

Chemically and Thermally Activated Illite Clay from Latvia

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Abstract — Materials were synthesized from illite based clay from Latvia by chemical and thermal activation using both NaOH and KOH solutions (4–6 M). Compressive strength and apparent porosity were measured. Effect of concentration of both solutions on the mechanical properties of the material was investigated by means of infrared spectroscopy (IR). Compressive strength data showed that alternative building materials could be obtained via alkaline activation.

Keywords — Alkaline activation, compressive strength, illite clay.

I. INTRODUCTION

Illite clays, due to their high natural occurrence and colloidal properties, play an important role in the rheological behavior of soils and granulates used in construction materials, particularly in concrete.

Information on structural and chemical characteristics of clay containing minerals is essential for prediction of possible interactions between clay minerals and concentrated alkaline solutions and for derivation of relations between their chemical and physicochemical properties on one side and the sorption properties on the other. Such relations are of high importance as they allow a rational development of sorbents and advanced structural materials. Illite clay minerals are phyllosilicates containing one $[\text{Al}_2(\text{OH})_4]^{2+}$ octahedral layer sandwiched between two $(\text{Si}_2\text{O}_5)^{2-}$ tetrahedral sheets [1]. As aluminosilicates, dioctahedral clay minerals react in strong alkaline aqueous solutions and consequently amorphous 3-dimensional frameworks consisting of linked $[\text{SiO}_4]^{2-}$ and $[\text{AlO}_4]^{5-}$ tetrahedra form [2]–[3]. The charge deficit is balanced by cations (Na^+ , K^+ , Ca^{2+} , etc.) of the base that is used. NaOH and KOH are commonly used as alkaline activators [4]. Traditionally, $\text{Ca}(\text{OH})_2$ has also been used. Clay particles are strongly anisotropic and have faces and edges that are very different by surface area and in chemical behavior [1].

Reaction products of clays or other reactive aluminosilicates treated with highly alkaline solutions are often referred to as geopolymers, alkali-activated cements or hydroceramics. They have been described as zeolite-like, poorly-crystalline or amorphous alkali aluminosilicates with strong cementing properties [4]. Such materials require less energy for their manufacture and have higher sustainability. In fact, geopolymer technology has the potential to reduce emissions even by 80 % compared, for instance, with those of Portland cement technology. From geological point of view Latvia is rich in clay resources to be used for material production (currently and, at least, for centuries) [5].

The aim of this work was to continue [6] the study of alkaline dissolution and geopolymerisation of natural Quaternary illite clay from Latvia.

II. MATERIALS AND METHODS

Quaternary illite clay from Latvia which was used for alkaline and thermal activation was characterized by means of chemical analysis and X-ray diffraction (model *Rigaku*, Japan, with CuK_α radiation at a 2θ scanning interval from 10° to 60° and speed $4^\circ/\text{min.}$). In order to investigate the effect of the activators (NaOH and KOH) on the properties of the material, two series of tests were made. Sodium and potassium hydroxide solutions were obtained by dissolving dried pellets with 99 % purity in distilled water. 4–6 M activator solutions were added to clay powders and mechanically mixed. Afterwards cylindrical samples ($h = 25$ mm, $d = 16$ mm) were made from plastic clay mass with 25–30 % average moisture content. One set of samples were cured at 60°C , 80°C or 100°C temperature for 4 h or 24 h and then were allowed to mature at room temperature for 28 days. Another set of samples were allowed to mature at room temperature for 7 days to increase their mechanical strength. Afterwards samples were thermally activated at 600°C , 700°C or 800°C temperature. Finished samples were used for mechanical strength tests and apparent porosity measurements. Compressive strength was determined by means of “*Compression Test Plant ToniNorm*” *Toni-Technic* by *Zwick* (300 kN). Fourier transform infrared spectroscopy was carried out on materials using “*21 Prestige, Shimadzu Corp*” FTIR spectrophotometer in transmittance mode. FTIR spectra were gathered between 400 cm^{-1} and 1700 cm^{-1} [6].

III. RESULTS AND DISCUSSION

Table I shows chemical composition of the studied clay. In order to obtain high mechanical strength, Davidovits [7] recommends the use of $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio from 2.5 up to 4.5. In this case $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio was 4.0. The Al_2O_3 and CaO are regarded as more significant elements than others, because AlO_4^- and silicate oligomers combine to form geopolymers [8]. The presence of calcium provides extra nucleation sites for precipitation of dissolved species, which may be used to tailor setting times and material properties [9]. CaO content of source material appears to strengthen the geopolymer by forming amorphously structured Ca-Al-Si gel during geopolymerisation [10].

