

NITRIC ACID ATTACK ON HARDENED PASTE OF GEOPOLYMERIC CEMENTS

PART 1

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Corrosion of hardened paste of geopolymeric cements in solutions of nitric acid starts by a leaching process in which the soluble contents of the material (i.e. sodium and calcium) are depleted and replaced by H^+ or H_3O^+ ions from solution along with an attack by acidic protons on polymeric Si-O-Al bonds (to be discussed in Part 2) resulting in the formation of a partially dealcalized and decalcified Si-, Al-rich residue.

Keywords: Geopolymeric cement, Acid attack, Fly ash, Blast furnace slag

INTRODUCTION

The use of alkali-activated cementitious materials, specially over the past decades, has significantly been increased not only due to their potential in reducing CO₂ emission from manufacture of Portland cement, but also due to their superior, long-term engineering properties. Alkali-activation process which was originally limited to the slag cements, recently has been extended for several silica and/or alumina rich minerals and industrial by-products including ; clay, ground rock, slag, and fly ash [1 - 6]. The nature of these binders and their engineering properties have been discussed in a number of papers [1 - 11].

An interesting property of geopolymeric cements (a new class of alkali-activated materials) is their relatively high resistance to acidic media. The papers presented by Changgao [12], Blaakmeer [13], and Xincheng [14] indicate that acid resistance of geopolymeric cements produced by activating blast furnace slag or mixtures of fly ash and blast furnace slag (i.e. Diabind and FKJ respectively) greatly exceeds that of normal Portland cement. Rostami and Silverstrim [4, 5], developed an alkali-activated material called, chemically activated fly ash (CAFA) which is an activated Class F fly ash. They tested CAFA concrete [4, 5] for durability properties. The resistance of CAFA concrete to chemical attack by acids such as sulfuric, nitric, hydrochloric, and organic acids was claimed to be far better than that of Portland cement concrete [4]. According to Silverstrim *et al* [5], CAFA specimen exposed to 70 vol.% nitric acid for 3 months retained its dense microstructure.

The phenomenon of acidic corrosion of hydrated cement based materials has been thoroughly discussed in previous publications [15, 16]. The present work is devoted to the study of the mechanism of nitric acid attack on hardened paste of geopolymeric cements. The geopolymeric cement used in this work was produced according to the work of Škvára and Bohuněk [3]. They reported that alkali-activation brought about by the use of NaOH and Na₂SiO₃ solutions is capable of increasing significantly the reactivity of substances with latent hydraulic properties such as fly ash or mixtures of fly ash and blast furnace slag. Very high compressive strengths of 120 to 170 MPa were achieved with ash-slag mixtures over the composition range of 50 - 70 wt.% fly ash and 30-50 wt.% slag, activated with alkali-activator having a silica modulus ($M_s = SiO_2/Na_2O$) of 0.6, and containing 7 wt.% Na₂O.

EXPERIMENTAL PART

Materials

Materials used for this study include: fly ash from Chvaletice power plant and granulated blast furnace slag from Vítkovitz steel plant (both plants in Czech Rep.). These materials were firstly ground in a vibration mill to attain higher specific surface areas. The blast furnace slag was ground in the presence of a liquid grinding aid (ALSON TEA, Chemotex Company, Czech Rep.). The attained specific surface areas were 650 and 420 m² kg⁻¹ respectively for fly ash and blast furnace slag. Chemical compositions of these materials

Table 1. Chemical composition of fly ash and slag (wt.%).

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	TiO ₂	L.O.I.
ash	52.40	26.70	8.60	3.10	1.16	0.87	1.64	0.37	2.08	1.71
slag	43.25	7.51	0.50	41.47	4.36	0.82	0.63			

are listed in table 1. Solutions of nitric acid with *pH* values of 1, 2, and 3 were used.

Specimens preparation

The ground fly ash and ground granulated blast furnace slag were mixed in equal proportions. The SiO₂/Na₂O ratio (M_s = modulus) of the alkali-activator was adjusted to $M_s = 0.6$ by adding NaOH to water glass which itself had a modulus of $M_s = 1.68$. The concentration of the alkali-activator was so adjusted that the total Na₂O content of the binder amounted to 7 % of the binder weight. Enough water was added to the alkali-activator for preparing a paste with a water/binder ratio of 0.30 that had an acceptable workability. The prepared alkali-activator was then added in the form of liquid to the dry mixture and the paste was then formed into specimens of 20×20×20 mm in size. The curing procedure used was 24 hrs in moulds at 95 % relative humidity at 20 °C, then heating up to 60 °C during 1 hour (20 °C / 0.5 hr) followed by heating to 80 °C during 2 hours (10 °C / 1 hr) in a saturated steam atmosphere. The specimens were then held at the final temperature for an additional 4 hours. After that, the specimens were stored dry at 20 °C at 40 - 50 % relative humidity.

