

Investigating the Geopolymerization Process of Aluminosilicates and Its Impact on the Compressive Strength of the Produced Geopolymers

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Abstract—This paper investigates multiple factors that impact the formation of geopolymers and their compressive strength to be utilized in construction as an environmentally-friendly material. Bentonite and Kaolinite were thermally calcinated at 750 °C to obtain Metabentonite and Metakaolinite with higher reactivity. Both source materials were activated using a solution of sodium hydroxide (NaOH). Thereafter, samples were cured at different temperatures. The samples were analyzed chemically using a host of spectroscopic techniques. The bulk density and compressive strength of the produced geopolymer pastes were studied. Findings indicate that the ratio of NaOH solution to source material affects the compressive strength, being optimal at 0.54. Moreover, controlled heat curing was proven effective to improve compressive strength. The existence of characteristic Fourier Transform Infrared Spectroscopy (FTIR) peaks at approximately 1020 cm^{-1} and 460 cm^{-1} which correspond to the asymmetric stretching vibration of Si-O-T and bending vibration of Si-O-Si, hence, confirming the formation of the target geopolymer.

Keywords—Alcination of metakaolinite, compressive strength, FTIR analysis, geopolymer, green cement.

I. INTRODUCTION

GEOPOLYMERS are considered the next generation of Cementitious materials. They demonstrate excellent physical and mechanical properties in addition to minimal detrimental impact on the environment. Fly ash geopolymer concrete yields comparable stress-strain relationship and higher tensile strength than Ordinary Portland Cement (OPC) concrete of the same compressive strength [1]. They are synthesized through a polycondensation reaction commonly known as geopolymerization. It requires an aluminosilicate source material, highly alkaline solution and suitable curing conditions, as described by Duxon et al. in their generic model for the mechanism of Geopolymerization including dissolution, speciation, reorientation and polycondensation [2].

The commonly used precursors for the geopolymer synthesis are fly ash, blast furnace slag and meta-kaolinite, while further research on other aluminosilicates is still ongoing. Bentonite was successfully activated using alkaline solution of sodium hydroxide and sodium aluminate resulting in geopolymer with considerable strength of 30 MPa [3]. The calcination process and the characteristics of the source materials, such as its chemical composition, particle size, the nature of oxides (whether crystalline or amorphous) and the silica to alumina

ratio, all determine the suitability of the source to undergo geopolymerization.

Calcination is a thermal treatment where hydroxyl groups are removed from alumina, converting it from penta or hexa coordinated to tetraordinated which is the most reactive form of alumina. The calcination alters the atomic structure, converting kaolinite to more disordered structure through the separation of the hydroxyl group when exposed to temperature higher than 550 °C [4]. The temperature, duration and rate of calcination have direct impacts on the polymerization process. When the calcination temperature of Kaolinite was varied at 650 °C, 700 °C, 750 °C, 800 °C and 850 °C in electric furnace for 6 hours, the optimum temperature was 750 °C resulting in the highest compressive strength [5]. The rate of calcination has been proven to change the chemical properties of kaolinite, thus, affecting the output geopolymer strength, such that the slower the calcination rate, the higher the strength [6].

The mixing ratios of the synthesis materials and curing conditions play fundamental roles in the development of the microstructure and properties of the geopolymers. Oven curing for 4 hours at 40 °C was found to expedite the strength gain of metakaolinite geopolymer mortar whereas curing at 60 °C and 80 °C for the same duration caused deterioration in 28-day strength [7]. Aredes et al. reported that oven curing at 65 °C for one hour resulted in compressive strength of 12 MPa and compact structure with the least pores [8].

The presence of amorphous tetrahedral silicate and aluminate (bonded together) confirms geopolymer formation. This is detected through number of characterization techniques especially X-ray Diffraction, Al, Si and Na Magic Angle Spinning Nuclear Magnetic Resonance (MAS NMR) [9], FTIR [10], Isothermal Conduction Calorimetry [11] and Raman spectroscopy [12].

