

## Kaolin-based geopolymers with various NaOH concentrations

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**Abstract:** Kaolin geopolymers were produced by the alkali-activation of kaolin with an activator solution (a mixture of NaOH and sodium silicate solutions). The NaOH solution was prepared at a concentration of 6–14 mol/L and was mixed with the sodium silicate solution at a  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 0.24 to prepare an activator solution. The kaolin-to-activator solution mass ratio used was 0.80. This paper aimed to analyze the effect of NaOH concentration on the compressive strength of kaolin geopolymers at 80°C for 1, 2, and 3 d. Kaolin geopolymers were stable in water, and strength results showed that the kaolin binder had adequate compressive strength with 12 mol/L of NaOH concentration. When the NaOH concentration increased, the  $\text{SiO}_2/\text{Na}_2\text{O}$  decreased. The increased  $\text{Na}_2\text{O}$  content enhanced the dissolution of kaolin as shown in X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) analyses. However, excess in this content was not beneficial for the strength development of kaolin geopolymers. In addition, there was the formation of more geopolymeric gel in 12 mol/L samples. The XRD pattern of the samples showed a higher amorphous content and a more geopolymer bonding existed as proved by FTIR analysis.

**Keywords:** geopolymers; kaolin; compressive strength; sodium hydroxide

## 1. Introduction

The history of alkali-activated cements has started since 1940's. Davidovits prepared mineral polymers by dissolving aluminosilicate oxide, alkali, and colloidal silica sol or alkali polysilicate in water and heated up to a temperature of 120°C. This process is related to the synthesis of zeolites or molecular sieves that has characteristic 3D frameworks with successions of tetrahedron  $\text{TO}_4$ , where T is aluminum, silicon, and so on. Previously, Davidovits has found that the synthesis of plastic, feldspathoids, and zeolites was influenced by hydrothermal conditions requiring high alkalinity, concentrated alkali, atmospheric pressure, and thermoset at temperatures below 150°C [1]. However, the products are very porous and have poor mechanical properties. Later, Davidovits has successfully invented a

novel mineral polymer with hard surfaces, thermal stability and high surface smoothness, and precision [2].

In 1978, Davidovits coined the term “geopolymers” to describe the mineral polymers in amorphous to semicrystalline three-dimensional silico-aluminate structure [1]. They have the empirical formula of  $\text{M}_n\{-(\text{SiO}_2)_z-\text{AlO}_2\} \cdot w\text{H}_2\text{O}$ , where M is a cation ( $\text{K}^+$ ,  $\text{Na}^+$ , or  $\text{Ca}^{2+}$ ),  $n$  is a degree of polycondensation, and  $z$  is 1, 2, and 3. Formerly, various names have been used to describe these materials, such as alkali-bounded ceramics, hydroceramics, and alkali-activated cements. According to Ref. [3], the most suitable term to describe these materials is “inorganic polymers”.

Following the work of Davidovits, many researchers have started works on these alkali-activated systems.

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Later, Palomo *et al.* [4] established two models of alkali-activated cement systems that attract attention from all over the world. These two cements are known as a typical representation of the alkali-activated cements. The alkali-activated system includes the activation of blast furnace slag (Si + Ca) with mild alkaline solution and the activation of metakaolin or fly ash (Si + Al) with medium to high alkaline solution. The first system has calcium silicate hydrate (CSH) as main reaction products, while the second system has zeolite-like polymers as main products.

Geopolymers are synthesized by the polycondensation of silico-aluminate structure. Highly alkaline solutes, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), are incorporated into source materials rich in SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> [5]. The geopolymer binders show good bonding properties and utilize materials, such as fly ash or metakaolin as the source of silicon and aluminum for alkali reaction. Aluminosilicate kaolinite reacts with the NaOH solution and polycondenses into hydrated sodalite or hydro-sodalite [6].

Kaolinite materials have been used as an aluminosilicate oxide source to synthesize geopolymer products [6-8]. The aluminosilicate is the main structure forming species to the overall geopolymerization process. According to previous studies [8-9], the addition of kaolinite to others aluminosilicate sources is necessary. However, kaolinite possesses a slower rate of Al dissolution and requires sufficient time for interactions among the source materials.

The reaction is similar for kaolin and metakaolin in the process of solidification [10]. The only difference between them is that kaolin experiences alkaline attack that starts on the outer faces of the kaolinite crystal, and it continues layer by layer from the edges to the insides. On the other hand, alkali attack on metakaolin starts at the edges, upper, and lower surfaces of the amorphous region. The metakaolin's surface area of reaction is higher than that of kaolin, hence leading to higher reactivity and faster hardening for the metakaolin geopolymer [6]. In this study, kaolin was chosen as the aluminosilicate sources. Although the use of kaolinite in addition to other aluminosilicate sources, such as fly ash, has been reported previously [11], there is no detailed study on the use of kaolin alone in geopolymer synthesis. Kaolin was chosen to investigate how much kaolin can stand alone in geopolymer synthesis as well as to eliminate the complex interpretation of results as result of the utilization of complex raw material (e.g., fly ash) with the presence of impurities.

Inorganic polymer concretes have emerged as novel engineering materials with the potential to form a substantial element of environmentally sustainable construction and building products. Extensive researches have been carried out [11-16] for the development of geopolymers. Parameters, such as NaOH concentration and curing regime, are important parameters that must be taken into consideration when designing a kaolin-based geopolymer product for a specific application [11, 17].

The main objective of this paper was to study the effect of various NaOH concentrations (6-14 mol/L) on the compressive strength of kaolin geopolymers at 80°C for 1, 2, and 3 d. The scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR) analyses were also presented and discussed.

