealing with alkali-activated materials [3]–[10] took place since 1978.

The Institute of Glass and Ceramics ICT Prague has been investigating the problems associated with the alkali activation since 1973. The attention has been focused on processes of alkali activation of Portland cements, ground Portland cement clinker, blast-furnace slags, kaolinite materials and other inorganic wastes. The investigation into high-strength gypsum-free Portland cements (alkali-activated) lasted several years and eventually ended successfully with an industrial viability stage [11]–[13]. Our present-day attention focuses on alkali-activation processes of fly ashes occurring during the combustion of brown and bituminous types of coal. Fly ash wastes represent a considerable environmental problem in the Czech Republic because more than 10 million tons are rejected annually with the majority of this amount being disposed of on disposal sites.

## 1.1 Alkali activation

Alkali activation of alumino-silicate materials represents a complex process that has not been described to the full yet. The reaction of alumino-silicate materials in a strong alkaline environment results, first of all, in a breakdown of Si-O-Si bonds; later, new phases arise and the mechanism of their formation seems to be a process that includes a solution (“synthesis via solution”). The penetration of Al atoms into the original Si-O-Si structure represents a substantial feature of this reaction. Alumino-silicate gels (zeolite precursors) are mostly formed. Their composition can be characterized by the formula  $M_n[-(Si-O)_z - Al-O]_n \cdot wH_2O$ . The C-S-H and C-A-H phases may also be originated in dependence on the composition of the starting materials and the conditions of the reaction. Even secondary  $H_2O$  may be formed during these (poly-condensation) reactions. Amorphous (gel-like) or partially amorphous or crystalline substances may be originated in dependence on the character of starting raw materials and on the conditions of the reaction. The concentration of the solid matter plays a substantial role in the process of alkali activation. Mostly crystalline zeolite-type products (analcime, hydro-sodalite and other) are formed in extremely diluted suspensions



( $w > 1 \dots 10$ ). Therefore, the above process is used for the synthesis of man-made zeolites as this is corroborated by numerous publications. Predominantly amorphous products are originated at higher concentrations of the solid phase in the suspension ( $w < 1$ ).

It was for the above amorphous bodies that Davidovits [14] proposed a terminology of poly-sialates by taking into consideration the Si-to-Al ratio.

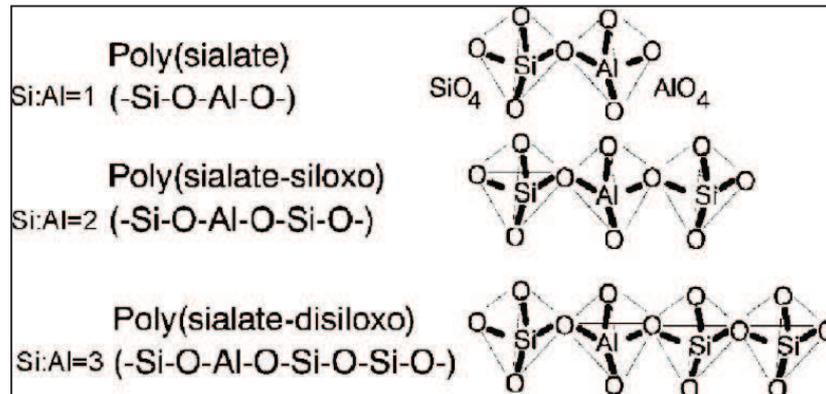


Fig. 2.

In 1976 to 1979 Davidovits [15] coined a new term “**geopolymer**” (almost 20 years after the Gluchovskij’s term “gruntosilikat”, “grunto-cement-geocement”).

Davidovits defines the material as follows: **The geopolymer is a material originated by inorganic poly-condensation, ie by so-called “geo-polymerization”** (in result of the alkali activation of aluminosilicate materials –our note).

A three-dimensional alumino-silicate network characterized by the empirical formula  $\text{Na, K}_n \{-(\text{Si-O})_z -\text{Al-O}\}_n \cdot w\text{H}_2\text{O}$  where  $M \dots \text{K, Na or Ca}$  atom,  $n \dots$  the degree of poly-condensation,  $z \dots 1, 2, 3$  or more than 3 is formed. The network is configured of  $\text{SiO}_4$  and  $\text{AlO}_4$  tetrahedrons united together by oxygen bridges. Chains or rings united by Si-O-Al bridges are formed. Positive ions ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{++}$ ) must compensate the negative charge of Al that is present in the coordination 4.