Test procedure

At the age of 28 days the specimens were immersed in three different solutions of nitric acid with *pH* values of 1 ± 0.01 , 2 ± 0.03 , and 3 ± 0.05 . All the solutions were prepared by adding concentrated acid to tap water. The *pH* levels of the solutions were monitored daily with a portable *pH*-meter and concentrated acids were added to maintain the *pH* values to the stated tolerances. During *pH* monitoring, an electrical mixer was used for mixing each of the solutions thoroughly. All the solutions were renewed monthly, and the temperature of the solutions was kept constant at 20 °C.

XRD (Seifert XRD 3000P), EPMA (JEOL Superprobe 733), SEM (JEOL), MAS NMRS (BRUKER AVANCE DSX 200) and FTIRS (Nicolet 740) were the principal laboratory techniques used to study the corroded specimens. For studies with EPMA and SEM a number of corroded specimens were cut into halves to expose the total internal surface including the cross section of the corroded layer. The suitable halves were then impregnated with an epoxy resin, polished, and coated with carbon. The electron probe

measurements were performed not only on selected points, but also along imaginary lines extended from the corroded part through corrosion zone and towards the internal unaffected part of the specimen. This part of article (Part 1) is allocated to the results of XRD, EPMA, and SEM techniques. The results of MAS NMR and FTIR spectroscopy techniques will be presented and discussed in the second part.

RESULTS AND DISCUSSION

Attempts were made by X-ray diffractometry to identify the hydration products of the material. The basic hydration products are generally of a prevailing amorphous character, only seldom containing a few crystalline minerals (figure 1). As seen the only

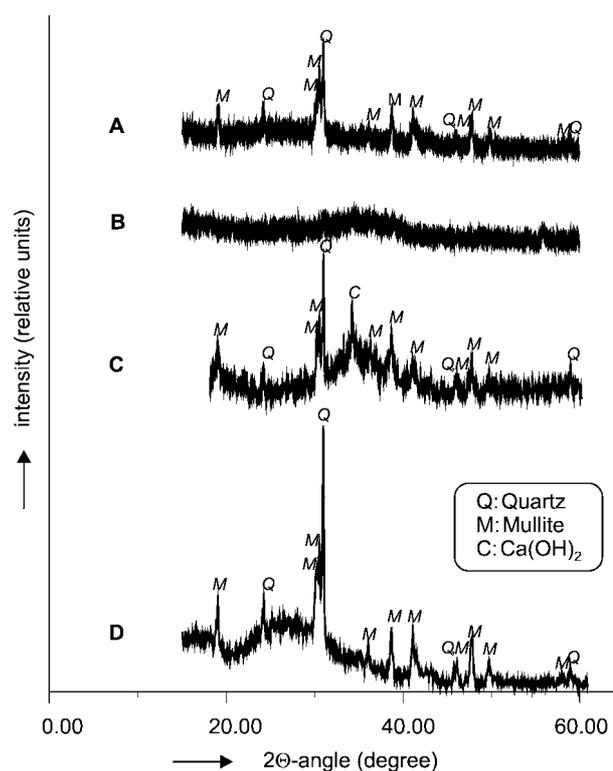


Figure 1. X-ray diffraction patterns.

A - Fly ash, B - Blast-furnace slag, C - Geopolymeric cement (alkali-activated ash/slag mixture after 24 hours in mould at 95 % relative humidity at 20 °C, followed by a hydrothermal curing and 27 days of dry cure), D - Corroded layer of Geopolymeric cement developed at *pH* = 1 nitric acid after an exposure period of 90 days.

different part from those of the original materials (Quartz and Mullite) is a broad diffuse halo at $d = 0.304$ nm, obviously corresponding to C-S-H phase that occurs in systems with a high content of slag. Recent investigations [1 - 9] on some of these materials however suggest that alkali-activation results in the formation of amorphous zeolite-like aluminosilicate hydrates. Changgao [11, 12] reported that the main hydration products in fly ash/slag-alkali system (FKJ) possibly is sodium-aluminosilicate hydrate of analcite type, along with a number of secondary mineral phases including calcium-aluminosilicate hydrate of scolecite type and sodium-calcium-aluminosilicate hydrate of thomsonite type, and a small amount of low alkaline calcium-silicate hydrate.