TABLE I
CHEMICAL COMPOSITION OF THE STUDIED CLAY, WT%

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	LOI*
Q	51.0	12.9	6.8	0.6	6.7	4.0	0.5	3.8	13.7

* LOI – loss on ignition at 1000 °C

X-ray analysis showed that studied clay contains clearly visible reflections of quartz (SiO₂) and illite (KAl₂AlSi₃O₁₀(OH)₂·H₂O). Illite is an aluminosilicate mineral that reacts in alkaline solutions. Its presence in clay is therefore a significant feature that affects geopolymerisation [11]. Reflections of calcite (CaCO₃), and dolomite (CaMg(CO₃)₂) were observed in the clay as well. Previous studies have shown that the addition of moderate amount of calcium-containing material can have a significant effect on the structure and properties of the final material [12]–[13]. Besides clay also contains feldspar – plagioclase (CaNa(AlSi₃O₈)). It is observed that not only alkali metal cations in alkaline solutions, but also alkali metals structured in Al-Si containing materials affect the formation of the gel phase and the geopolymerisation [14].

It is known that the cation involved in the activation process could affect the development of the structure. In general it can be stated that OH⁻ ions act as a reaction catalysts, but alkaline metal cations act as a structure forming element that balances the negative framework charge carried by tetrahedral aluminium.

Fig. 1, Fig. 2 and Fig. 3 show the role of KOH and NaOH as chemical activator on the compressive strength of final materials obtained from the studied clay and matured at different temperatures.

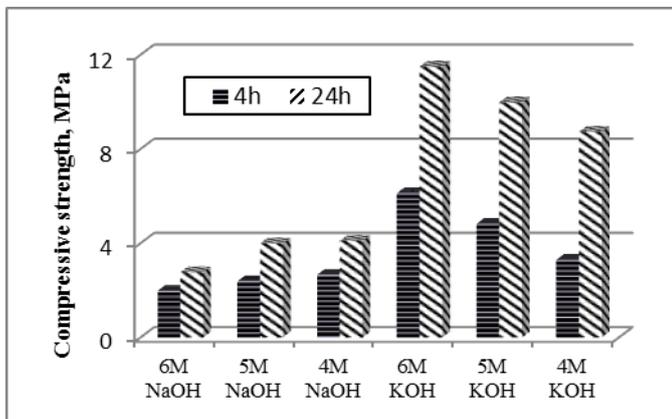


Fig. 1. Dependence of compressive strength of materials on KOH and NaOH concentration for materials matured at 60 °C for 4 h and 24 h.

Compressive strength of materials obtained by chemical activation process and matured at 60 °C temperature was higher when KOH solution was used as an activator. It was stated that maturing time had an essential influence on the results — compressive strength was 2–2.5 times higher when maturing time was prolonged from 4 h to 24 h. KOH solution with the highest concentration used (6 M) provided the highest strength enhancement (reaching 11.5 MPa). Using 5 M and 4 M KOH solutions for activation, compressive strength values were 10.0 MPa and 8.7 MPa, respectively.

Compressive strength of materials obtained in chemical activation process and matured at 80 °C temperature differed from results shown in Fig. 1. There was no essential difference among compressive strength results using either NaOH or KOH solutions as chemical activators. Activation using NaOH solutions gave a bit higher results in comparison to activation using KOH solution. The highest compressive strength value (10.5 MPa) was obtained when 6 M NaOH solution was used. Obviously, higher concentration of NaOH solution facilitated the dissociation of different silicate and aluminate species promoting further polymerization that resulted in higher compressive strength.

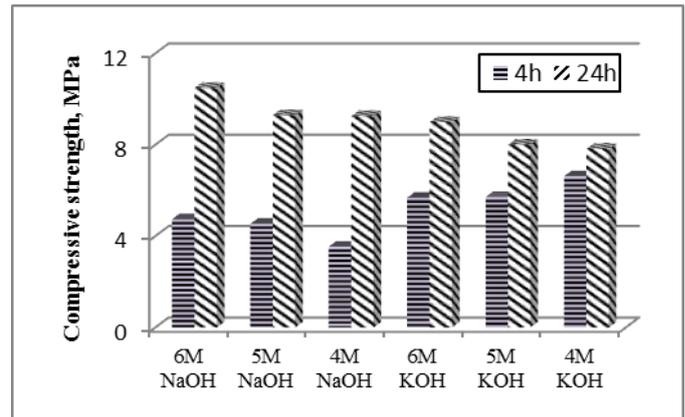


Fig. 2. Dependence of compressive strength of materials on KOH and NaOH concentration for materials matured at 80 °C for 4 h and 24 h.