## 2. Experimental

### 2.1. Materials

Kaolin was supplied by Associated Kaolin Industries Sendirian Berhad, Malaysia. The general chemical composition is tabulated in Table 1. The physical form of kaolin used was powder type and has minimum 40% of particles sized less than 2 µm and maximum 2% of moisture content. It was used as the Si-Al cementitious materials.

A technical grade Na<sub>2</sub>SiO<sub>3</sub> solution (waterglass) was supplied by South Pacific Chemicals Industries Sendirian Berhad (SPCI), Malaysia, with the chemical composition of 30.1% SiO<sub>2</sub>, 9.4% Na<sub>2</sub>O, and 60.5% H<sub>2</sub>O with the modulus SiO<sub>2</sub>/Na<sub>2</sub>O of 3.2, density at 20°C of 1.4 g/cm<sup>3</sup>, and viscosity at 20°C of 0.4 Pa·s. The NaOH pellet was caustic soda micropearls, 99% purity under the brand name of Formosoda-P, and was made in Taiwan. Distilled water was used throughout the experiment.

### 2.2. Sample preparation

NaOH solutions of desired concentrations of 6-14 mol/L were prepared and cooled down to room temperature. NaOH solutions with varying concentrations were mixed with a Na<sub>2</sub>SiO<sub>3</sub> solution to prepare a liquid alkali activator at 24 h prior to use. The ratio of Na<sub>2</sub>SiO<sub>3</sub> to NaOH used was 0.24 by mass [18]. Kaolin powder was mixed well with the alkali activator at a mass ratio of 0.80 for few minutes by mixer. The detail of mixture proportions and the important oxide molar ratios calculated is given in Table 2. The fresh paste was then rapidly poured into 50 mm × 50 mm × 50 mm steel molds; the samples were compacted approximately one half of the depth (about 25 mm) of the

Table 1. Chemical composition of kaolin

wt%

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	K <sub>2</sub> O	Na <sub>2</sub> O	MgO
50.00-52.00	33.00-35.00	0.60-1.00	0.50-0.90	< 0.05	1.50-2.00	0.01-0.05	0.30-0.70

Table 2. Detail of mixture proportion

NaOH molarity / ( $\text{mol}\cdot\text{L}^{-1}$ )	Molar ratios			
	$\text{SiO}_2/\text{Al}_2\text{O}_3$	$\text{SiO}_2/\text{Na}_2\text{O}$	$\text{H}_2\text{O}/\text{Na}_2\text{O}$	$\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$
6	3.28	3.58	19.53	0.92
8	3.28	2.78	14.36	1.18
10	3.28	2.36	11.69	1.39
12	3.28	2.07	9.86	1.58
14	3.28	1.87	8.53	1.76

mold in the entire cube compartments; and the paste was tamped in each cube compartment at each layer as described in ASTM C109 [19]. Finally, the samples were put into an oven at  $80^\circ\text{C}$  for up to 3 d for curing purpose. The samples were sealed with thin plastic layers during the curing stage to prevent moisture loss.

### 2.3. Testing and analysis methods

The physical observation of kaolin geopolymers was performed by immersing the kaolin geopolymers in water. Observation was recorded for 28 d.

The bulk density of the geopolymer samples was measured according to BS EN12390-7 [20]. Bulk density measurement was carried out on samples with different NaOH concentrations at day 3 of curing.

Compressive strength tests of all specimens were carried out according to ASTM C109/C109M-08 [19] by using an Instron machine series 5569 mechanical tester. A minimum of three specimens of each concentration was taken out from oven at days 1, 2, and 3 and examined by compression test to evaluate the early strength gain for the specimens.

JSM-6460LA model scanning electron microscope (JEOL) analyses was performed to reveal the microstructure of kaolin geopolymers and to observe the different degrees of reaction at different NaOH concentrations. The specimens were cut into small pieces and coated by auto fine coater, model JEOL JFC 1600, before the examination. Energy dispersive X-ray spectroscopy (EDX) of the kaolin geopolymers was also performed.

Samples were prepared in powder form and undergone XRD examination. XRD-6000, Shimadzu X-ray diffractometer equipped with auto-search/match software, as standard to aid qualitative analysis, was used.

A Perkin Elmer FTIR spectrum RX1 spectrometer was used to evaluate the functional group of the samples. A small amount of potassium bromide (KBr) and geopolymer powder was put into a mould. By a cold press machine, mould, which contains powder and KBr, was pressed at 39.2 kN for 2 min to produce specimens for examination.

## 3. Results and discussion

### 3.1. Physical observation

Fig. 1 shows the states of kaolin geopolymers after im-

mersion in water for 28 d. All kaolin geopolymers did not disintegrate in water at 28 d except for 1 d cured samples with 10 mol/L of NaOH concentration. This sample showed slight disintegration at 28 d. This observation was complied with the very low compressive strength obtained (Fig. 2). For other kaolin geopolymer samples, no crack was observed. This suggested the stability of kaolin geopolymers in water.

### 3.2. Bulk density

Fig. 2 displays bulk density of kaolin geopolymers at day 3. The samples for the 12 mol/L of NaOH concentration showed the highest bulk density than the other samples. These bulk densities measured complied with the compressive strength results in which a higher compressive strength shows a higher bulk density.

### 3.3. Compressive strength

Compressive strength development of kaolin geopolymers with NaOH concentration (6, 8, 10, 12, and 14 mol/L) after 1, 2, and 3 d curing in an oven at  $80^\circ\text{C}$  is shown in Fig. 2. Generally, the compressive strength increased as the specimens were cured for a longer period. Kaolin geopolymers cured for 3 d exhibited a higher strength than 1 and 2 d cured samples but with no definite trend.

