EFECTUL CONCENTRAȚIEI ALCALIILOR ASUPRA PROPRIETĂȚILOR MECANICE ALE GEOPOLIMERILOR PE BAZĂ DE CAOLIN

EFFECT OF ALKALI CONCENTRATION ON MECHANICAL PROPERTIES OF KAOLIN GEOPOLYMERS

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Alkali concentration is the most significant factor in geopolymerization process that must be taken into consideration during the synthesis of kaolin geopolymers. The kaolin geopolymers were prepared by mixing kaolin and alkali activator solution. The alkali activator solution used was mixture of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). This study aims to analyze the effect of NaOH concentration (6-14M) on mechanical properties of kaolin geopolymers. Compressive strength results showed that the optimum NaOH concentration is 8M. SEM, XRD and FTIR analysis were performed to study the transformation taken place during the geopolymer synthesis.

Concentrația alcaliilor este cel mai semnificativ factor al procesului de geopolimerizare, care trebuie luat în considerare în timpul sintezei geopolimerilor pe bază de caolin.

Geopolimerii pe bază de caolin au fost preparați prin amestecarea caolinului cu o soluție de activator alcalin. Soluția de activator alcalin utilizată a fost un amestec de hidroxid de sodiu (NaOH) şi silicat de sodiu (Na_2SiO_3). Prezentul studiu urmăreşte analizarea efectului concentrației de NaOH (6-14M) asupra proprietăților mecanice ale geopolimerului pe bază de caolin. Rezultatele rezistenței la compresiune au indicat ca optimă concentrația de NaOH 8M. Analizele SEM, XRD şi FTIR au fost realizate pentru studierea transformărilor care au loc în timpul sintezei de geopolimeirzare.

Keywords: *kaolin, geopolymers, alkali concentration, mechanical properties*

1.Introduction

Kaolin is a clay mineral that traditionally has been used in the manufacture of porcelain. Kaolin is a soft, lightweight, often chalk-like sedimentary rock that has an earthy odor. Kaolinite, the main mineral in kaolin, is formed by rock weathering. It is white, greyish-white, or slightly colored and formed mainly by decomposition of feldspars (potassium feldspars), granite, and aluminum silicates. Kaolinite is the main structure forming species in the overall geopolymerization process. Davidovits [1] used kaolinite and metakaolin obtained by thermal treatment at 750ºC for 6 hours as source of alumino-silicate oxides to synthesize and produce geopolymers. Other researchers [2, 3] have also focused on the manufacture of geopolymeric products and their industrial applications by using either kaolinite or metakaolinite as the main reactant.

Geopolymer is synthesized by the polycondensation of silico-aluminate structures. Highly alkaline solutes such as NaOH and KOH are incorporated with source materials rich in SiO_2 and Al_2O_3 [4] in order to produce geopolymers. Synthesis of geopolymer consists of three basic steps. The first is the dissolution of alumino-silicate under strong alkali solution; this is followed by reorientation of free ion clusters, and the last step is polycondensation [1].

Rowles & O'Connor [5] stated that, in the production of the inorganic polymer, the amount of Na^+ and OH^- are coupled, as the source of these ions is the NaOH present in the activating solution. In samples with low sodium content, there will be both insufficient OH^- to completely dissolve Si^{4+} and Al^{3+} from the source material and insufficient Na^+ to allow for complete polymerization of the network, hence both lead to lower strength of the resulted samples. Wang *et al.* [3, 6] have proven experimentally that the compressive strength and the content of the amorphous phase of metakaolinite-based geopolymers increase with an increase of NaOH concentration. This can be attributed to the enhanced dissolution of the metakaolinite particulates and hence the accelerated condensation of the monomer in the presence of the higher NaOH concentration. Granizo *et al.* [3] supported the idea that the alkali activation of metakaolin using solutions containing sodium silicate and NaOH results in the production of materials exhibiting higher mechanical strength compared to activation with only NaOH. Other researchers Rattanasak and Chindaprasirt [7] studied the influence of NaOH solution on the synthesis of fly ash geopolymer, the results revea-

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led that solubility of fly ash depends on concentration of NaOH and duration of mixing with NaOH where the use of 10M and 15M NaOH gave relatively high strength. As expected, 5M NaOH gave low strength due to low leaching of Si and Al ions in NaOH solution.

The development of kaolin as a new binder has not yet been explored. According to previous research on metakaolin-based geopolymers, Alonso and Palomo [8] have found that the rate of polymer formation is influenced by parameters such as curing temperature, alkali concentration, and initial solid content, among others. In the previous research, metakaolin was used instead of kaolin. Hence, the effect of NaOH concentration on the mechanical properties of kaolin-based geopolymers was may be assessed in this study.