Taking into account that all the four patterns plotted on the same scales were conducted by the same XRD instrument and under the same conditions, it is seen that in the pattern of the corroded layer the diffraction lines of Quartz are considerably intensified compared to the corresponding lines in the pattern of the unaffected sample.

Visual observations

During the course of corrosion the changes in the appearance of the specimens were visually monitored. The observations include:

$pH = 1$ and 2 ; No change in colour, shrinkage cracks (very fine at $pH = 2$); relatively hard but brittle corroded layers that upon crushing changed to small hard particles ($< 5\text{mm}$).

$pH = 3$; No change in colour and appearance; a relatively soft and easily removable surface layer.

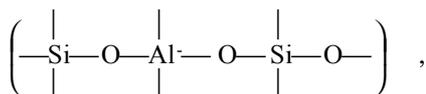
Attack at $pH = 1$

After 60 days of exposure to $pH 1$ nitric acid a corroded specimen was removed and prepared for investigations by scanning electron microscope (SEM). Figures 2 through 7 show a number of SEM images of the microstructure of an attacked specimen at different regions across the corroded layer and the unaffected part of the specimen. Typical $4000\times$ magnified SEM images of the microstructure of the corroded areas at regions very close to the acid-exposed surface and somewhere in the middle of the corroded layer are shown in figures 2 and 3. In figure 3 some corroded fly ash particles, an empty space of a slag particle along with partially corroded but still uniform glassy matrix can be seen. Typical SEM images of the unaffected microstructure of the same specimen and at the same magnification of $4000\times$ are shown in figures 4 and 5. Comparing the corroded microstructure (figures 2 and 3) to the unaffected one (figures 4 and 5), it is seen that the process of acidic corrosion does not result in complete destruction of the microstructure. As investigated by SEM, the extent of damage to microstructure across the corroded layer from the acid-

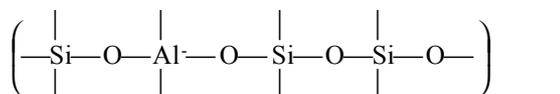
exposed surface toward the unaffected part was more or less the same. A typical SEM image of the unaffected microstructure of the specimen at the same magnification of $4000\times$ is shown in figure 6. As seen the unaffected microstructure of the tested geopolymeric cement consists of a very uniform glassy matrix in which small fly ash and slag particles are embedded. The microcracks seen in figures 4 and 5 are probably mechanical microcracks produced when cutting the specimen. Figure 7 shows parts of the corroded layer and the unaffected area adjacent to it at a lower magnification of $40\times$. As seen the corroded layer (on the left side) consists of a number of relatively large shrinkage cracks perpendicular to the acid-exposed surface.

To investigate the relative changes in chemical composition of the corroded layer, a number of X-ray line analysis (energy dispersion measurement, EDS) were conducted by electron probe microanalysis (EPMA). Figure 8 shows concentration profiles of Al, Si, Na, and Ca obtained from such a line analysis. Each profile shows the relative changes in concentration of a different element along an imaginary line extended from somewhere in the corroded part on the left side through corrosion zone and toward the unaffected part on the right side. The total length of line at a magnification of $40\times$ is 2.910 mm. As seen the process of acidic corrosion results in the leaching of both sodium and calcium (sodium a little bit more deeper than calcium). An enrichment in concentration of silicon can also be seen. The interesting point is the concentration of aluminium in the corroded layer showing no significant change even at regions close to the acid-exposed surface.

It is worth discussing briefly the chemistry and molecular structure of geopolymers according to Davidovits [17, 18]. Geopolymers based on aluminosilicates are designated by the term poly(sialate). The term sialate is used as an abbreviation for silicon-oxo-aluminate. The sialate network which may be considered as the result of the condensation of a still hypothetical monomer i.e. orthosialate; $(\text{OH})_3\text{-Si-O-Al-(OH)}_3$ or its ion, consists of SiO_4 and AlO_4 tetrahedra linked alternatively by sharing all the oxygens. Positive ions (Na^+ , K^+ , Ca^{++} , Mg^{++} , Ba^{++} , NH_4^+ , H_3O^+) must be present in the framework cavities to balance the negative charge of Al in 4 or 6-fold coordination. Condensation of orthosialate with silicic acid Si(OH)_4 results in the formation of a number of polysialates including poly(sialate-siloxo), i.e.



poly(sialate-disiloxo), i.e.