According to researchers [8, 21–22], geopolymerization takes place in three steps: (1) dissolution of aluminosilicate sources in alkaline solution, (2) reorganization and diffusion of dissolved ions with the formation of small coagulated structure, and (3) polycondensation of soluble species to form hydrated products. Due to the low reactivity of kaolin itself, the dissolution process was extremely slow and only occurred on the surface particles of kaolin. The former was clearly shown by the slow strength gain at days 1 and 2, while the latter was obviously revealed in microstructures in Section 3.4. It was believed that the marked increase in strength at day 3 is related to the dissolution of ions in kaolin. It was estimated that the slow reactivity of kaolin provides insufficient dissolved ions for further reorganization and polycondensation reaction to form hydrated products and thus leads to a sudden increase in strength at day 3.

Curing time played important roles in both acceleration of chemical reaction and determination of the extent of reaction [23]. Prolonging the curing time at  $80^\circ\text{C}$  of the geopolymer mixture improved the geopolymerization process, yielding a higher compressive strength [16, 23].

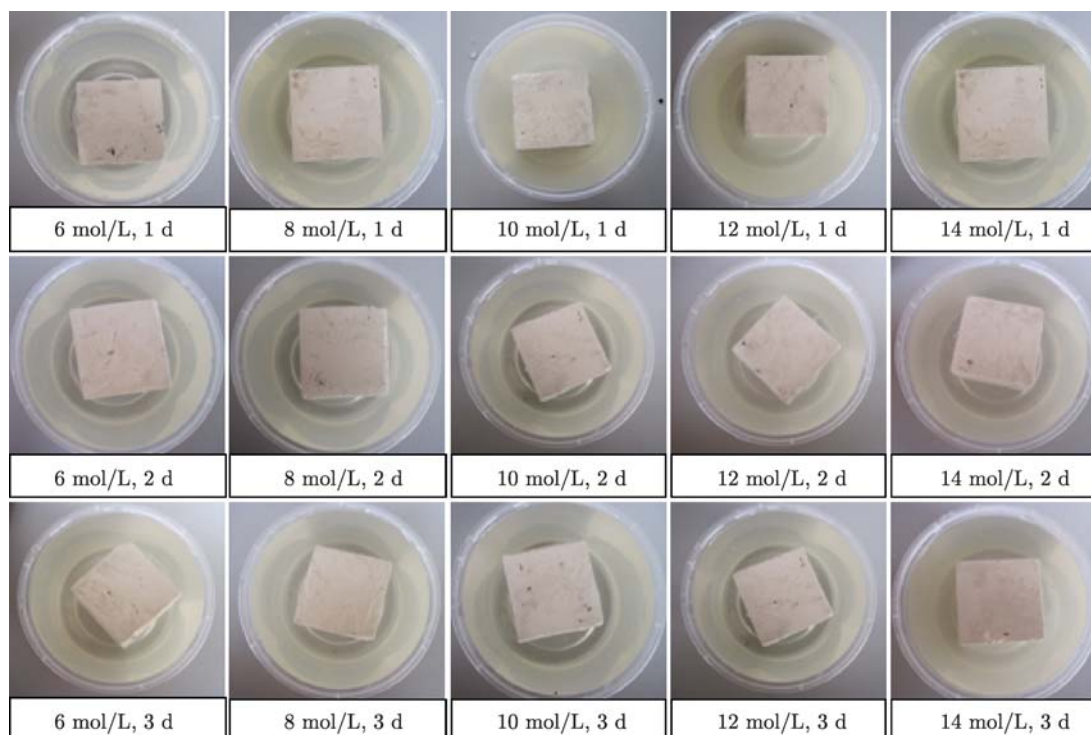


Fig. 1. States of kaolin geopolymers after immersion in water for 28 d.

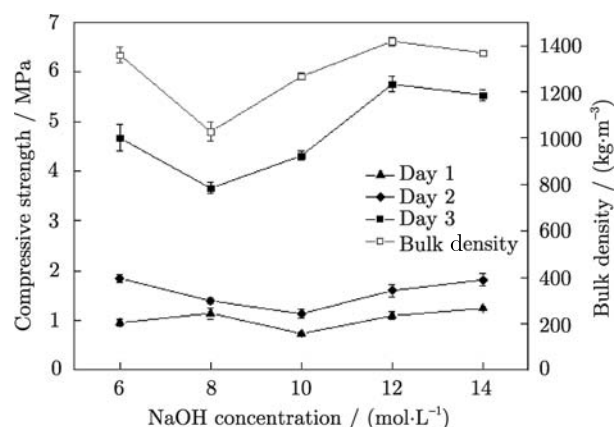


Fig. 2. Compressive strength of kaolin geopolymers at days 1, 2, and 3 and bulk density of kaolin geopolymers at day 3 with various NaOH concentrations.