## 1.2 Conception of geopolymers

Based on his research work Davidovits [16] elaborated a structural model of the geopolymer (in this case, the model was designed for a poly-sialate – silox type formed by alkali activation of meta-kaolin). In his model he assumed an essentially monolithic polymer similar to organic polymers. (Fig.3).



Further progress in the investigation into the microstructure of the products of alkali activation (geopolymerization) and, in particular, the results obtained by using the methods of thermal analysis, mercury porosimetry, data obtained by measurement of BET isothermal curves and MAS NMR spectroscopy (Si, Al and especially Na) permitted a certain revision of this conception. Based on the above results Barbosa [17] proposed a modified model. Geopolymer materials are similar to vitreous bodies – the NMR spectra of aluminosilicate glasses and those of geopolymers are similar. Both materials exhibit the same three-dimensional arrangement. However, there is no water present in the glass structure, and the glass is practically without pores.

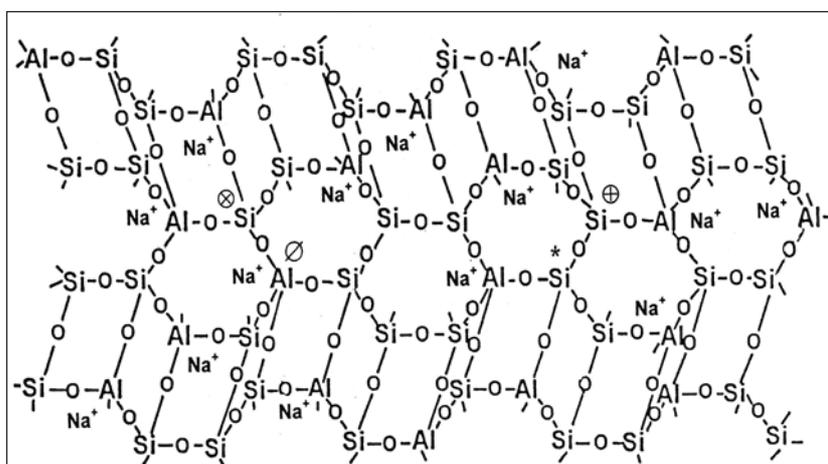


Fig. 3. Davidovits model.

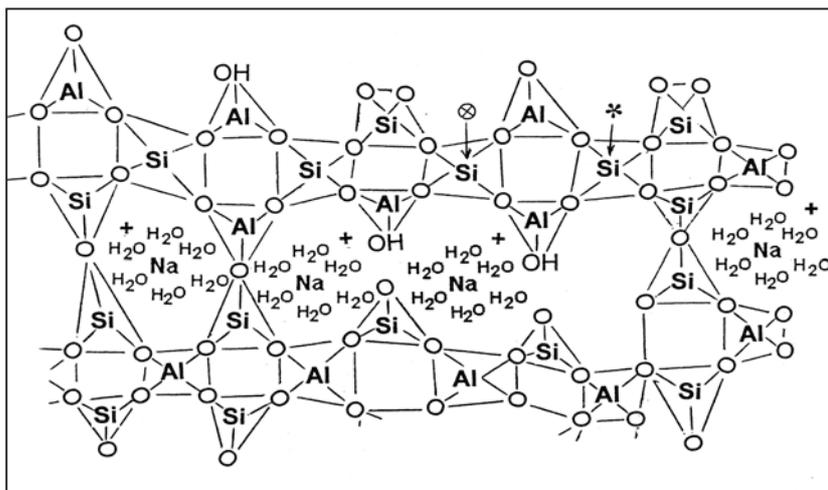
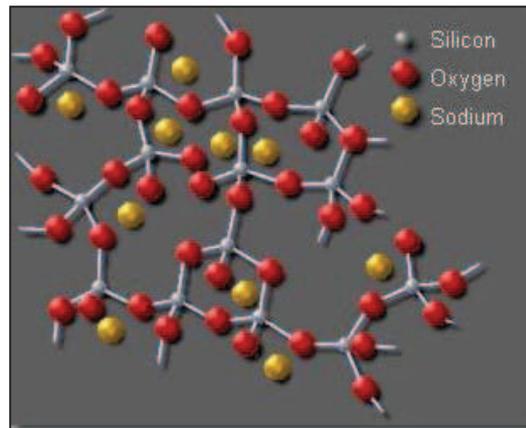


Fig. 4. Barbosa model.