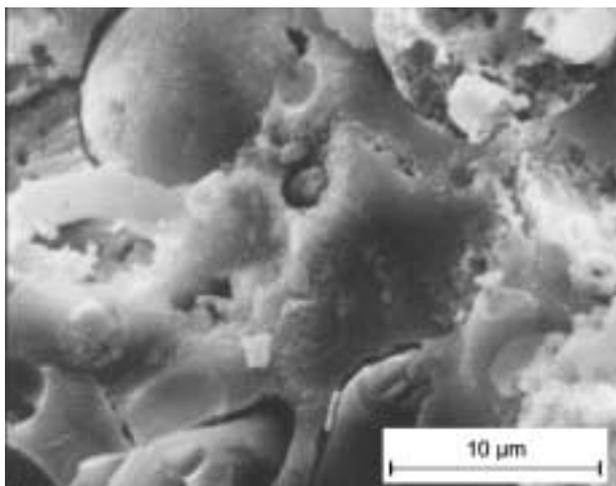


Figure 2. Microstructure of the corroded area at a region very close to the acid exposed surface of the specimen.

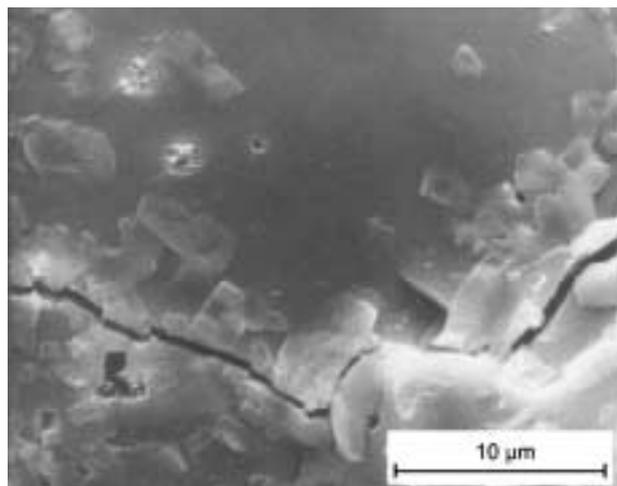


Figure 5. Unaffected microstructure of the specimen (uniform glassy matrix).

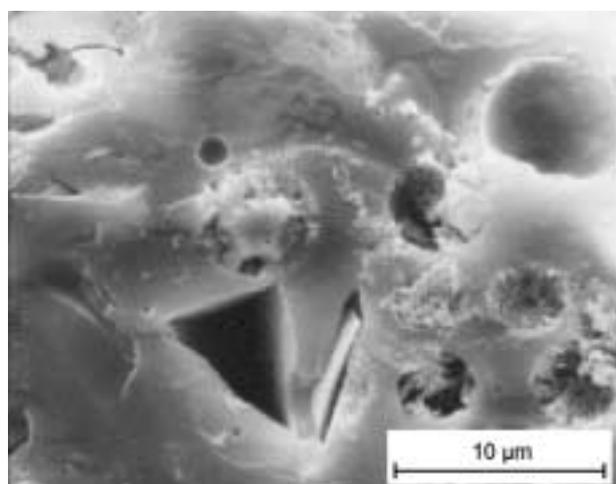


Figure 3. Microstructure of the corroded area somewhere in the middle of the corroded layer.

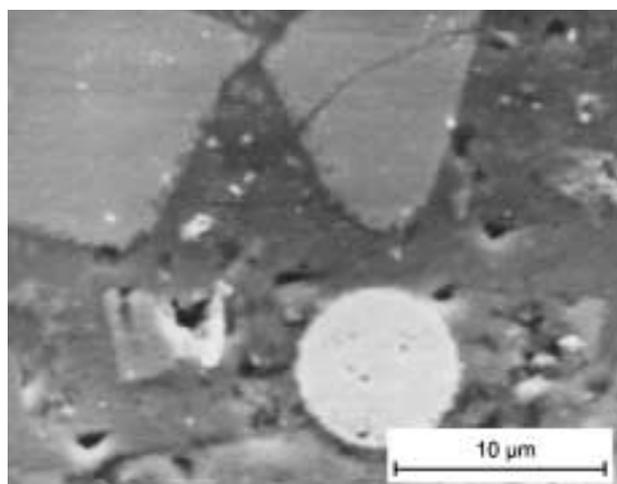


Figure 6. Unaffected microstructure of the specimen (slag and ash particles embedded in the uniform matrix).