From the results, geopolymers showed an increasing trend of the compressive strength after 1, 2, and 3 d of curing. For 1 and 2 d cured specimens, there was insignificant development of strength gain. 3 d cured samples with 12 mol/L NaOH solution showed a higher (optimum) compressive strength than the others. This implied that there is an optimum alkalinity (Na concentration) for activating kaolin, which was influenced by the NaOH solution [14–15]. However, the compressive strength decreased when the NaOH concentration was increased up to 14 mol/L. It was probably because of excess of  $\text{Na}^+$  ions [16]. In the geopolymerization process, the function of Na cations is for

the balancing of negative charges created by the formation of Si–O–Al bonding or nonbridging oxygen ions remained in the system, while  $\text{OH}^-$  is consumed during the hydrolysis of kaolin. In high concentration of NaOH solution, there were more  $\text{Na}^+$  and  $\text{OH}^-$  ions present. As kaolin has low reactivity, the rates of ion dissolution and strength development were slow. Thus, when higher concentration of NaOH solution was used, the system, in comparison, contained only very less dissolved ions and hydrated products but high  $\text{Na}^+$  contents. In other words, there was insufficient formation of Si–O–Al bonding or nonbridging oxygen ions to be charge-balanced by large amount of  $\text{Na}^+$  ions. In addition, from Table 2, the  $\text{SiO}_2/\text{Na}_2\text{O}$  molar ratio decreased at high NaOH concentration. The aluminosilicate sources and  $\text{Na}_2\text{SiO}_3$  solution contributed the  $\text{SiO}_2$  content, while the  $\text{Na}_2\text{O}$  content was contributed by the  $\text{Na}_2\text{SiO}_3$  solution and the NaOH solution. At constant solid/liquid (S/L) and  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio, the  $\text{Na}_2\text{O}$  content increased as the NaOH concentration was increased. Although the  $\text{Na}_2\text{O}$  content may have improved the solubility of the aluminosilicate source, this excess  $\text{Na}_2\text{O}$  content in the system could seriously weaken the structure formed.

On the other hand, kaolin geopolymers with 6 mol/L of NaOH solution showed a higher strength gain on day 2 than the others. This strength result was unexpected and was most probably because the 6 mol/L mix has excess mixing water due to a lower NaOH concentration. 3 d cured samples with 6 mol/L of NaOH solution showed

a slightly higher strength than those with 8 and 10 mol/L but lower than those with 12 and 14 mol/L. In comparison with high concentration of NaOH solution, 6 mol/L of NaOH solution contained more water as shown by a higher  $\text{H}_2\text{O}/\text{Na}_2\text{O}$  molar ratio in Table 2. The compressive strength of the samples with 6 mol/L was expected lower than that of higher molarity samples, owing to their lower alkalinity, which indicates a lower dissolution of kaolin. However, the compressive strength measured here was slightly higher than those of 8 and 10 mol/L. This suggested that water plays an important role in easing the transportation of dissolved ions during the process of geopolymerization.

This showed that kaolin geopolymers were affected by NaOH concentration, water content, and curing regimes. Nevertheless, the optimum compressive strength obtained was 5.75 MPa with the 12 mol/L NaOH concentration. Kaolin geopolymers exhibit low reactivity and forms weak structure, thus contributes to slow compressive strength development of geopolymers, which agreed with previous studies [8-9].

### 3.4. Microstructure of geopolymers

Fig. 3 shows the morphological features of pure kaolin, and Figs. 4 and 5 show the changes in morphologies of geopolymer pastes for various concentrations at 1 and 3 d of curing. Overall, the activation of kaolin occurred at the surface of kaolin particles, as shown in Figs. 4 and 5. Kaolin geopolymers revealed that the partially reacted kaolin particles coexist with the unreacted kaolin particles. Similar to the activation of metakaolin, the activation of kaolin with alkaline solution occurred at the surface of kaolin particles [22]. However, the microstructural comparison of metakaolin geopolymers and kaolin geopolymers showed that metakaolin geopolymers contain more intervening and denser structure with lesser unreacted particles [24-25].

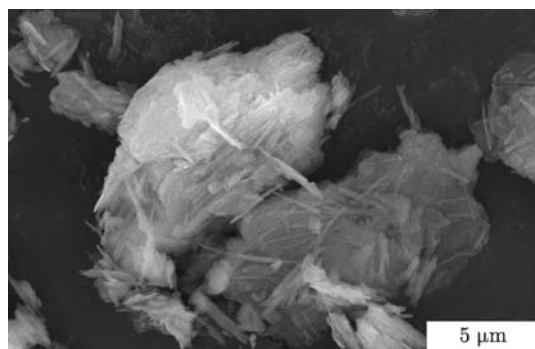


Fig. 3. SEM micrograph of kaolin.