2. Experimental Methods

2.1. Materials

NaOH powder used was made in Taiwan with a classification of caustic soda micropearls of 99% purity. The brand name is Formosoda-P. The sodium silicate (Na_2SiO_3) was of technical grade and was supplied by South Pacific Chemicals Industries Sdn. Bhd. (SPCI), Malaysia. The Na_2SiO_3 comprised of 30.1% SiO_2, 9.4% Na_2O and 60.5% H_2O with modulus SiO_2/Na_2O of 3.2, specific gravity at 20°C = 1.4 g/cm^3 and viscosity at 20°C = 0.4 Pa·s. Kaolin was supplied by Associated Kaolin Industries Sdn. Bhd., Malaysia. The general chemical composition of kaolin characterized using X-ray fluorescence (XRF) analysis is tabulated in Table 1. The physical form of the kaolin was powder type and has minimum 40% of the particle sized less than 2μm and maximum 2% of moisture content. Distilled water was used throughout the experimental.

2.2. Sample Preparation

NaOH solutions were prepared with molar concentration ranging from 6-14M and were cooled down to room temperature. The NaOH solution was mixed with Na_2SiO_3 solution at Na_2SiO_3-to-NaOH ratio of 0.24, by mass, to prepare liquid alkali activator 24 h prior to use. Then, kaolin was mixed well with alkali activator solution at kaolin-to-activator solution ratio of 0.80, by mass, for few minutes by using mechanical mixer.

Table 1

Compoziția chimică a caolinului
Chemical Composition of Kaolin

Element / *Element*	Greutate / *Weight* (%)
SiO_2	54.0
Al_2O_3	31.7
Fe_2O_3	4.89
TiO_2	1.41
ZrO_2	0.10
K_2O	6.05
MnO_2	0.11
LOI	1.74

Table 2 represents the Summary of SiO_2/Al_2O_3, SiO_2/Na_2O, H_2O/Na_2O and Al_2O_3/Na_2O molar ratios and water-to-geopolymer solids, by mass at different NaOH concentrations. The fresh paste was then rapidly poured into 50-mm steel moulds and the samples were compacted approximately one half of the depth (about 1 in. or [25 mm]) of the mould in the entire cube compartments and the paste was tamped in each cube compartment at each layer. The samples were put into the oven at 80°C for 24 h for curing purpose and later were kept in room temperature until the day of testing. The samples were sealed with thin plastic at exposed portion of the mould during curing.

2.3. Compressive Testing

Compressive strength testing of all specimens was performed by using the Instron machine series 5569 Mechanical Tester. After curing, the samples were taken out from oven and were kept in room temperature until the day of testing. Compression test was carried out to evaluate the strength development for the specimens. The samples were compressed at 1, 3, 7, 28, 60, and 180 days.

2.4. Scanning Electron Microscope (SEM)

JSM-6460LA model Scanning Electron Microscope (JEOL) was performed to reveal the microstructure of kaolin geopolymers and changes in microstructure based on various NaOH concentrations. The specimens were prepared and were coated by using Auto Fine Coater; model JEOL JFC 1600 prior to examination.

Table 2

Summary of SiO_2/Al_2O_3, SiO_2/Na_2O, H_2O/Na_2O and Al_2O_3/Na_2O molar ratios and water-to-geopolymer solids, by mass at different NaOH concentrations / *Rezumatul SiO_2/Al_2O_3, SiO_2/Na_2O, H_2O/Na_2O şi raporturi Al_2O_3/Na_2O Molar şi apă-geopolymer solide, de masă la concentrații diferite de NaOH*

NaOH Molarity *Molaritatea NaOH*	Molar Ratios / *Rapoarte molare*				Water-to-geopolymer solids, by mass / *Apă față de geopolimerul solid, în greutate*
	SiO_2/Al_2O_3	SiO_2/Na_2O	H_2O/Na_2O	Al_2O_3/Na_2O	
6M	3.28	3.58	19.53	1.09	0.74
8M	3.28	2.78	14.36	0.85	0.65
10M	3.28	2.36	11.69	0.72	0.59
12M	3.28	2.07	9.86	0.63	0.54
14M	3.28	1.87	8.53	0.57	0.50

2.5. X-ray Diffraction (XRD)

XRD-6000, Shimadzu x-ray diffractometer equipped with auto-search/match software as standard to aid qualitative analysis was performed to analyze the presence of crystalline phases. The specimens were prepared in powder form.