Literature has reported wide range of properties particularly the compressive strength [13], [14]. There are multiple reasons for the low strength, categorized mainly as unviable source material, insufficient proportions of the mix, and inadequate curing environment.

II. TESTING PROGRAM AND PROCEDURES

This study examines the effect of chemical composition, ratio between the alkaline solution and source and curing

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conditions on the microstructure formation and the compressive strength development of geopolymer paste synthesized from different precursors.

A. Materials

Two different source materials, Bentonite (brand name SRL) and Kaolinite (brand name Top chem) supplied by Alfa Chemicals Egypt, were used in this study. Kaolinite is anhydrous natural occurring aluminosilicate material that undergo calcination at 750 °C to be converted to Metakaolinite. Bentonite is fine grained montmorillonite clay that undergoes the same calcination process to become Metabentonite.

The kaolinite was divided into two batches (MK1 and MK2) to be calcinated at different rates. Both batches were heated for 7 hours at 750 °C. The main difference was the rate of heating to reach the desired temperature of 750 °C. It was varied being 2.27 °C/min and 5 °C/min for MK1 and MK2, respectively.

The only alkaline solution used is sodium hydroxide (NaOH pellets, purity 99%) which was also supplied by Alfa Chemicals Egypt.

B. Characterization of Source Material Used

The chemical composition and the loss-on-ignition of the source materials were determined using the X-ray Fluorescence (XRF) to measure the exact percentage of oxides especially SiO₂ and Al₂O₃, as shown in Table I.

TABLE I
CHEMICAL COMPOSITION OF KAOLINITE (BATCH 1) AND BENTONITE BEFORE AND AFTER CALCINATION USING XRF

Component Weight (%)	Kaolinite	Meta-kaolinite	Bentonite	Meta-Bentonite
SiO ₂	45.18	51.7	45.96	49.4
Al ₂ O ₃	33.1	36.10	18.5	19.3
Fe ₂ O ₃	1.24	1.82	15.7	17.1
CaO	2.02	2.5	0.76	1.26
MgO	1.18	1.34	1.54	1.83
Na ₂ O	0.27	0.28	2.15	2.17
K ₂ O	0.07	0.08	1.23	1.2
TiO ₂	2.99	4.18	3.02	3.26
MnO	-	-	0.26	0.36
LOI	13.3	1	9.04	2.87

C. Mixing Proportions

Several synthesis ratios were attempted, but the procedures were kept constant. The alkaline solution was prepared by mixing the calculated amount of NaOH pellets in distilled water using a magnetic stirrer to obtain a concentration of 10 molarity (M). The geopolymer paste was prepared by mixing the source material with the alkaline activator for 2-3 minutes in a mechanical mixer until a homogenous mix is obtained. The proportions of each mixture are shown in Table II.

Mixtures 7 and 8 were the replication of Mixtures 3 and 4, respectively using MK2 to study the effect of rate of calcination of kaolinite on compressive strength.

The paste was placed and compacted in 50 x 50 x 50 mm steel molds on three layers and vibrated using a vibrating table to expel any air voids. The samples were removed from the mold and were left for curing either at room temperatures or at

controlled high temperature.

TABLE II
DETAILS OF THE MIXING RATIOS OF THE MIXTURES

Mixture No	% Source Materials		NaOH / Source Material	Curing Conditions
	Meta-kaolinite	Meta-Bentonite		
1	100 % of MK1	0%	0.47	Room temperature
2	100 % of MK1	0%	0.51	Room temperature
3	100 % of MK1	0%	0.54	Room temperature
4	100 % of MK1	0%	0.57	Room temperature
5	100 % of MK1	0%	0.6	Room temperature
6	100 % of MK1	0%	0.51	Oven curing at 60 °C for 24 h
7	100 % of MK2	0%	0.54	Room temperature
8	100 % of MK2	0%	0.57	Room temperature
9	50 % of MK1	50%	0.54	Room temperature
10	0%	100 %	0.54	Room temperature

D. Characterization Methods

The source materials used and geopolymer synthesized were characterized by Nicolet 380 FTIR with spectrum range (4000 – 400) using the KBr pellets to examine the characteristic molecular bonding.