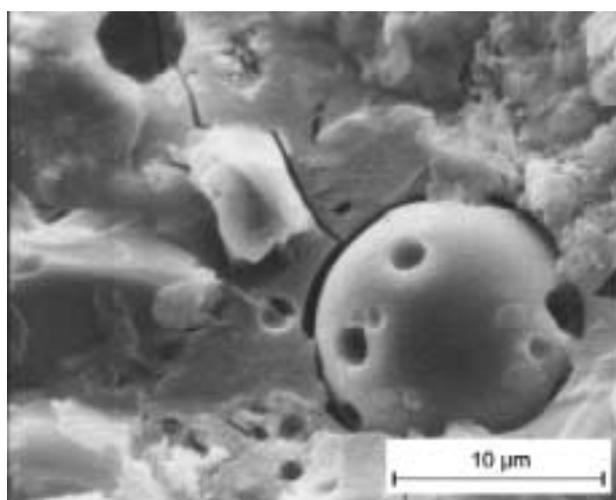


Figure 4. Unaffected microstructure of the specimen (uniform matrix along with an embedded fly ash particle).

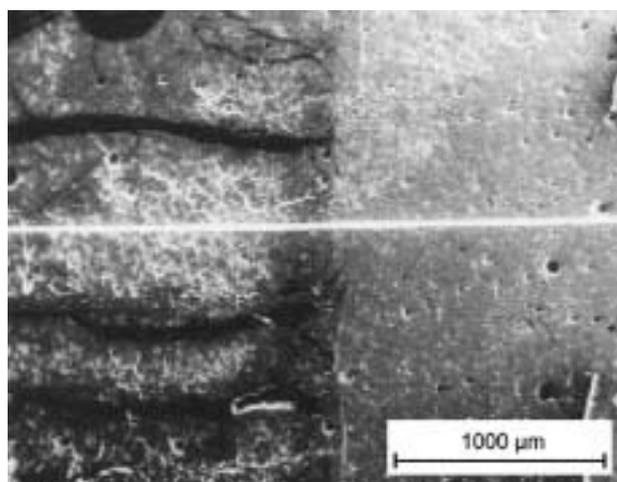


Figure 7. Corroded layer developed after 60 days exposure to $pH = 1$ nitric acid.

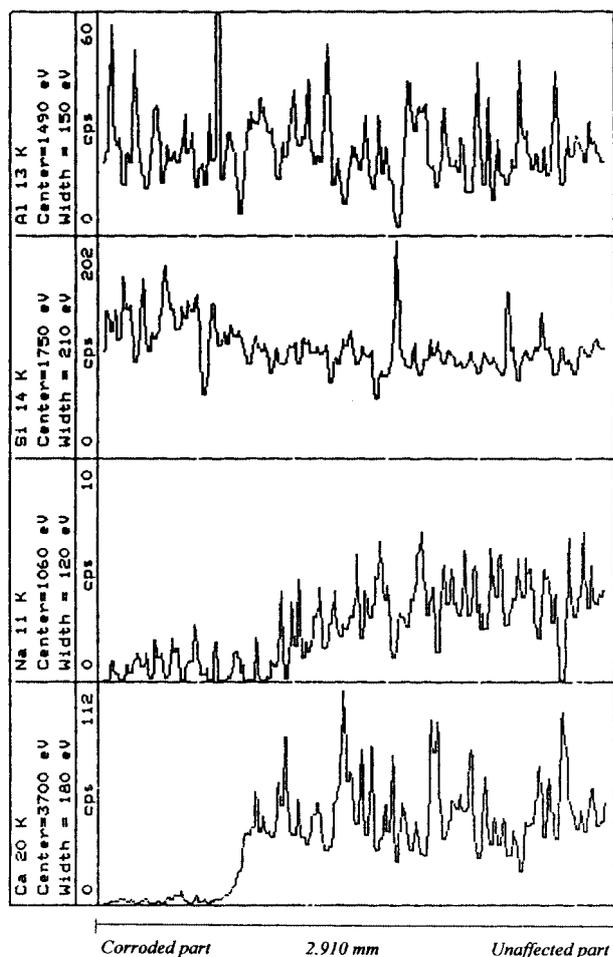


Figure 8. X-ray line analysis (EDS) of the specimen after 60 days exposure to $pH = 1$ nitric acid. (magnification = $40\times$).

and so on. The polysialates therefore have an empirical formula of the form;



in which; M is a cation (Na^+ , Ca^{++} ,...), n is degree of condensation, and z is the ratio Si/Al which may be 1, 2, 3, or $\gg 3$. The exact mechanism by which geopolymer setting and hardening occurs, is not fully understood. Most of the proposed mechanisms consists of dissolution, orientation and polycondensation [19, 20, 21]. The possible mechanisms of formation for a number of geopolymers (with different Si/Al ratio) including a geopolymeric cement, i.e. PZ-Geopoly cement, have been discussed by Davidovits [17]. Simply, one can say that the general possible mechanism for the geopolymeric cements involves the chemical reaction of a material rich in silica and alumina with alkalis and alkali-polysilicates resulting in the formation of an aluminosilicate framework with polymeric Si-O-Al bonds.