It was stated that maturing time in this case had more essential influence on the results when NaOH solution was used — the compressive strength was 2–2.2 times higher when maturing time was prolonged from 4 h to 24 h.

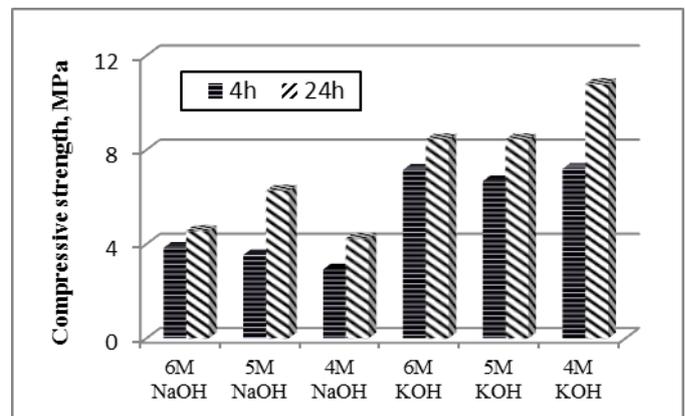


Fig. 3. Dependence of compressive strength of materials on KOH and NaOH concentration for materials matured at 100 °C for 4 h and 24 h.

Compressive strength of materials obtained in chemical activation process and matured at 100 °C temperature was higher when KOH solution was used as an activator. Such thermal activation conditions promoted initial strength when the materials were obtained by maturing samples for 4 h. Prolongation of maturing time from 4 h to 24 h had more effect on material when 4 M KOH was used as a chemical activator. When alkaline solutions with higher concentration were used, the connectivity of silicate anions was reduced; this

resulted in poor polymerization and lower compressive strength values.

X-ray diffractograms of materials formed from differently activated clay are shown in Fig. 4. Diffractograms exhibited characteristic peaks of quartz, illite and calcite; this means that these minerals were not dissolved during chemical activation. The most intensive halo peak was registered between 26° and $28^\circ 2\theta$ and was attributed to amorphous phase consisting of SiO_4 tetrahedra sharing oxygen atoms and lacking any long-range order. This halo peak indicates the formation of amorphous aluminosilicate phase [15].

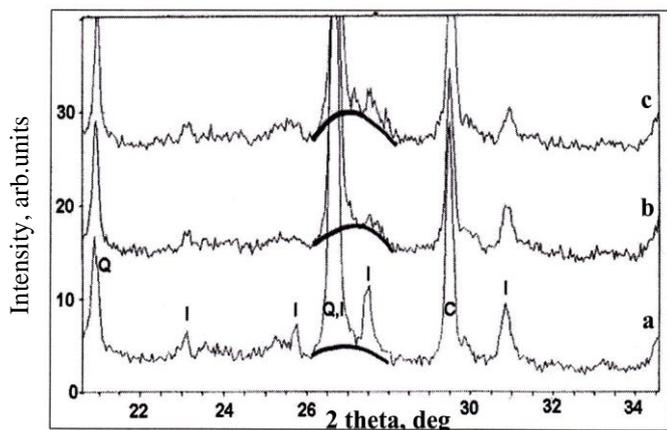


Fig. 4. XRD patterns of: a — unactivated clay, b — clay chemically activated using 4 M KOH and maturated at 100°C , c — clay chemically activated using 6 M KOH and maturated at 60°C ; Q — quartz, I — illite, C — calcite.

More intensive halo peak in comparison to chemically untreated clay (Fig. 4 a) appeared in X-ray diffraction pattern of material obtained from clay that was chemically activated using 6 M KOH and maturated at 60°C temperature (Fig. 4 c). These results confirmed the results obtained on compressive strength tests.