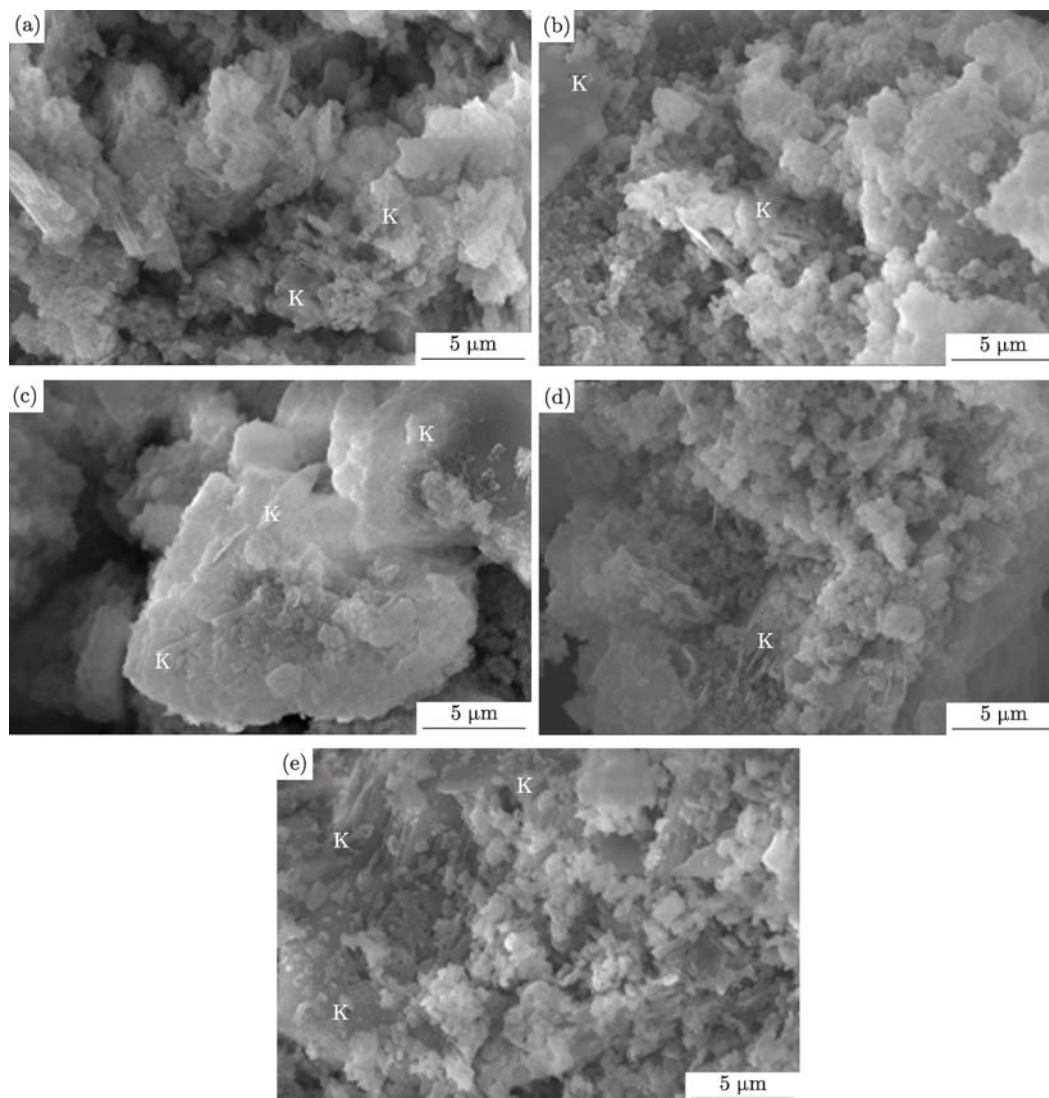
Differences in microstructures could be distinguished at various NaOH concentrations. It was clear that the morphology of pure kaolin crystals is plate-like stacking to-

gether [26], as seen in Fig. 3. The microstructure showed a fairly smooth surface with some pores distributed all over. This was agreed with results reported by Varga [27] and disagreed with those by Sun *et al.* [24], which shows sphere-shaped metakaolin particles. This could be due to the effect of testing conditions. The kaolin particles changed in edges when activated with alkaline solution [26]. After activation with alkaline activating solution, the geopolymeric gel (spherical aggregates) formed, suggesting that the structure experiences growth. This meant that the activating solution has dissolved the kaolin to release  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  for geopolymerization reaction to take place.

On 1 d cured kaolin geopolymer samples, the formation of geopolymer gel on the kaolin surface was observed. However, it was clear that kaolin geopolymers with 10 mol/L of NaOH solution displayed the least formation of geopolymer gel. Large portion of unreacted raw materials could be seen. This microstructure was agreed with compressive strength recorded in Fig. 2.

On 3 d curing, samples with 12 mol/L of NaOH solution appeared to have more geopolymeric gel than other samples, which contribute to the highest compressive strength at day 3, which is 5.75 MPa. The microstructures of the samples with 12 mol/L of NaOH solution were more homogeneous and contained less unreacted particles compared to others. This showed that the alkaline activation is more effective with 12 mol/L of NaOH concentration. However, there was a large part of unreacted kaolin which could still be observed in all samples, which was also observed through XRD and FTIR analyses. The microstructures of kaolin-NaOH activated samples showed loose-grained structure with unreacted clay particles compared with those of fly ash-based and metakaolin-based geopolymers. These imperfect microstructures of kaolin geopolymers would be one of the main causes of poor compressive strength [13].

Strength may be increased if the unreacted part reacted to form a denser structure. When 8 mol/L of alkaline solution was used, the degree of reaction was the lowest. This was displayed by the slight surface activation of particles with few partially reacted particles and a large amount of unreacted particles. The degree of reaction for the samples with 6 mol/L of NaOH solution was slightly higher compared to those with 8 mol/L of NaOH solution. This was because 6 mol/L of NaOH solution has a higher water content as stated above. The water eased the geopolymerization process, leading to a higher transportation of ions, and hence, the micrograph showed a denser structure. Conversely, when 14 mol/L of NaOH solution was employed, geopolymeric gel was slightly lesser. This might probably because of the excess of  $\text{Na}^+$ .



**Fig. 4.** SEM micrographs of kaolin geopolymers using NaOH concentrations of 6 mol/L (a), 8 mol/L (b), 10 mol/L (c), 12 mol/L (d), and 14 mol/L (e) at day 1 of curing (K sites show unreacted kaolin zones).

Fig. 6 shows the EDX spectra of kaolin and kaolin geopolymers with 12 mol/L of NaOH solution. The chemical compositions of the kaolin and kaolin geopolymers are summarized in Table 3. The main elements in kaolin geopolymers were Si, Al, and Na. These elements were in good agreement with their respective components. The Si/Al ratio of kaolin geopolymers increased when compared to that of kaolin after the incorporation of the activator solution [28]. Furthermore, the Na content was also increased in kaolin geopolymers.