**Fig. 5.** Structure of glass.

There is also a certain similarity between the geopolymer materials and the Portland cement because both materials are resistant to the action of water after their hardening. Therefore, they behave like a hydraulic binder. The hardened Portland cement is also a porous material with pores ranging from nanometers to micrometers. In contrast to the geopolymer, the Portland cement contains not only amorphous hydrates but also the crystalline ones ( $\text{Ca(OH)}_2$ , ettringite, etc.). There is  $\text{Ca(OH)}_2$  present in the pores of hardened Portland cement and the surface is coated by a thin layer of  $\text{CaCO}_3$  (because of the carbonate formation). The water is used (“consumed”) for the hydrate formation during the hardening of the Portland cement whereas it stays in the structure during the geopolymer hardening (water serves predominantly as a “carrier” of the alkali activating agent).

The results published up to now (they are in full agreement with our data) give the possibility to declare that a geopolymer material (on the basis of fly ashes, metakaolin):

- does not possess any monolithic structure of the poly-sialate - silox type,
- the alkali cation (Na, K) is present in the structure in a solvated form; furthermore, it is bonded more weakly than in the crystalline structure of zeolites (this fact represents a potential danger as regards the efflorescence occurrence),
- is rather characterized by a random three-dimensional arrangement,
- represents a porous body with pores ranging from nanometers to micrometers,
- contains water in pores and in the gel;

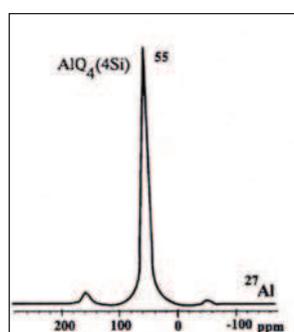


- the water plays only a role of a carrier of the alkali activating agent and that of a “rheological” water;
- crystalline and amorphous hydrates occur only rarely and only if a slag or Ca-containing material is present.

### Hence, what is a geopolymer?

Davidovits says [16], [18]: “ ... the  $^{27}\text{Al}$  NMR spectra of the product must exhibit a peak only at about 55 ppm. Al may be present in the product structure only and exclusively in the coordination 4...

*If it is not so, the product may not be called a geopolymer but only an alkali-activated material ...”*



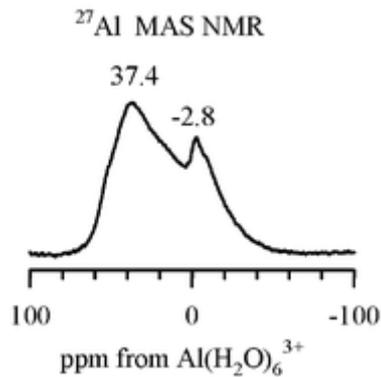
**Fig. 6.** NMR MAS  $^{27}\text{Al}$  of „true“, „genuine“ geopolymer [16], [18].

This very strictly formulated definition satisfies essentially only the materials obtained by alkali activation of pure metakaolin.

There is a variety of question marks coming to mind when the materials originated by alkali activation (geo-polymerization) are analyzed and classified:

- Do the materials obtained on the basis of other raw materials than meta-kaolin belong to geopolymers too?
- Can also the materials that contain Al in the coordination six, eg unreacted mullite residues (fly ashes) be regarded as geopolymers?
- Can the materials that also contain the C-S-H phase (in the case of slags or clinker) be called geopolymers, too?
- Can the materials formed only at a temperature of 20 C or during a hydrothermal synthesis or at a higher temperature be called geopolymers?
- Can the materials that also contain B and P atoms in addition to Si and Al atoms be classified as geopolymers (fig.6a)?





**Fig. 6a.** NMR MAS <sup>27</sup>Al of geopolymer containing P [21].

A scientific discussion on the above topics is necessary.

### What do have alkali-activated aluminosilicate materials in common?

The presence of the  $\text{Na, K}_n\{-(\text{Si-O})_z - \text{Al} - \text{O}\}_n \cdot w\text{H}_2\text{O}$  phase.

How should we call this phase?

An **N-S-A-H** or **K-S-A-H** phase (as would be customary among the cement scientists)?