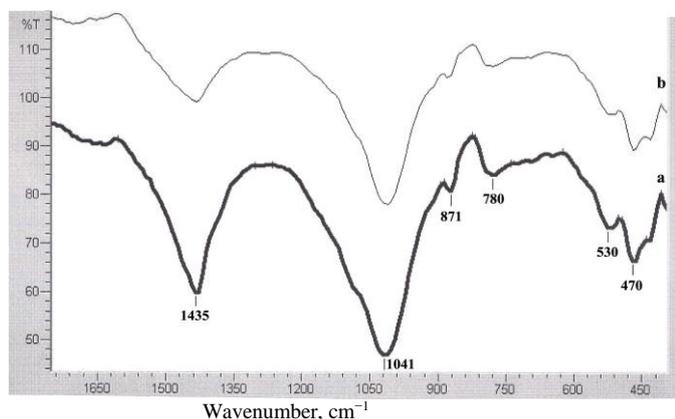


Fig. 5. FTIR spectra of materials: a — chemically activated with 6 M KOH solution and maturated at 60°C , b — chemically activated with 4 M KOH and maturated at 100°C .

FTIR spectra of different chemically activated and maturated materials show very similar absorption frequencies. Band located at 1435 cm^{-1} was attributed to stretching vibrations of O-C-O bond. This indicates the presence of potassium

bicarbonate, which is suggested to occur due to the atmospheric carbonation of highly alkaline KOH aqueous phase that is diffused on the surface of geopolymeric materials [16]. Normally during geopolymerization process Al transfers to fourth coordination forming $[\text{Al}(\text{OH})_4]^-$, and the alkaline element maintains charge balance in the geopolymer. If the geopolymerization is incomplete, excess K^+ is deposited on the pore surfaces forming a salt. When exposed to air, potassium and sodium are naturally transformed into bicarbonates [17]. The formation of bicarbonate species could be explained by the highly basic nature of materials and the presence of unreacted raw material. The band at 1041 cm^{-1} was attributed to the Si-O bond stretching in the unreacted clay. The frequency at 871 cm^{-1} also is related to CO_3^{2-} [18]. The absorption bands at 780 cm^{-1} are attributed to symmetric vibrations of Si-O-Al and Si-O-Si bonding of AlO_4 and SiO_4 tetrahedrons [15], [19], [20] confirming that the material has undergone geopolymerization. The small band seen at approximately 530 cm^{-1} is mainly due to the symmetric stretching vibrations of Si-O-Si and Al-O-Si which is related to the formation of amorphous to semi-crystalline aluminosilicate materials [21]. The band at 470 cm^{-1} is due to in plane Si-O bending and Al-O linkages originating from within individual tetrahedra [22].

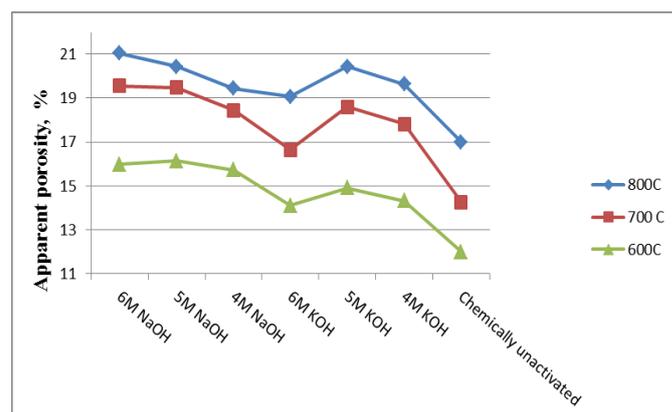


Fig. 6. Dependence of apparent porosity of materials on the concentrations of KOH and NaOH after thermal treatment at different temperatures.

Dependence of evolution of apparent porosity together with the thermal treatment temperature increase on alkalinity of KOH and NaOH solutions is shown in Fig. 6. Porosity increased together with temperature for all materials obtained via chemical activation. Material obtained from clay activated with 6 M NaOH solution and thermally treated at 800°C temperature had the highest porosity value (21.0 %). If lower alkalinity NaOH solution was used for chemical activation, porosity decreased reaching 20.4 % and 19.4 %; These materials had higher porosity than materials obtained from unactivated clay at the same temperature (17.0 %). If KOH was used as an activator, material chemically activated with 5 M solution (21.0 %) had the highest porosity values; the lowest — if 6 M KOH solution (19.1 %) was used.