The bulk density of the geopolymer samples was calculated from the mass and volume. The 7-day compressive strength of each mixture was measured using a Universal Testing Machine. Three samples of each mixture were prepared, tested, and the average value was calculated.

III. RESULTS AND DISCUSSION

A. Bulk Density

The bulk density of metakaolinite geopolymer paste ranged between 1600-1700 kg/m³ which varied depending on the mixing ratios used. This value is relatively low compared to the OPC paste which averages around 1800 kg/m³. In previous research on kaolin geopolymer paste, Heah et al. reported the bulk density to be even as low as 1250-1500 kg/m³ [15]. A wider range of bulk densities, 1.64-1.95 gm/cm³, was obtained when different molarities of NaOH were used to activate Metakaolinite. The density was reported to increase with increasing molarity [16].

B. FTIR

FTIR Spectra of the Metakaolinite (MK1) and Metabentonite are shown in Figs. 1 and 2, respectively. Both calcinated aluminosilicate sources shared peaks of 3440 cm⁻¹, and 1638 cm⁻¹ which were attributed to stretching vibration of (OH-) and bending vibrations of H-O-H bond, respectively. The presence of these peaks implied that calcination process was not completed as explained by [17]. In addition, peaks at 1045 cm⁻¹, 609 cm⁻¹, 469 cm⁻¹ were found in Metabentonite spectra, yet they were slightly shifted to 1036 cm⁻¹, 539 cm⁻¹ and 467 cm⁻¹ in Metakaolinite. The first peak could be assigned to asymmetric stretching vibration of Si-O-T band (T= Si or Al). The peak in the range 467-450 cm⁻¹ could be assigned to tetrahedral silicon bending vibrations.

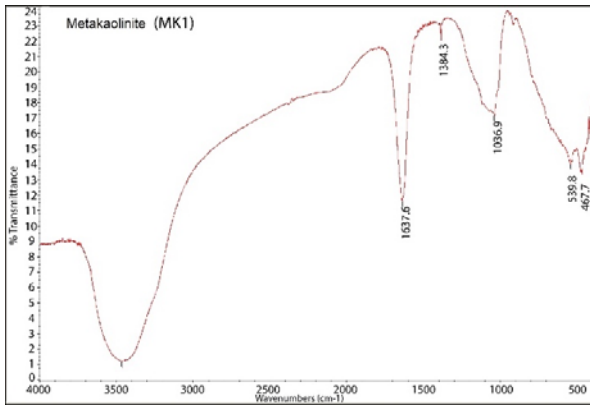


Fig. 1 FTIR Spectra of Metakaolinite (MK1)

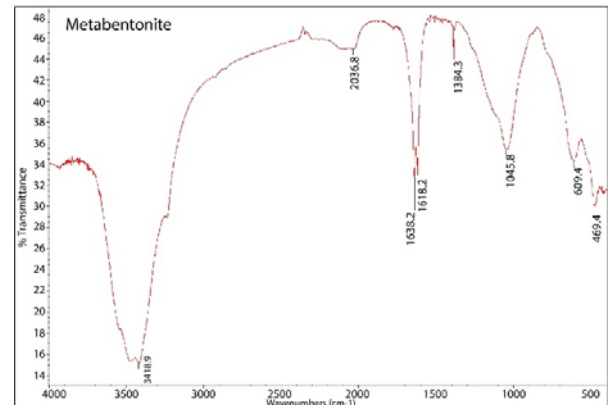


Fig. 2 FTIR spectra of Metabentonite

The produced geopolymer mixtures were also studied to examine their characteristic bands and to monitor the changes that occurred due to polymerization process. The obtained IR spectra of metakaolinite geopolymer are shown in Fig. 3. It was observed that the peak at 1036 cm⁻¹ in metakaolinite was shifted to 1020 cm⁻¹, 1021 cm⁻¹, 1013 cm⁻¹ in Mixtures 3, 4 and 5, respectively indicating a formation of new products. It was expected to see this shifting due to development of Si-O-Al bond rather than the Si-O-Si bonds [18]. Also, the appearance of strong peaks around 1638-1639 cm⁻¹ related to the bending vibration of (H-O-H) bond can be an evidence that geopolymerization still in progress since water is a product of silicate and aluminate polycondensation reaction.