Or a **zeolite-like phase** (Gluchovskii)?

(analogy to the tobermorite-like phase, the C-S-H phase today).

Or shouldn't we rather use a quite different denomination: **polymer silicate** ?

If we forget the strict definition of the geopolymer formulated by Davidovits (related to pure metakaolin) we shall reach a new very large group of inorganic binders with substantial ecological and energetic potentials as well as the binders with properties that have not been described yet. Then, such materials could be defined in accordance with the starting raw materials, eg:

**geopolymer on the fly ash basis**

**geopolymer on the metakaolin basis etc.**

If the geopolymers are conceptualized as a large group of binders, then significant differences in the product character and composition can be found during the alkali activation of various alumino-silicate raw materials (fig.7):

There are visible trends that can be observed in the series starting with clinker, slags, fly ashes and ending with kaolinitic materials:



- The necessity to increase the  $\text{Na}_2\text{O}$  content in the alkali activating agent is obvious.
- “Stronger” activating agents (alkali carbonate, alkali silicate, respectively alkali hydroxide) must be used.
- The content in the  $\text{M}_n [-(\text{Si} - \text{O})_z - \text{Al} - \text{O}]_n \cdot w\text{H}_2\text{O}$  phase increases.
- The Ca content in the product drops and, hence, also the content of the C-S-H and C-A-H phase decreases.

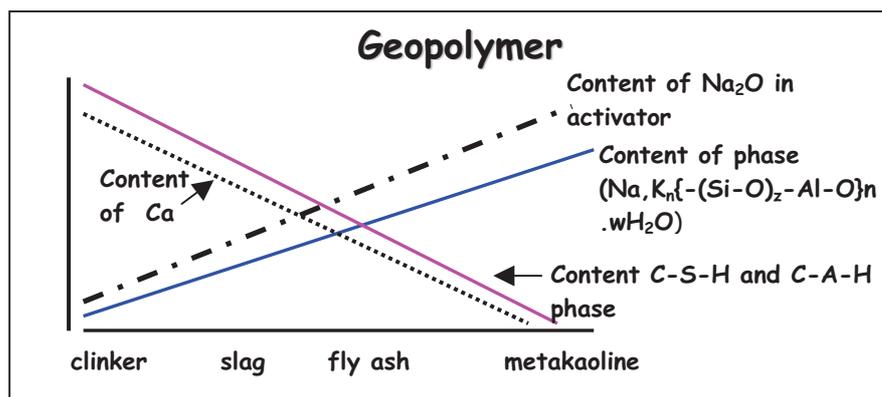


Fig. 7.

Therefore, two phases co-exist one along another in geopolymer materials the  $\text{M}_n [-(\text{Si} - \text{O})_z - \text{Al} - \text{O}]_n \cdot w\text{H}_2\text{O}$  phase having the character of a zeolite and the C-S-H phase, respectively the C-A-H phase. This coexistence affects the properties of geopolymer materials and we may state that the highest values of strength (up to 160 MPa compression strength) could be achieved in geopolymer materials that exhibited the coexistence of both types of phases.

The results of system analyses [19] of a wide range of aluminosilicate raw materials used for the preparation of geopolymers show that meta-kaolin occupies the top position as regards the consumption of energy, environmental impacts and cost (the cost/benefit factor amounting to 7-8); slags and ceramic waste are less demanding and the fly ashes (the cost/benefit factor amounting to 0.5) are the least demanding raw materials. The cost/benefit factor of the Portland cement (1.2) was determined in the same way.



## 2 Geopolymer based on brown coal fly ash (Czech Republic) [20]

### 2.1 Strength

From our experiments, strength of geopolymer's pastes, mortars and concretes based on brown coal 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).

Composition of fly ash

| wt.%    | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | CaO  | MgO  | SO <sub>3</sub> | K <sub>2</sub> O | Na <sub>2</sub> O | TiO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> |
|---------|------------------|--------------------------------|--------------------------------|------|------|-----------------|------------------|-------------------|------------------|-------------------------------|
| Fly ash | 53.79            | 32.97                          | 5.51                           | 1.84 | 0.92 | 0.46            | 1.76             | 0.37              | 2.1              | 0.15                          |

Compressive strength of geopolymer materials (in MPa)

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

### 2.2 Composition of geopolymer

The basic material of the geopolymer based on fly ash is of prevailingly amorphous character only seldom containing needle-shaped minority crystals. XRD patterns show that the geopolymer materials are prevailingly 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. 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. Zone corresponding to Si-O and Al-O vibrations in original fly ash is 1080 – 1090 cm<sup>-1</sup>, 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 [SiO<sub>4</sub>]<sup>4-</sup> net.