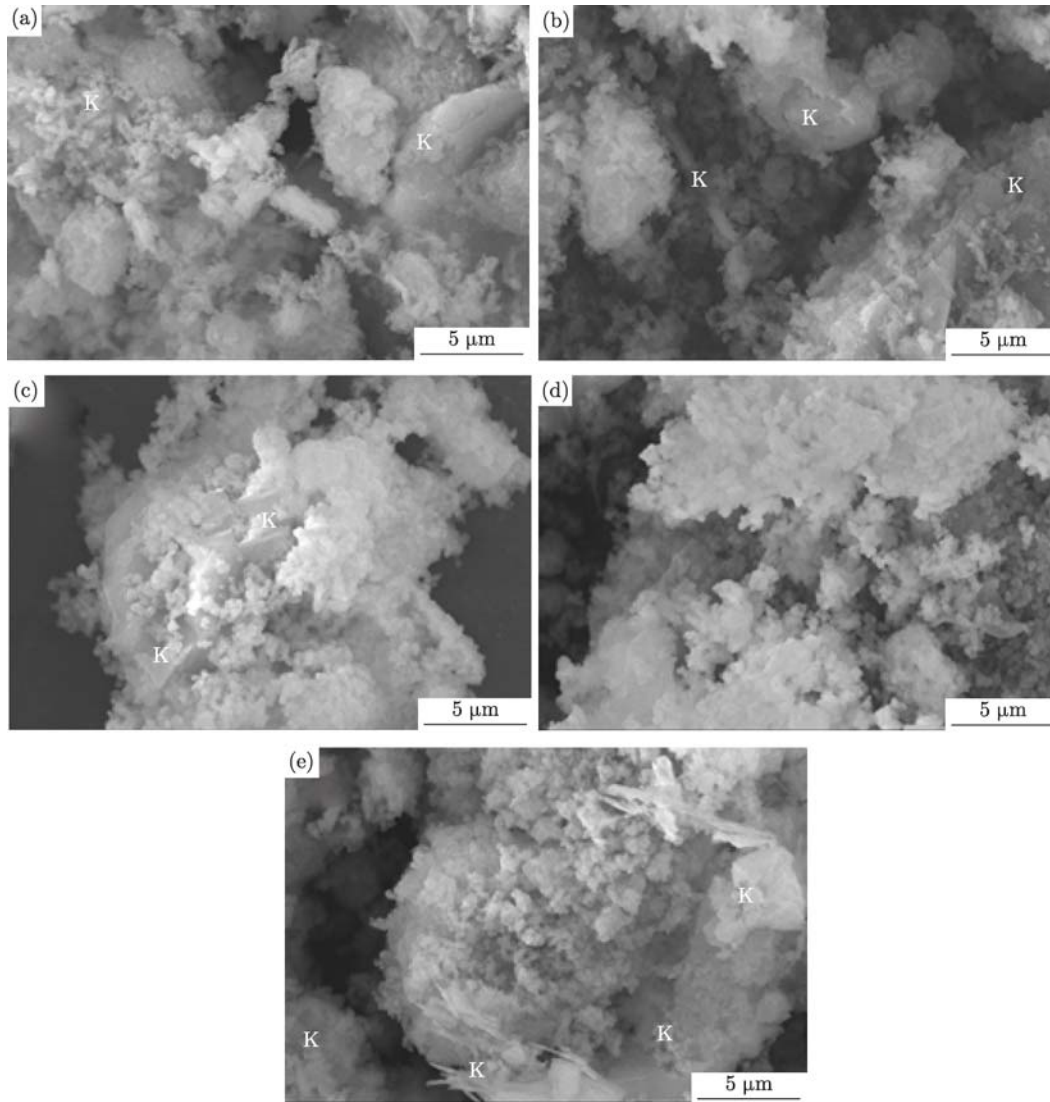
### 3.5. XRD analyses

XRD patterns in Fig. 7 showed that kaolin composed of kaolinite (K) as major minerals and some dickite (D) and quartz (Q). Illite (I) and alunite (A) could be found in trace amount. A number of characteristic kaolinite peaks could be seen in spectra of geopolymer samples. The char-

acteristic kaolinite peaks are at  $2\theta$  values of  $12.3^\circ$ ,  $19.8^\circ$ ,  $24.9^\circ$ ,  $45.4^\circ$ ,  $55.1^\circ$ , and  $62.2^\circ$  [13]. These kaolinite peaks in all geopolymer products decreased in intensity with the increase in NaOH concentration. This meant that the dissolution ability was better with the increase of NaOH concentration. The quartz reflection peak ( $\sim 26.5^\circ$ ) was still remained in the system, suggesting that it did not take part in the geopolymerization process, but its intensities were slightly lower due to a dilution effect [29].

In general, XRD patterns of geopolymer samples showed that a large part of unreacted materials remains. Small intensities in the XRD patterns of geopolymer products between  $18^\circ$  and  $25^\circ$  indicated that the geopolymer products have amorphous structure [29]. After alkaline activation, the peak shifted to the right. The slightly higher  $2\theta$  value was related to the formation of the amorphous sodium alumino-silicates gel. This is the main binding





**Fig. 5.** SEM micrographs of kaolin geopolymers using NaOH concentration of 6 mol/L (a), 8 mol/L (b), 10 mol/L (c), 12 mol/L (d), and 14 mol/L (e) at day 3 of curing (K sites show unreacted kaolin zones).