2.6. Fourier Transform Infrared (FTIR) Spectroscopy

Perkin Elmer FTIR Spectrum RX1 Spectrometer was used to evaluate the functional group of the sample. Small amount of potassium bromide (KBr) and geopolymer powder were put into a mould; the ratio between the KBr and geopolymer powder was set to be 1:0.01. By using cold press machine, mould which contains powder and KBr pellets was pressed at 295 MPa for 2 minutes to produce specimen for examination.

3. Result and Discussion

3.1. Compressive Strength

Compressive strength measurements were used as main tool to assess the success of geopolymerization reaction. This is because of low cost and simplicity of the compressive test.

Figure 1 shows the strength development of kaolin geopolymers at different NaOH concentrations (6-14M) up to 180 days. Geopolymers synthesized showed obvious strength differences when different concentrations of NaOH were applied. Therefore, the concentration of NaOH solutions has significant effects on the mechanical strength of kaolin-based geopolymers. Alkali concentration is the most significant factor affecting geopolymerization where alkali are required to activate the Si and Al in kaolin, allowing the structure to totally or partially dissolve and transform into a very compacted composite [9].

Initially, the strength gain before day 7 was a bit fluctuated between 0.53 MPa and 1.18 MPa. Low compressive strength values suggested that the early strength gain was quite slow for kaolin-based geopolymers. It seemed that the strength at 28 days and above presented steady increment until maximum strength nearly 3.98 MPa was reached at 180 days. In other words, compressive strength was maximized at NaOH concentration of 8M at 180 days, implying that there is an optimum alkalinity for activating kaolin. The optimum concentration of NaOH for kaolin-based geopolymer was 8M as it showed more stable strength development and the highest strength among others. With an optimum concentration, there are sufficient OH^- ions to dissolve the aluminosilicates sources releasing the Si^{4+} and Al^{3+} ions. This is followed by the exchange and the oligomerization process between silicate and aluminate species from the aluminosilicates sources and the silicate species from the activating solution. Finally, the polycondensation reaction and hardening of gel structure to form rigid structure [10, 11].

Curing plays important role in both the acceleration of chemical reaction and the determination of extent of reaction [12]. Heat applied during the curing stage increased the reaction rate and hence reduced the reaction time as according to the kinetic theory. This accelerated the dissolution of the aluminosilcate sources into the system for further reaction to form more geopolymer bonding and ensured the completion of the geopolymerization reaction. All the kaolin geopolymers samples set and formed hard structure within 24h after curing in oven. Curing time generally showed positive effect on the performance of geopolymers [13] and prolonged curing time of geopolymer mixture improved the geopolymerization process, yielding higher com-

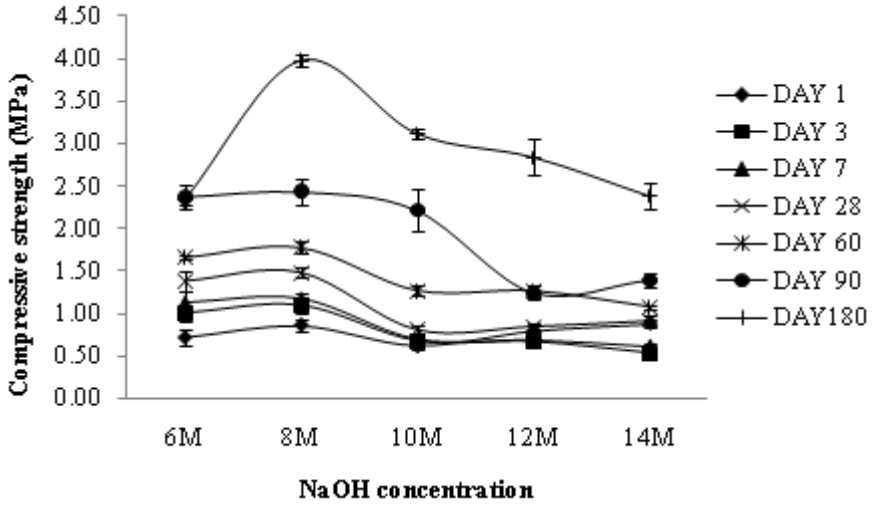

Fig. 1 - Compressive strength of kaolin-based geopolymers using NaOH of different concentration (6 - 14M) over time (1, 3, 7, 28, 60, 90 and 180 days) / *Rezistența la compresiune a geopolimerilor pe bază de caolin folosind NaOH de concentrații diferite (6 - 14m), în timp (1, 3, 7, 28, 60, 90 şi 180 de zile).*

pressive strength [14]. This was expected that strength will continue to increase if the sample was to be tested after 180 days.