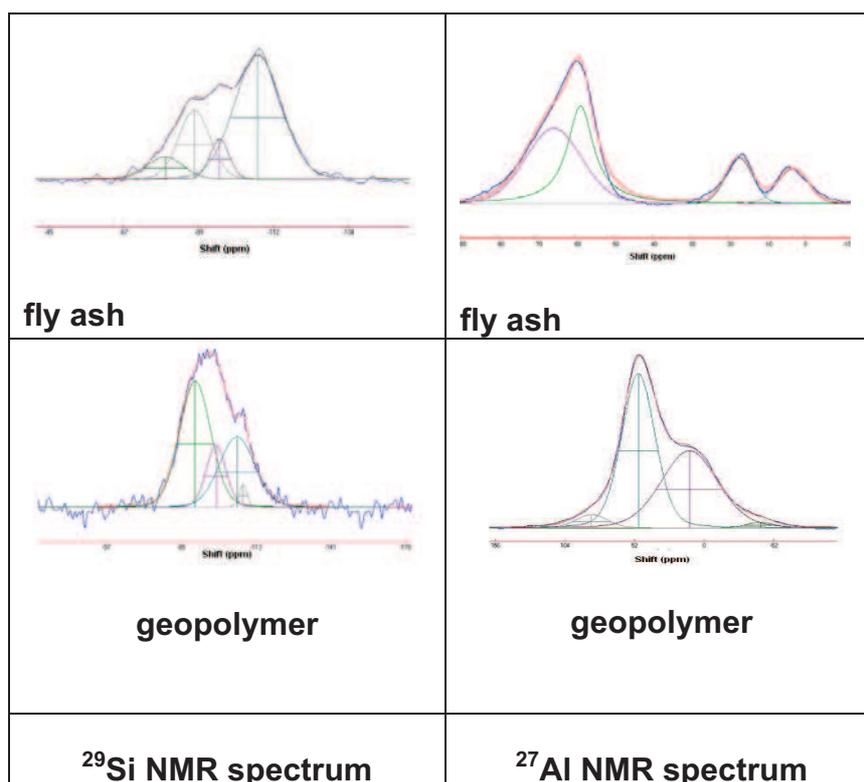


Fig. 8.

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  $^{29}\text{Si}$  NMR MAS spectra of the original fly ash is regarded as the most important one; also the shifts equal to  $-95$ ,  $87$  and  $79$  ppm corresponding to the  $\text{SiQ}^4(2\text{-}3\text{Al})$  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  $^{27}\text{Al}$  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  $\text{SiQ}^4(2\text{-}3\text{Al})$  were found in the  $^{29}\text{Si}$  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  $^{27}\text{Al}$  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. Weak shift  $3$  ppm probably belong the mullite phase in unreacted fly ash, see Fig.8.

It is obvious from these results that the structure of product after alkali fly ash activation (geopolymeration) is completely different from the



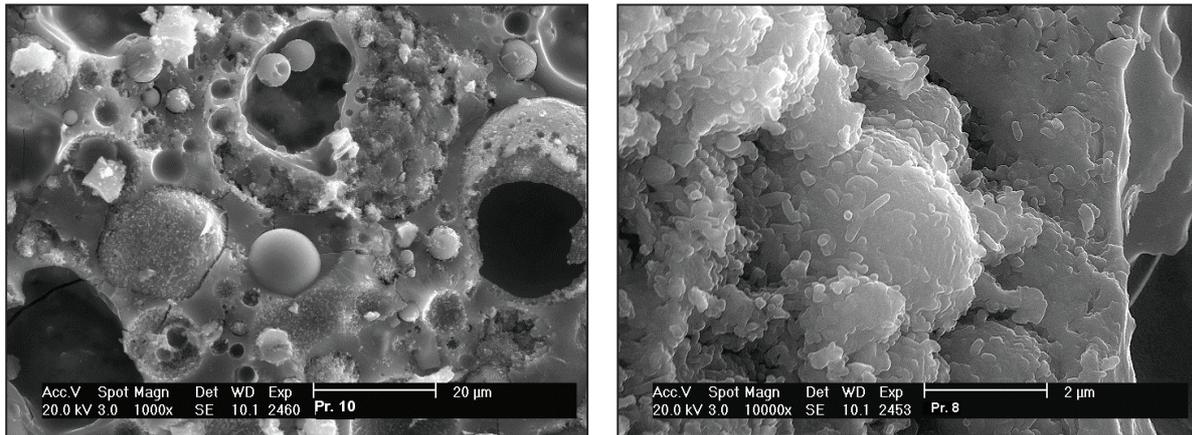


Fig. 9. Microstructure of fly ash based geopolymer.