Now as seen in figure 8, the aluminum of geopolymeric cements which along with silicon was

incorporated into an aluminosilicate framework remains almost insoluble even at pH values as low as 1. Such an acid resistant three-dimensional aluminosilicate framework that encompasses the entire structure through polymeric Si-O-Al bonding is more durable in acidic media than structures based on foiled sheets of vulnerable calcium silicate hydrates and this is why unlike normal Portland cement, geopolymeric cements are not dissolved by acidic solutions.

The presence of sodium (or potassium) in the process of geopolymerization and in the form of an alkali metal salt and/or hydroxide is necessary not only for dissolution of silica and alumina, and for the catalysis of the condensation reaction, but also for filling the framework cavities to balance the negative charge of Al in 4-fold coordination. The presence of calcium however is not a necessity. Calcium in geopolymeric cements can be present not only in the form of a cation for balancing the negative charge of Al in 6-fold coordination [17, 18, 19, 22], but also in the form of calcium silicate hydrate [22]. Knowing that the electron charge of the 4 or 6-fold coordinated aluminium in the aluminosilicate framework can be compensated by hydronium ion [17, 18], one can conclude that after completion of geopolymerization process and once the framework of aluminosilicate has been formed the charge compensating cations (Na^+ , K^+ , and Ca^{2+}) can be exchanged by hydronium ions from the acidic solutions. Therefore taking into account the possibility at very low pH values for electrophilic attack by acid protons on polymeric Si-O-Al bonds, resulting in the destruction of aluminosilicate framework, the corroded layer can be considered as a partially dealcalized and decalcified Si/Al-rich residue, which is relatively hard and has potential to inhibit further corrosion by acting as a barrier to the acid ions provided that shrinkage cracks are minimized. The molecular structure of the corroded layer and the extent of damage to the aluminosilicate framework due to electrophilic attack by acid protons that is currently under investigation by using MAS-NMR and FTIR spectroscopy techniques will be discussed in the second part of this article.

The presence of calcium in the form of calcium silicate hydrate, is a disadvantage for the acid resistance of geopolymeric cements. Calcium silicate hydrate is vulnerable to attack by acidic solutions and dissolution of which by acids producing soluble salts, i.e. nitric acid, results in the formation of shrinkage cracks [23, 24, 25, 26] that would accelerate the transport of ions through the corroded layer. The extent of damage therefore is dependent on the amount of calcium silicate hydrate and it is expected that a decrease in the calcium content of geopolymeric cements will result in a higher acid resistance.

The structure of aluminosilicate framework of geopolymeric cements is very similar to that of aluminosilicate glasses. Addition of limited amounts of alumina to alkali-silicate and soda-lime-silicate glasses for improving their chemical durability is a well known

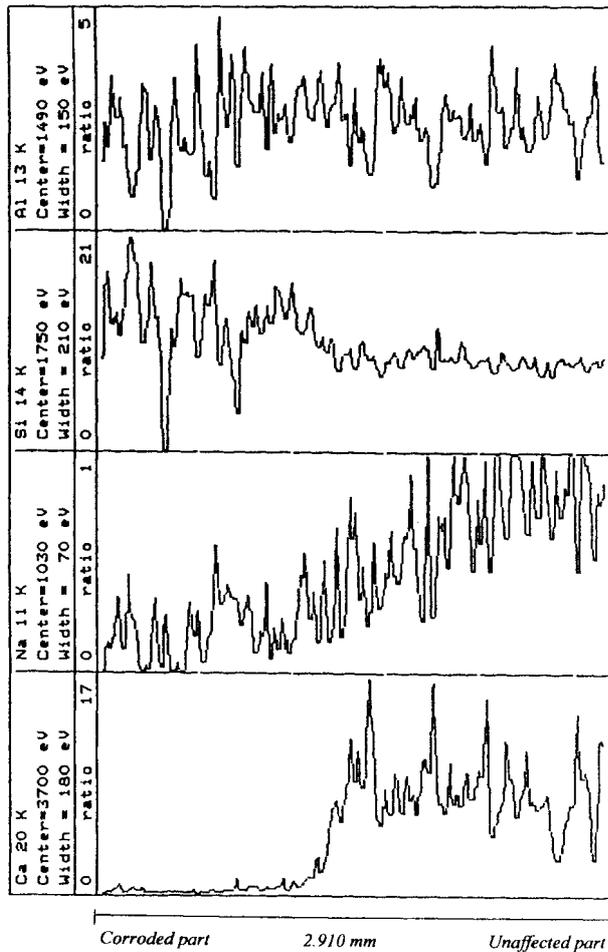


Figure 9. X-ray line analysis (EDS) of the specimen after 60 days of exposure to nitric acid at $pH = 2$ (magnification = $40\times$).