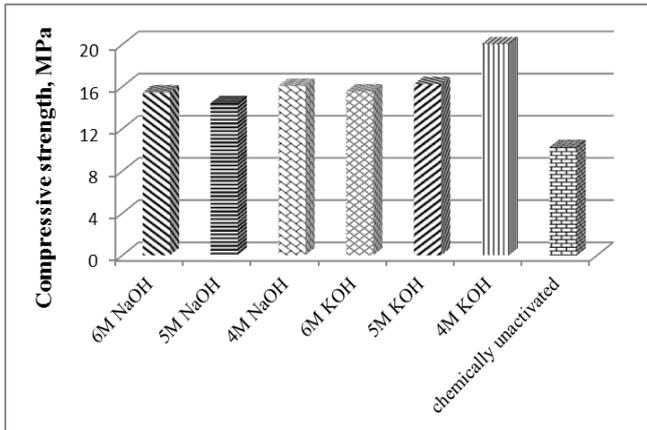


Fig. 7. Compressive strength evolution depending on KOH and NaOH concentration, after thermal treatment at 700 °C.

Results presented in Fig. 7 and Fig. 8 show the effect of both used solutions on the compressive strength of materials obtained by thermal treatment process.

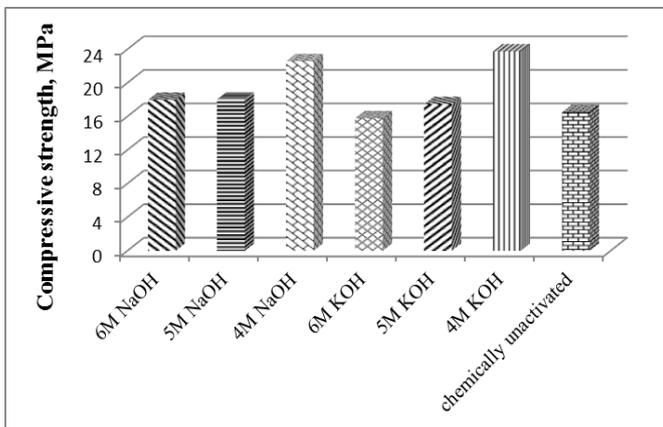


Fig. 8. Compressive strength evolution depending on KOH and NaOH concentration, after thermal treatment at 800 °C.

Compressive strength increased together with increase of thermal treatment temperature for all obtained materials. Besides that, the strength increased along with the decrease of alkalinity of both NaOH and KOH solutions. In other words, lower $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{K}_2\text{O}/\text{Al}_2\text{O}_3$ ratios increased the strength in comparison to higher ratios in case of this clay. If higher than 4 M concentrations of KOH or NaOH solutions were used for chemical activation, the excess hydroxide ion concentration caused aluminosilicate gel precipitation at very early stages, resulting in lower compressive strength. The material activated with 4 M KOH and thermally treated at 800 °C had the highest compressive strength (23.8 MPa) which is 1.5 times higher than that of a chemically untreated clay (16.0 MPa) after thermal treatment at the same temperature.

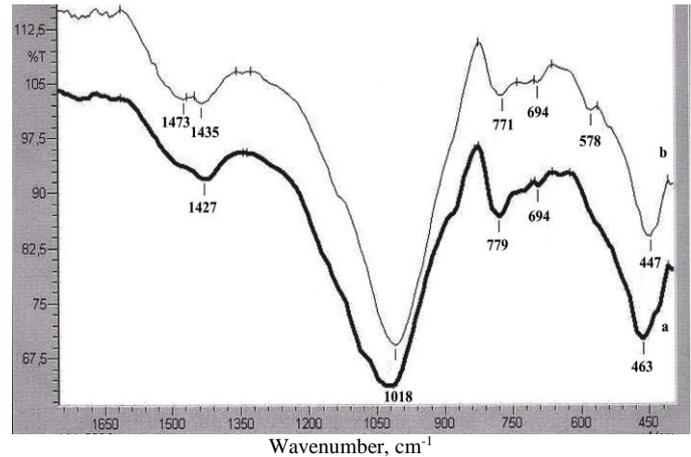


Fig. 9. FTIR spectra of materials thermally treated at 800 °C: a — chemically unactivated; b — activated using 4 M KOH solution.