**Table 3.** Chemical compositions of kaolin and kaolin geopolymers

Element	Kaolin	Kaolin geopolymers (12 mol/L NaOH concentration at 3 d)	wt%
Si	26.36	26.64	
Al	18.33	17.39	
Na	0.20	2.47	
Si:Al	1.44	1.53	

phase [30]. Peaks at  $2\theta$  values of  $14^\circ$ ,  $32^\circ$ ,  $35^\circ$ ,  $43.5^\circ$ ,  $50.5^\circ$ ,  $52.5^\circ$ ,  $59^\circ$ , and  $61^\circ$  were corresponding to zeolite A (ICDD# 71-0370) [31]. This result was contradicted with the work done by Chandrasekhar and Pramada [32], who have found the formation of zeolite X when kaolin was activated by alkaline solution. On the other hand, hydrosodalite was obtained in the work by Heller-Kallai and Lapides [33]. This phase was higher in intensity at low

NaOH concentration and decreased with increasing NaOH concentration.

According to previous research [30], the higher the concentration of NaOH solution, the greater the amorphous content of the reaction products. However, the samples with 12 mol/L of NaOH solution showed a higher amorphous content of geopolymer products that agree with the higher compressive strength than others. Previous

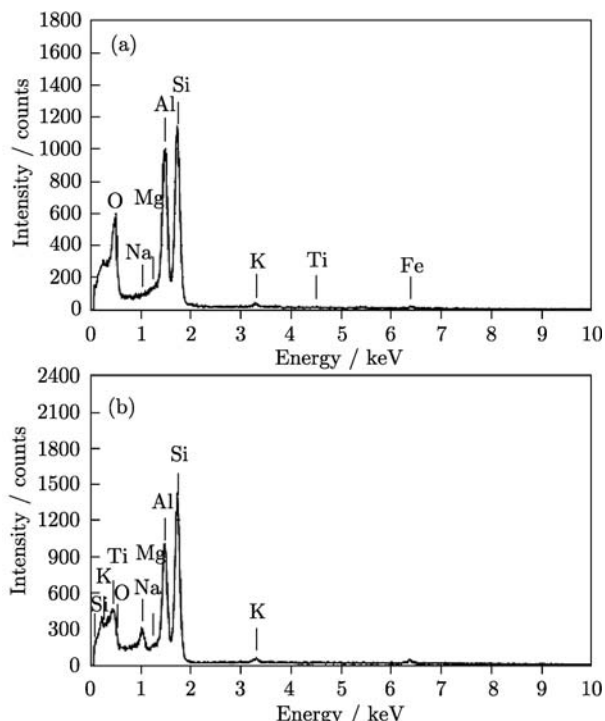


Fig. 6. EDX spectra of kaolin (a) and kaolin geopolymers (b) using 12 mol/L of NaOH concentration at day 3.

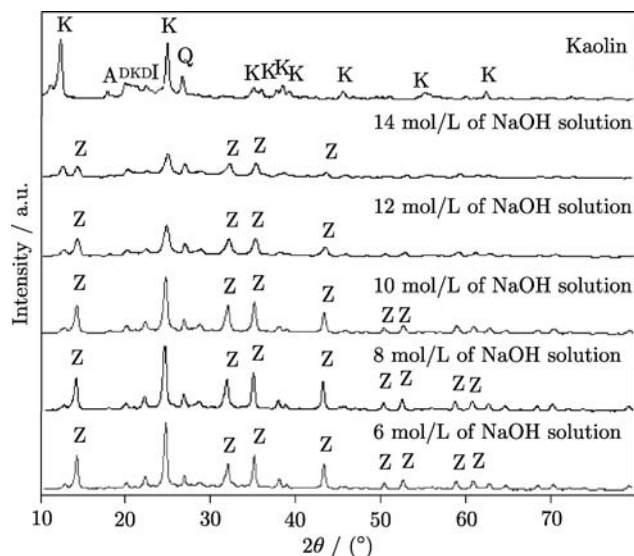


Fig. 7. XRD patterns of kaolin and geopolymer products at day 3 (K, kaolinite; Q, quartz; A, alunite; D, dickite; I, illite; and Z, zeolite).

researchers also reported the diffuse halo peak at around  $30^\circ$  ( $2\theta$ ) that related to the typical amorphous character of geopolymers [29], which complies with the theory where the major feature of XRD patterns of geopolymers is a largely featureless 'hump' centered at approximately  $27^\circ$ – $29^\circ$  ( $2\theta$ ) [34]. High-resolution microscopy of geopolymeric

systems has shown this gel phase to be present largely in the form of nano-sized alumino-silicate particles [35–36]. Reaction between the activation solution and kaolin will increase with properly increasing curing temperature. However, the higher temperature will cause a decline in compressive strength, due to the vaporization of mixing water [29].

### 3.6. Fourier transform infrared spectroscopy (FTIR)

Fig. 8 displays the FTIR spectra of kaolin and geopolymer products synthesized using  $\text{Na}_2\text{SiO}_3$  solution and various NaOH concentrations (6–14 mol/L) for day-3-cured samples. That transformation took place during synthesis was indicated by the different absorption frequencies of kaolin and the synthesized geopolymers [18]. This was also shown in XRD patterns (Fig. 7).