practice [27,28]. In simple alkali-silicate glasses, all modifier cations are charge compensated by non-bridging oxygens. Presence of non-bridging oxygens lowers the effective crosslink density of the glass and create Q_3 and Q_2 sites which are more prone to network hydrolysis and allows formation of silanol groups via ion exchange. It is widely accepted that in aluminosilicate glasses the modifier cations are associated to both AlO_4^- tetrahedra (or AlO_6^{3-} octahedra) and non-bridging oxygens [27, 28, 29]. Anionic tetrahedral sites of AlO_4^- modify not only the extended structure of the glass, but also its inherent reactivity. The modifier cations associated to AlO_4^- are approximately five orders of magnitude more resistant to ion exchange by protons than those associated to non-bridging oxygens [30, 31]. Chemically durable sodium-aluminosilicate glasses provide excellent matrices for the immobilization of radioactive fission products [32].

The phenomenon of acidic corrosion of aluminosilicate glasses has not been studied thoroughly. However it should be noted that in spite of similarities between geopolymeric cements and aluminosilicate

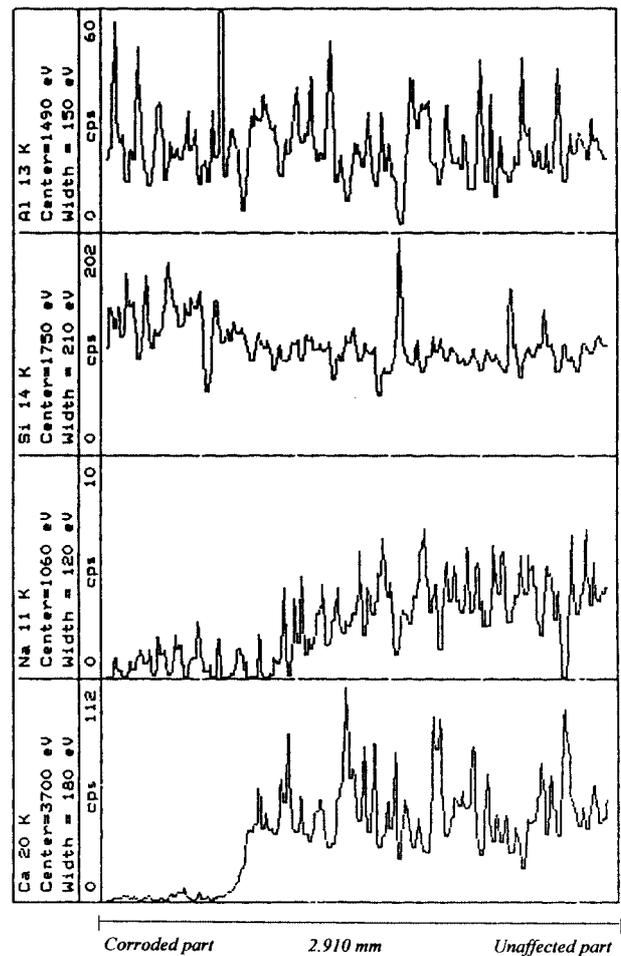


Figure 10. X-ray line analysis (EDS) of the specimen after 60 days of exposure to nitric acid at $pH = 3$ (magnification = $100\times$).

glasses, the process of their acidic corrosion can be greatly influenced by differences in their chemical and microstructural homogeneity and pore content. Aluminosilicate glasses are relatively homogenous in both chemical composition and microstructure with zero pore content, whereas geopolymeric cements are relatively heterogeneous with a comparatively very high pore content which effectively accelerates the process of acidic corrosion.

Attack at $pH = 2$ and 3

As confirmed by visual observations, scanning electron microscopy, and X-ray line analyses, the mechanism of corrosion at pH values of 2 and 3 is exactly the same as that at $pH = 1$. The only difference is the severity of the attack that decreases significantly at higher pH values. In fact at higher pH values of nitric acid, the soluble contents of specimens, i.e. sodium and calcium, are leached not only to a lower depth but also to a lesser extent. Figures 9 and 10 show concentration profiles of typical X-ray line analyses performed on