However, kaolin geopolymer was not following the trend as reported in previous research on other alkali-activated binders. In previous research, strength increased with an increase in Na concentration in the activating solutions [15] and activation of raw materials are quicker and stronger in high NaOH concentration [16]. Also, solubility of alumino-silicate increased when NaOH concentration increased [14, 17] or in more simple explanation, higher concentration of NaOH yielded higher compressive strength [9].

In contrast, kaolin-based geopolymers achieved highest strength in 8M of NaOH solution and the strength dropped as the NaOH concentration increased to 14M. This was probably due to excess of Na^+ ions in high NaOH concentration [13]. On the other hand, 6M of NaOH solution showed the high strength gain just slightly below 8M most probably because 6M has excess water due to lower concentration that eases the ions transportation during the geopolymerization process. In nature, kaolin appeared sticky in present of alkali solution, thus increasing in the NaOH concentration may decrease the workability of the kaolin geopolymers. At high concentration, the transportation of ions was limited.

The nature and behavior of the starting materials and the actual concentrations in solution affects the formation and setting of the geopolymerization product [2, 3]. It was expected that the strength of kaolin-based geopolymer would be lower which agreed with the results of Xu and Van Deventer [3, 18]. According to the authors, the rate of Al dissolution from kaolin is insufficient to produce a gel of the desired composition and, due to the low reactivity of kaolinite, additional time for interactions among the raw materials used is required for gel formation.

In addition, if a large amount of kaolinite is present, not all kaolinite taking part in the synthesis reaction as summarized by Van Jaarsveld et al. [19]. Therefore, due to the nature of low reactivity of kaolinite, a weak structure is formed, thus contributes to slow compressive strength development of geopolymers [3, 18]. Excessive NaOH results in undesirable morphology and non-uniformity of hydration products in the pastes, thereby reducing the binder strength [9]. This was clearly shown in the SEM micrographs. At high NaOH concentration (10M and 14M of NaOH solutions), the microstructures as shown in Fig. 3, Fig. 4 and Fig. 5 revealed a less compacted and less homogeneous structure than those shown by lower NaOH concentration (8M of NaOH solution).

3.2. Morphology Study

Scanning Electron Microscope (SEM) depicts morphological features of kaolin geopolymers synthesized with activation medium of different concentrations. It could be observed that changes in morphological features of kaolin-based geopolymer paste have occurred and difference in microstructures could be distinguished. Figure 2 revealed that morphology of pure kaolin is plate-like structure forming stacking of cards [20]

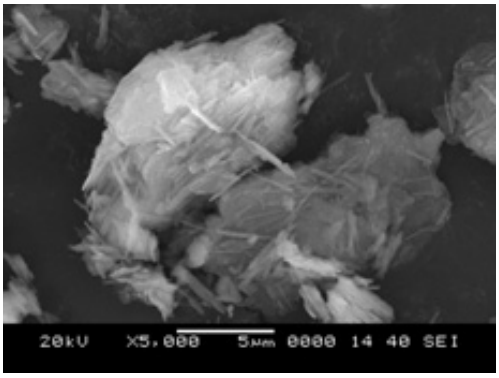

Fig. 2 - SEM micrograph of kaolin / Imaginea *SEM a caolinului.*

From Figure 3, micrographs showed that kaolin has been slightly activated by the NaOH concentrations of alkali activator.

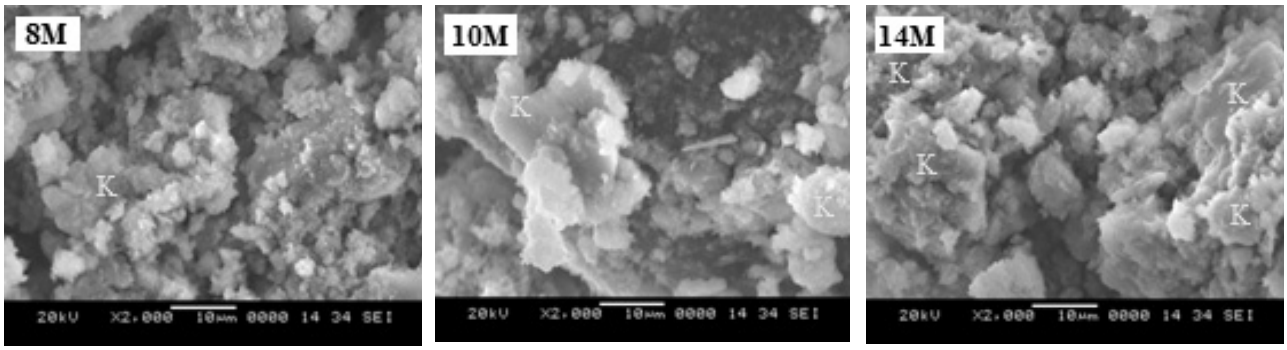

Fig. 3 - SEM micrograph of geopolymer product at day-1 (K = unreacted kaolin) / *Micrografii SEM a geopolimerului la o zi (K = caolin nereacționat).*