Differences in absorption frequencies between unactivated clay and clay chemically activated using 4 M KOH predicts transformations taking place during material synthesis. The bands at 463 cm^{-1} seen in spectrum of specimen from chemically unactivated clay are due to bending vibrations of Si-O-Si and O-Si-O bonds [23]. These bands shift to lower frequencies (approximately 448 cm^{-1}) in spectrum for chemically activated specimen. The bands at 694 cm^{-1} are due to the presence of quartz [24]. Characteristic band at 1033 cm^{-1} has been assigned to asymmetric stretching of Al-O and Si-O bonds originating from within individual tetrahedra. This band shifts to lower frequencies (1011 cm^{-1}) in FTIR spectra of activated material indicating the formation of a new product (the amorphous aluminosilicate gel phase) [25]. Moreover, the structural reorganization of clay is evidenced by the disappearance of the absorption band at approximately 779 cm^{-1} and the appearance of a new band at lower frequencies (approximately 771 cm^{-1}) which is assigned to the symmetric stretching vibrations of Si-O-Si and Al-O-Si (referring to the formation of amorphous to semi-crystalline aluminosilicate materials) [19]–[21]. Two new vibration bands appear at 578 cm^{-1} and 1474 cm^{-1} ; these are typical for semi-crystalline materials [26].

Alkali metal cations had a catalytic role and could have contributed to strong structure formations. It was shown that clay had higher compressive strength after activation mainly in KOH solution rather than in NaOH solution. KOH provides more inorganic polymer precursors compared to NaOH thus stimulating higher compressive strength.

IV. CONCLUSION

It can be concluded that the values of properties achieved after chemical and thermal activation are a highly non-linear function of numerous variables. The main factors affecting the synthesis of materials from illite clay from Latvia include the type of alkali, concentration and especially thermal treatment temperature. Material from Quaternary clay from Latvia, ($\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio 4.0) has the best compressive strength of approximately 24 MPa, if 4 M KOH and thermal treatment at 800 °C temperature is used to obtain the material. This

temperature is 200° lower than temperature used in factories to obtain ceramics for construction.

The present study showed that illite based Quaternary clay from Latvia could be a source material for synthesis of inorganic polymers.

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Ingunda Šperberga, Polīna Špeļa, Māris Rundāns, Andris Cimmers. Ķīmiski un termiski aktivēts Latvijas illīta māls.

Rakstā aplūkota Latvijas kvartāra illīta māla ķīmiska aktivēšana ar dažādas koncentrācijas KOH un NaOH šķīdumiem un tā piemērotība materiālu izstrādei dažādās temperatūrās (60–80 °C) un (600–800 °C). Illīts un plagioklāzs ir alumosilikāti, kas var reaģēt sārmainos apstākļos; to klātbūtnei mālos ir svarīga loma ģeopolimerizācijas procesā. Kā galvenais rādītājs ģeopolimerizācijas procesa sekmīgai norisei izvēlēta iegūto materiālu spiedes stiprība, kas pieaug līdz ar temperatūras paaugstināšanu (no 600 °C līdz 800 °C) karbonātus saturošos kvartāra mālos. Kā aktivētājs tiek izmantots KOH vai NaOH šķīdums. Šo mālu materiālu augstākā spiedes stiprība (23,8 MPa) tiek sasniegta apstrādājot 800 °C, ja kā ķīmiskais aktivators tiek izmantots 4 M KOH šķīdums. Ķīmiskās aktivācijas procesā ar NaOH šķīdumu tiek iegūti materiāli, kuru spiedes stiprības rādītāji ir zemāki par rādītājiem, kas iegūti izmantojot KOH šķīdumu. Tomēr salīdzinot ar ķīmiski neaktivēta māla spiedes stiprību (16 MPa), šajā gadījumā ir iegūts materiāls, kura spiedes stiprība ir 1,5 reizes lielāka (22 MPa) — kā šķīdums ķīmiskajai aktivācijai tiek izmantots 4 M NaOH un termiskās aktivācijas temperatūra ir 800 °C. Pētījumi parādīja, ka viena vai otra sārmu hidroksīda izmantošana ģeopolimerizācijas procesā deva dažādus rezultātus. Iegūto materiālu īpašības bija atkarīgas no vairākiem faktoriem, no kuriem svarīgākais bija termiskās apstrādes temperatūra. Sakarā ar katjonu katalītisko lomu, svarīgs ir katjons, kas ietilpst hidroksīda (NaOH vai KOH) sastāvā un ar kuru māls tiek ķīmiski aktivēts. Iegūto materiālu varētu izmantot kā tradicionālo būvmateriālu aizstājēju. Pētītais materiāls tiek iegūts zemākā temperatūrā, bet tas nemazina tā spiedes stiprību, salīdzinot ar tradicionāliem būvmateriāliem.