products obtained with alkali activation of Portland clinker (low porosity gypsum-free Portland cement).

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  $\text{Na}_{16}((\text{AlO}_2)_{10}(\text{SiO}_2)_{26}).2\text{H}_2\text{O}$ .

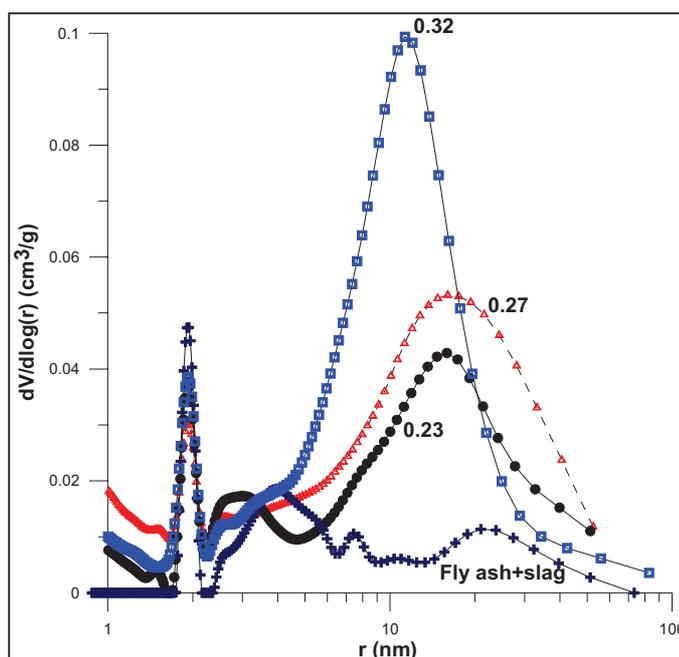
Process of geopolymeration (alkali activation of fly ash in water surrounding at  $\text{pH} > 12$ ), causing hardening the material, is different from the processes of hydration the inorganic binders, for example Portland cement. This process (obviously) mainly runs by mechanism „through the solution“, when parts of fly ash are firstly dissolved in strong alkali surrounding solution and then a new geopolymer structure is developed in this solution. An important role, apart from the conditions of preparing, is also played by presence of atoms 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  $\text{Na}^+$ .  $\text{Ca}^{2+}$  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. The presence of water was proved by thermal analysis. Water is obviously present in the form of „free water“, further the water molecules are inside the structure and further the groups  $\text{OH}^-$  are present. When heating to 150 – 200°C, the main part of water disappears.



**Geopolymer based on brown fly ash is possible to characterize as two up three-dimensional inorganic polymer (geopolymer) of common pattern  $M_n(-(Si-O)_2-Al-O)_n \cdot w H_2O$ .**

### 2.3 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.



**Fig. 10.** Pore size distribution of fly ash based geopolymer (BET).

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  $SiO_2/Na_2O$ , is displayed on the approached geopolymers' strength but also on pore size distribution, Fig.10. The geopolymer porosity is also evidently influenced by the value of the water-to-fly ash ratio. 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, Fig. 10, 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.