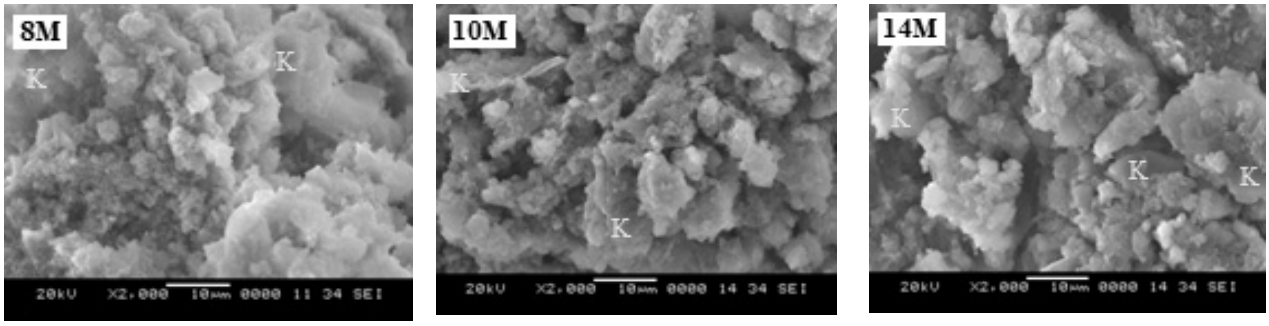

Fig. 4 - SEM micrograph of geopolymer product at day-7 (K = unreacted kaolin) / *Micrografii SEM a geopolimerului la 7 zile (K = caolin nereacționat).*

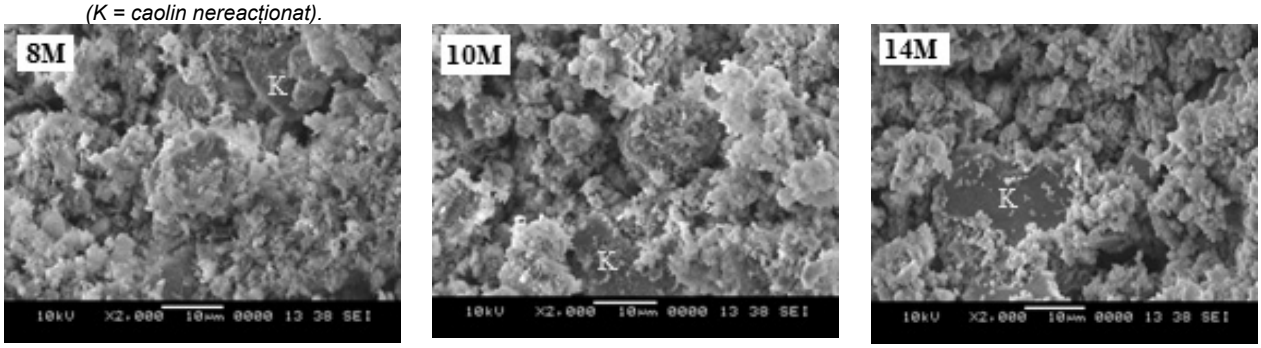

Fig. 5 - SEM micrograph of geopolymer product at day-60 (K = unreacted kaolin) / *Micrografii SEM a geopolimerului la 60 de zile (K = caolin nereacționat).*

Surface showed slight activation of particles with few partially reacted particles and large amount of un-reacted particles. In geopolymers with 8M NaOH solution, the structure appeared more homogeneous. Among the geopolymers samples, 10M has larger amount of un-reacted portion which led to the lowest strength.

Figure 4 to Figure 5 revealed that microstructure changed to denser structure compared to Figure 2. This indicated that structure experiences "growth" where geopolymerization reaction has taken place after putting in room temperature at day-7 and day-60 as well. The reaction was better in longer curing period. Sample with 8M of NaOH solution appeared to have more geopolymeric matrix than other samples, which contributed to highest compressive strength at day-60. The 8M NaOH solution always showed fastest reaction compared to others, leading to higher strength compared to all others concentration and more homogenous structure. Such observation agreed well with the compressive strength measured, showing that the alkaline activation is more effective. The higher the degree of reaction, the greater the compressive strength [13]. Conversely, when 14M of NaOH solution was employed, geopolymeric matrix is slightly lesser. This might probably because of the reason stated above. However, large part of un-reacted kaolin could still be observed in all samples, which could also be observed through XRD (Figure 6) and FTIR (Figure 7) analysis. Strength will definitely increase if the un-reacted materials reacted to form a more dense structure.