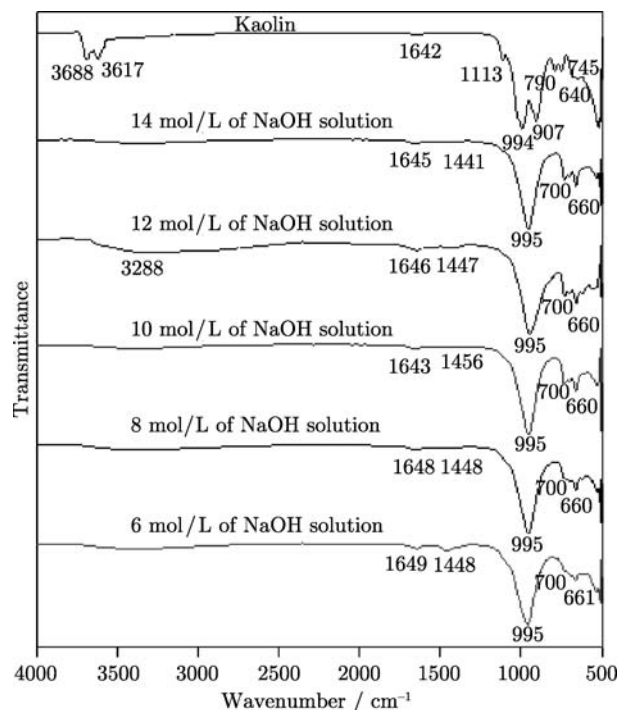


Fig. 8. FTIR spectra of kaolin and kaolin geopolymers at various NaOH solutions on day 3.

In the FTIR spectrum of kaolin, the peak around  $1113\text{ cm}^{-1}$  was attributed to Si–O vibration in  $\text{SiO}_4$  molecules, which vanished after geopolymerisation reaction. Also, a weak band of Si–O symmetrically stretching vibration was observed at  $640\text{ cm}^{-1}$ . Absorptions at  $995\text{ cm}^{-1}$  and  $790\text{ cm}^{-1}$  were assigned as alternating Si–O and Al–O bonds and Al–O stretching vibration in  $\text{AlO}_4$  tetrahedra, respectively. A shift of the asymmetric bending of the bonds O–Si–O and O–Al–O to lower frequencies could be observed, which confirmed the previous finding [6, 18, 37].

FTIR spectra of geopolymers showed similar



wavenumber regardless of NaOH concentration but with different intensities. The main band analyzed in the FTIR spectrum of geopolymers was in the region of 900-1300  $\text{cm}^{-1}$ , corresponding to the Si-O-T linkages. These Si-O-T linkages indicated the geopolymer bonding (T = Al or Si) [32-33]. This peak was the lowest in the samples with 6 mol/L of NaOH concentration and the highest in those with 12 mol/L of NaOH concentration, suggesting a more geopolymer bonding reflected in the measured strength. Other major bands were broad bands at 3000-3500  $\text{cm}^{-1}$  and 1650-1655  $\text{cm}^{-1}$ , which were the stretching and deformation vibration of OH and H-O-H groups from water molecules. A band at around 1400  $\text{cm}^{-1}$  was assigned to the Si-O-Si stretching, and bands at around 700  $\text{cm}^{-1}$  and 660  $\text{cm}^{-1}$  showed the characteristic of the formed amorphous polymer, which is the Si-O-Si and Si-O-Al symmetric stretching. Si-O-Si bonds are stronger than Si-O-Al bonds. These Si-O-Si bonds were absent in all day-1-cured samples except for the cured kaolin geopolymers with 14 mol/L of NaOH concentration. However, Si-O-Si bonds were present in all the 3 d cured samples and were the highest in the samples with 12 mol/L of NaOH concentration. These clearly proved the highest compressive strength of the samples. Peaks at 537  $\text{cm}^{-1}$  originated from Si-O-Al bonds, where Al was present in octahedral coordinate [29]. A band at 790  $\text{cm}^{-1}$  disappeared after the geopolymerization process. This band was then replaced by lower frequency bands at 700  $\text{cm}^{-1}$  and 537  $\text{cm}^{-1}$ .

These wavenumbers shifted from kaolin, suggesting that there are changes in chemical bonding taking place in the system. There is only little difference between FTIR spectra of kaolin and geopolymers synthesized within 500-1000  $\text{cm}^{-1}$ , suggesting that most part of unreacted kaolin still retains in the geopolymers synthesized [6]. The principal band associated with Si-O(Al) stretching vibration in  $\text{SiO}_4$  tetrahedra near 1000  $\text{cm}^{-1}$  was very broad.

## 4. Conclusions

This paper investigated the effect of NaOH concentration on the synthesis of kaolin geopolymers. NaOH concentration has significant effect on the compressive strength of geopolymers. Strength maximized at an optimal concentration and a higher concentration does not favor geopolymer formation in the case of kaolin geopolymers. Results from this investigation concluded the following.

(1) Kaolin geopolymers showed stability in water with no disintegration and cracks.

(2) Kaolin geopolymers exhibited different rates of strength development. The optimum strength was 5.75 MPa for the samples prepared with 12 mol/L of NaOH concentration and cured for 3 d.

(3) Bulk density of kaolin geopolymers complied with the measured compressive strength.

(4) The  $\text{Na}_2\text{O}$  content improved the dissolution of kaolin materials. However, the high  $\text{Na}_2\text{O}$  content degraded the strength of the kaolin geopolymers.

(5) No obvious increase in the formation of geopolymer gel was observed. And kaolin has been activated by the activator solution with various NaOH concentrations, showing that 12 mol/L of NaOH solution activated the system the most.

(6) XRD patterns indicated that the samples with 12 mol/L of NaOH solution presented a higher amorphous content of geopolymer products, which complies with the compressive strength measured.

(7) FTIR spectra of kaolin and geopolymer products synthesized showed changes in chemical bonding and most part of unreacted kaolin still retained in the geopolymers at day 3 curing.

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