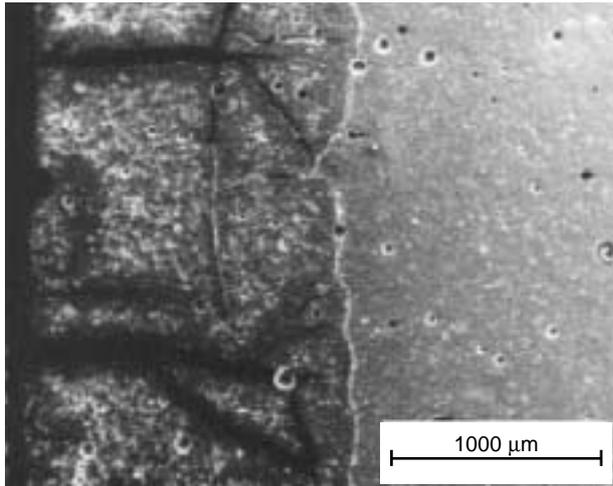


Figure 11. Corroded layer developed after 60 days exposure to $pH = 2$ nitric acid.

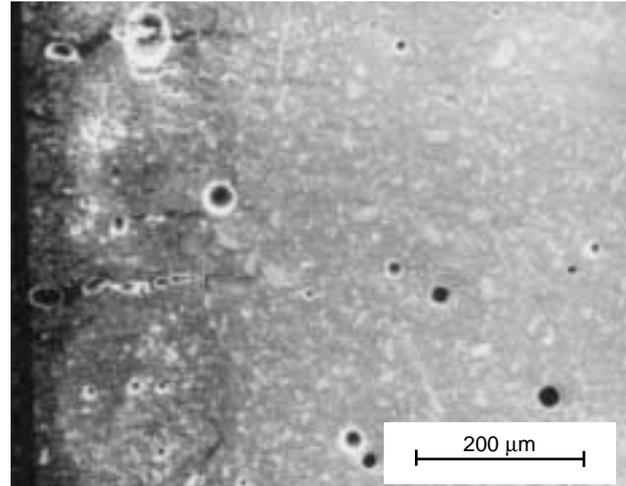


Figure 12. Corroded layer developed after 60 days exposure to $pH = 3$ nitric acid.

specimens after 60 days exposure to nitric acid at pH values of 2 and 3 respectively. The lines are extended from somewhere in the corroded layers on the left side through corrosion zones and toward the unaffected parts of the specimens on the right side. The total lengths of lines at magnifications of $40\times$ and $100\times$ respectively are 2.910 mm and 1.164 mm. As seen in the profiles of sodium and calcium, partial dissolution is less noticeable at higher pH values of 2 and 3 confirming that the considerably lower acidic strengths at pH values of 2 and in particular 3 significantly reduce the rate and the extent of dissolution of sodium and calcium in a relatively low porosity matrix. Partial dissolution of sodium and calcium at higher pH values of 2 and 3 results in the formation of significantly lesser and finer shrinkage cracks as shown in figures 11 and 12 ;

CONCLUSIONS

1. Geopolymeric cements are less vulnerable to nitric acid attack.
2. The mechanism of attack consists of a leaching process in which sodium and calcium ions are depleted and exchanged by H^+ or H_3O^+ ions from the solution and an electrophilic attack by acidic protons on polymeric Si-O-Al bonds (to be discussed in Part 2) resulting in the formation of a partially dealcalized and decalcified Si, Al-rich residue.
3. The volume contraction of the material during the leaching process results in the formation of shrinkage cracks. These shrinkage cracks can effectively lower the inhibiting effect of the corroded layer by making the transport of ions easier. It is expected that a decrease in the soluble content (sodium and calcium) of geopolymeric cements will result in a higher acid resistance.

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KOROZE ZATVRDLÝCH KAŠÍ GEOPOLYMERNÍCH
CEMENTŮ ROZTOKY HNO₃,
ČÁST 1

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Geopolymerní cement (alkalicky aktivovaný popílek, příp. ve směsi s granulovanou vysokopecní struskou) představuje anorganické pojivo s vysokou odolností vůči kyselinové korozi. Alkalickou aktivací popílku (působením směsi vodního skla a NaOH) vznikají amorfní hydratované alumosilikáty sodné podobné zeolitům. V práci byla studována koroze zatvrdlých geopolymerních cementů roztoky HNO₃ při $pH = 1 - 3$. Korozní produkty byly studovány RTG diffrakcí, IČ a NMR v pevné fázi. Koroze kaší zatvrdlých geopolymerních cementů roztoky HNO₃ způsobuje proces rozpouštění rozpustných částí materiálu (Na,Ca), kdy se vytváří vrstva se zvýšenou koncentrací Si/Al. Diskuse dosažených výsledků a navržený mechanismus koroze bude uveden v části 2.