3.3 X-Ray Diffraction

From XRD pattern shown in Figure 6, kaolin composed of kaolinite (K) and quartz (Q) as major minerals. Alunite (A) and dickite (D) could be found in trace amount. Kaolinite showed intense peaks at 2θ values of 12.5° and 25.2°, less intense peaks at 2θ values of 45.4°, 49.5°, 50.9° and 59.9°. In addition, kaolinite humps was also found at 2θ values between 19.8°-21.9°, 35.0°-36.0° and 37.8°-39.2°. The quartz peaks could be found at 2θ values of 20.8°, 26.5°, 55.2° and 62.3°.

After geopolymerization process, a number of characteristic kaolinite peaks was remained in spectra of geopolymer samples. This suggested large amount of un-reacted raw materials left in the system as shown by the SEM micrographs. Quartz phases were found to be largely un-reactive, where the reflection peaks remained after the alkali activation of kaolin. This indicated that quartz did not take part in the geopolymerization process, but their intensities were slightly lowered due to dilution effect [21, 22]. Zeolite A (PDF # 38-0241) were found in all the geopolymer samples. As in previous research [6], higher alkali concentration showed higher amorphous content. However, in this study, it was observed that geopolymer product produced from 8M of NaOH solution has higher amorphous content than the others. From the XRD analysis, 8M geopolymer samples showed overall lower peaks intensities than the other geopolymer samples with higher NaOH concentrations. This indicated that the lowering of the peak crystallinity in the samples. Furthermore, the percentage of

crystallinity analysis showed that 8M samples have lower crystalline phase (40%), followed by 10M (47%) and 14M samples (55%). In all the geopolymer products, XRD pattern of geopolymer samples showed that large part of unreacted materials remains in the system. These un-reacted parts contributed to the low strength of geopolymer products.

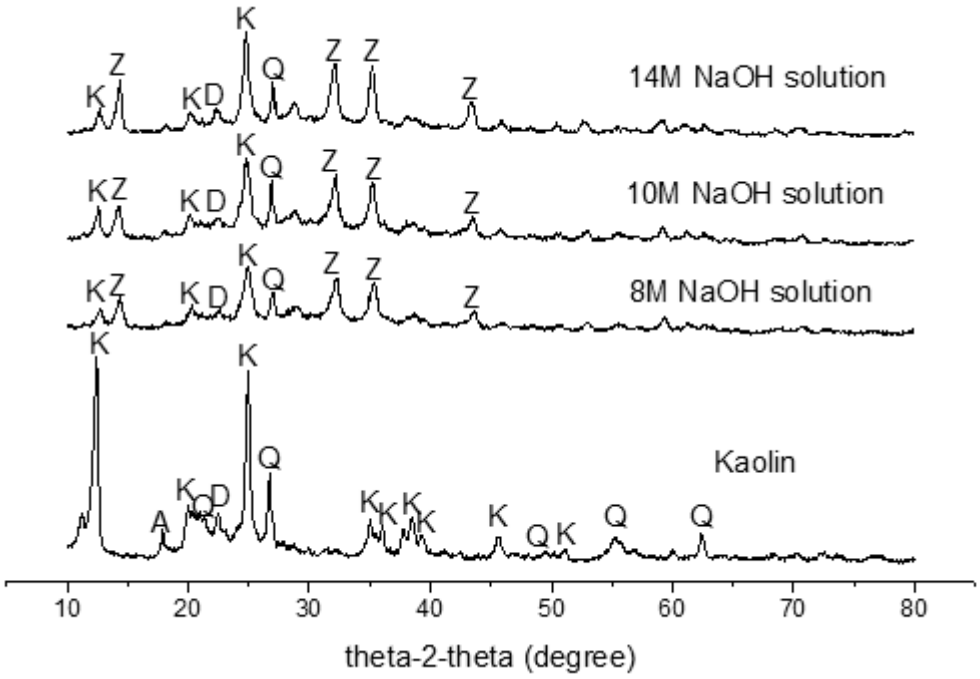

Fig. 6 - XRD pattern of kaolin and geopolymer products from different concentration of NaOH solution at day-7 (K- Kaolinite; Q- Quartz; A- Alunite; D- Dickite; Z- Zeolite) / *Difractogramele caolinului şi a geopolimerilor obținuți cu diferite concentrații de NaOH la 7 zile (K-caolinit, Q-cuarț; A-Alunite; D-Dickite; Z-zeolit).*