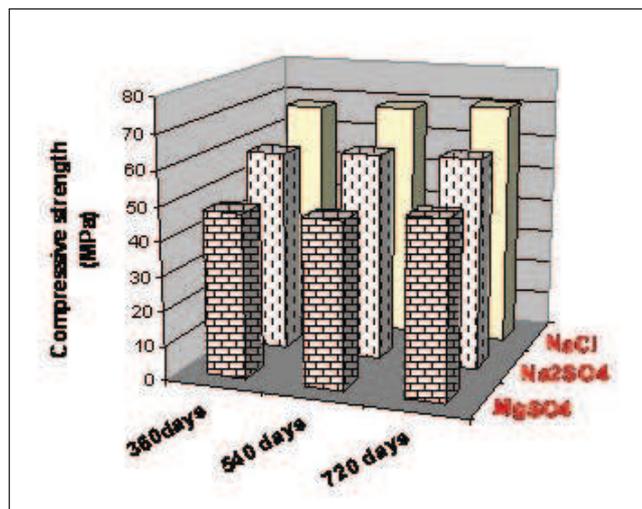
## 2.4 Durability of the geopolymers materials

The mass of the sample bodies did not change practically during the freezing and defrosting cycles taking place in the aqueous environment (no disintegration of the samples took place). The compressive strength of the sample bodies after the freezing cycles were lower as compared with those obtained for the samples after 28 days without any frost resistance tests. No visible defects or deformation could be observed after 150 cycles. After 150 freezing cycles, the strength values dropped to about 70% of the strength measured in the same period of time. It is obvious from the results obtained that the geopolymers materials on the basis of the fly ash possess an excellent frost resistance.

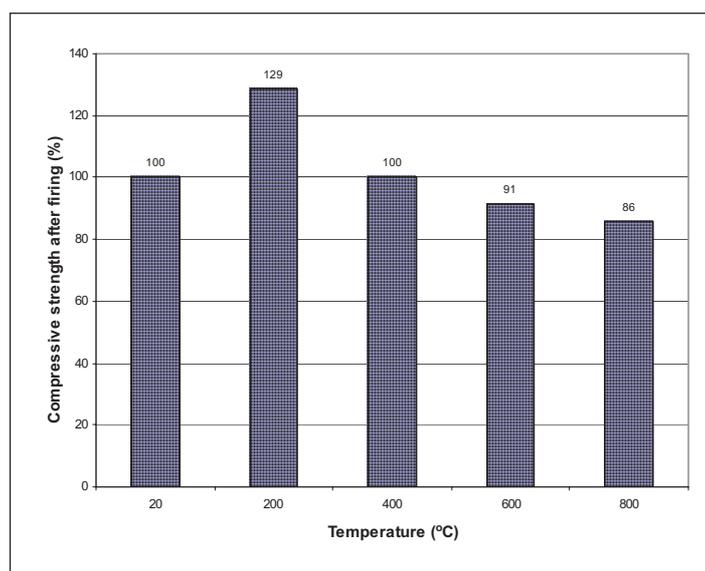
Geopolymer mortars and concretes kept in the NaCl, Na<sub>2</sub>SO<sub>4</sub> and MgSO<sub>4</sub> solution for long periods of time resist to corrosion without showing any signs of sample damage (fig.11). The exposure of the geopolymers materials to the NaCl solution resulted in the consistent increase in the compressive strength during the whole period of measurement (720 days) and the values were as high as 70 MPa. The penetration of chloride ions into the body of the geopolymers materials (point analyses, the measurement was carried out in the direction perpendicular to the surface in contact with the NaCl solution) shows a decreasing trend and the chloride concentration in the body is low. Almost no corrosion products could be found on the surface of the geopolymer materials while they were kept in the NaCl solution.

The exposure of geopolymers materials to the solutions of sulfates and NaCl did not result in the formation of new crystalline phases. The RTG diffraction analysis only confirmed the occurrence of original crystalline phases present in the fly ash. The absence of expansive products as, for instance, the ettringite (C<sub>6</sub>A $\bar{S}$ <sub>3</sub>H<sub>32</sub>) or Friedel salt (C<sub>4</sub>AClH<sub>11</sub>) in the samples after the two-year exposure to the salt solution is an important finding.





**Fig.11.** Compressive strength of fly ash based geopolymer immersed in salts solutions.



**Fig. 12.** Compressive strength vs. temperature of firing (fly ash based geopolymer). Strength measured after firing.

The effect of high temperatures on the properties of geopolymer mortars was investigated in the temperature range of 150 to 1100°C (fig.12). A geopolymer material exposed to the action of high temperatures loses a substantial part of its strength that drops to 40% of its original value. The strength already starts dropping at a temperature of 250°C and it remains practically the same at higher firing temperatures. The lowest values of the residual strength were observed in the temperature



range of 600 to 700°C; they were due to the presence of the melt that started forming. The values of the residual strength after the firing were rather higher than those characterizing the materials on the basis of Portland cement.

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