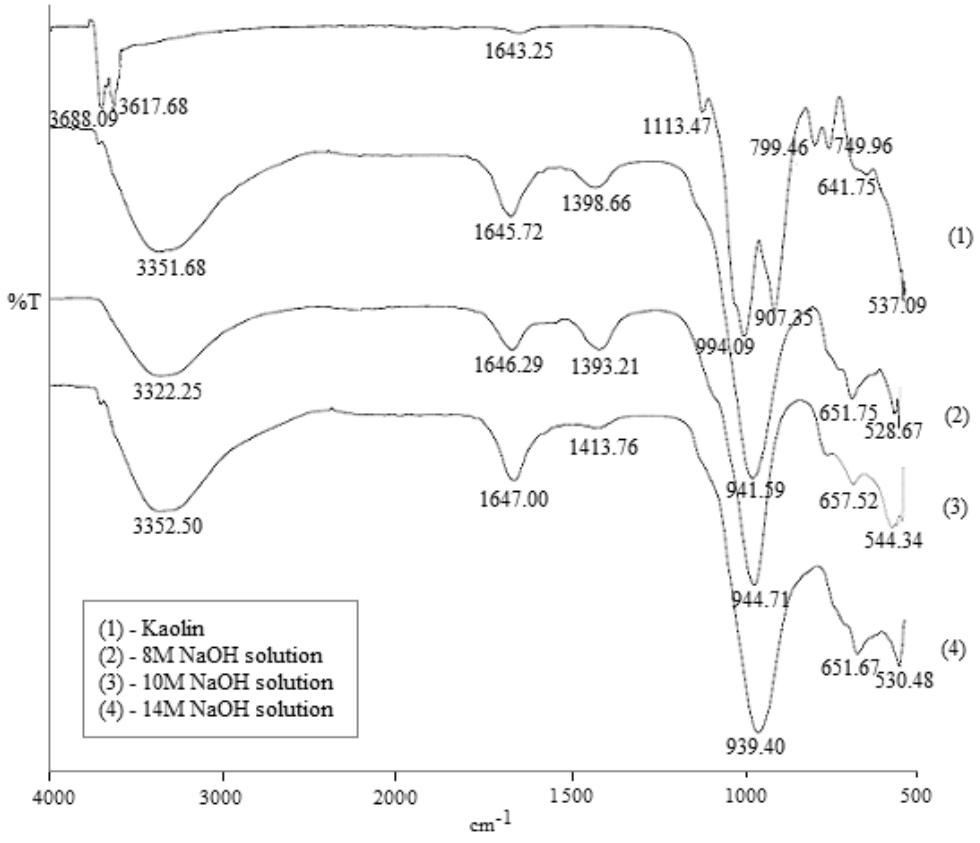

Fig. 7 - FTIR spectra of kaolin and geopolymer product at day-7 / *Spectrele FTIR a caolinului şi a geopolymerilor la 7 zile.*

3.4 FTIR spectroscopy

Figure 7 showed the IR spectra of kaolin and geopolymer products synthesized using sodium silicate solution and different NaOH concentration (8–14M). Transformation taken place during the synthesis was indicated by the different absorption frequencies of kaolin and the synthesized geopolymers [19].

In IR spectrum of kaolin, bands at 3688cm-$^{-1}$, 3617cm^{-1} and 1643cm^{-1} were corresponded to OH vibration. According to Davidovits [1], network of silico-aluminate based geopolymers consists of SiO_4 and AlO_4 tetrahedral linked alternately by sharing all the oxygens. In IR spectrum of kaolin, the peak around 1113 cm^{-1} was attributed to Si-O vibration in SiO_4 group, which vanished after geopolymerization reaction. Also, a weak band of Si-O symmetrically stretching vibration was observed at 640 cm^{-1}. Absorption at 995 cm^{-1} and 790 cm^{-1} are assigned as Al (IV)-OH (6 fold coordinated) and Al (IV)-O (6 fold coordinated), respectively. A shift of the asymmetric bending of the bonds O-Si-O and O-Al-O to lower frequencies could be observed, which was in accordance to previous research [10].

The main band analyzed in IR spectrum of geopolymer was in the region of 900-1300 cm^{-1}, corresponding to the Si-O-T linkages. Other major bands were broad band at 3000 – 3500 cm^{-1} and 1650 – 1655 cm^{-1} which corresponded to the stretching and deformation vibration of OH and H-O-H groups from water molecules. These peaks indicated the presence of weak H_2O bond absorbed in the surface or caught in the cavities of structure [22]. Wavenumber at around 1400 cm^{-1} were attributed to the Si-O-Si stretching. Wavenumber at around 660 cm^{-1} was the Si-O-Si and Si-O-Al symmetric stretching, whereas band at 537 cm^{-1} was Si-O-Al bonds [19]. These wavenumber shifted from kaolin suggesting that there are changes in chemical bonding taken place in the system. Only little difference between IR spectrum of kaolin and geopolymer synthesized within 500-1000 cm^{-1}, suggesting that most part of un-reacted kaolin still retained in the geopolymer synthesized [6].

4. Conclusion

As a conclusion, NaOH concentration has significant effect on the compressive strength of kaolin-based geopolymer. Kaolin-based geopolymer was not following the usual trend as other alkali-activated binders and 8M was recorded as the best concentration. SEM micrographs revealed that kaolin has been activated by different NaOH concentrations of alkali activator and transformed to denser structure. FT-IR spectra clearly indicated that transformation has taken place during the synthesis process due to the different absorption frequencies of starting material and the geopolymer products. Kaolin-based geopolymer produced from 8M of NaOH solution comprised of higher geopolymer bonding, thus exhibits higher strength. Besides this, XRD pattern showed that the geopolymer product has higher amorphous content than the others according to the percentage of crystallinity analysis.

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NOUTĂȚI / NEWS

Fluidizarea pulberilor nano şi sub-micronice folosind vibrația mecanică
Fluidization of nano and sub-micron powders using mechanical vibration

Comportamentul fluidizant al pulberilor nano şi sub-micronice aparținând grupei C în cadrul clasificării Geldat's a fost studiat într-un strat fluidizat prin vibrație mecanică (strat vibro-fluidizat) la temperatura camerei. S-a folosit aer pretratat ca gaz fluidizant, iar particulele solide au fost SiO_2, Al_2O_3, TiO_2, ZrSi, $BaSO_4$.

Amplitudinea vibrațiilor mecanice a fost de 5, 20, 30, 40 Hz cu scopul de a cerceta efectele frecvenței şi amplitudinii vibrațiilor mecanice asupra vitezei de fluidizare minime, asupra scăderii presiunii stratului, a expansiunii stratului şi a dimensiunii aglomeratelor şi a distribuției dimensionale. O nouă tehnică a fost folosită pentru a determina viteza aparentă minimă de fluidizare prin scăderea semnalelor presiunii. S-a folosit ecuația Richardson-Zaki, deoarece nanoparticulele prezintă comportament ca şi fluid atunci când sunt fluidizate. Dimensiunea medie a aglomeratelor formate în partea superioară a stratului a fost mai mică în comparație cu a celor de la partea inferioară. Distribuția dimensiunii aglomeratelor de la partea superioară a fost mai uniformă comparativ cu cea de lângă distribuitor. Aglomeratele mai mari de la partea inferioară formează o fracție mică a particulelor stratului. Dimensiunea medie a aglomeratelor submicronice scade cu creşterea frecvenței de vibrație, astfel că, nanoparticulele au fost mai puțin sensibile la schimbări ale frecvenței de vibrație. Vibrația mecanică a sporit calitatea fluidizării reducând fenomelele de formare de canale şi pori cauzate de forțele de coeziune dintre particule.

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The fluidization behavior of nano and sub-micron powders belonging to group C of Geldart's classification was studied in a mechanically vibrated fluidized bed (vibro-fluidized bed) at room temperature. Pretreated air was used as the fluidizing gas whereas SiO_2, Al_2O_3, TiO_2, ZrSi, $BaSO_4$ were solid particles.

Mechanical vibration amplitudes were 0.1, 0.25, 0.35, 0.45mm, while the frequencies were 5, 20, 30, 40 Hz to investigate the effects of frequency and amplitude of mechanical vibration on minimum fluidization velocity, bed pressure drop, bed expansion, and the agglomerate size and size distribution. A novel technique was employed to determine the apparent minimum fluidization velocity from pressure drop signals. Richardson–Zaki equation was employed as nano-particles showed fluid like behavior when fluidized. The average size of agglomerates formed on top of the bed was smaller than those at the bottom. Size distribution of agglomerates on top was also more uniform compared to those near the distributor. Larger agglomerates at the bottom of the bed formed a small fraction of the bed particles. Average size of submicron agglomerates decreased with increasing the frequency of vibration, however nano particles were less sensitive to change in vibration frequency. Mechanical vibration enhanced the quality of fluidization by reducing channeling and rat-holing phenomena caused by interparticle cohesive forces.

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