Mixtures 1 and 5 had a relatively noticeable peak at 1384 cm⁻¹ which was attributed to sodium carbonate (Na₂CO₃) which means that carbonation took place where the sodium in the mix reacted with the carbon dioxide in the atmosphere. This was mainly due to excess sodium ions in the system or inadequate curing conditions that extended the geopolymerization process [19].

The FTIR spectra of Mixtures 9 and 10 (containing metabentonite) are shown in Figs. 4 and 5, respectively. In Mixture 9, the relatively strong peak at 1004 cm⁻¹ is attributed to vibrations of Si-O-T bonds [6] which confirms the formation of geopolymer network. Contrarily, a very weak band appeared at 1100 cm⁻¹ in Mixture 10.

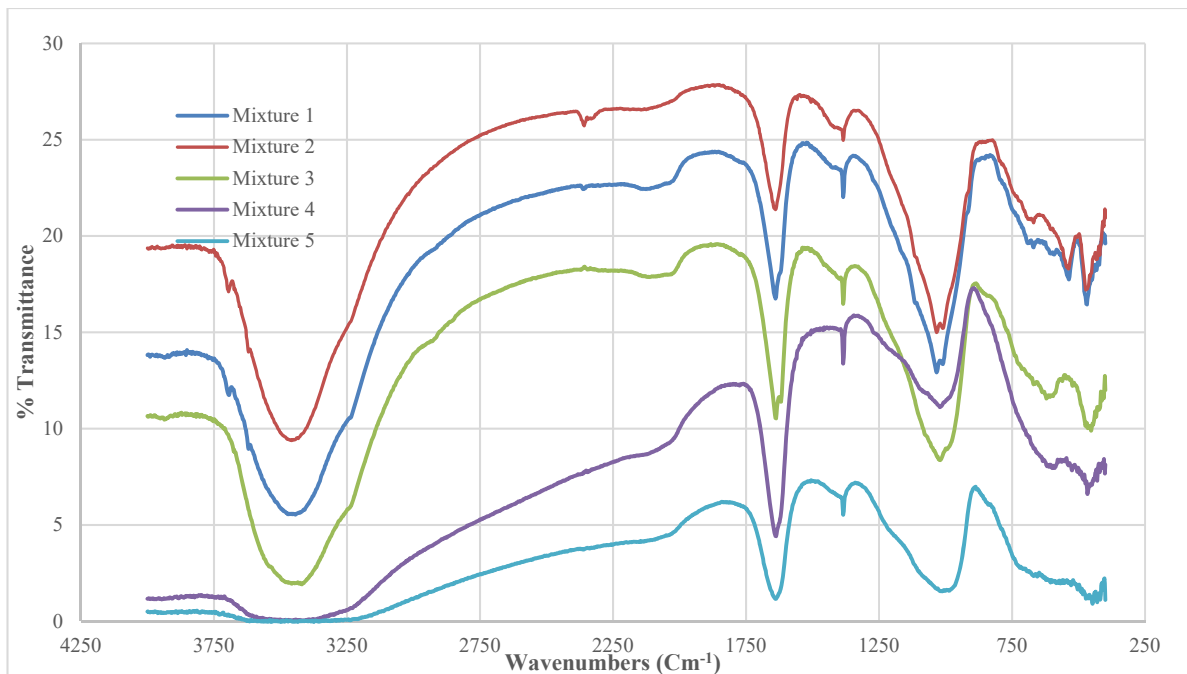


Fig. 3 Geopolymer Mixture 1 to Mixture 5 synthesized from Metakaolinite (MK1) and NaOH

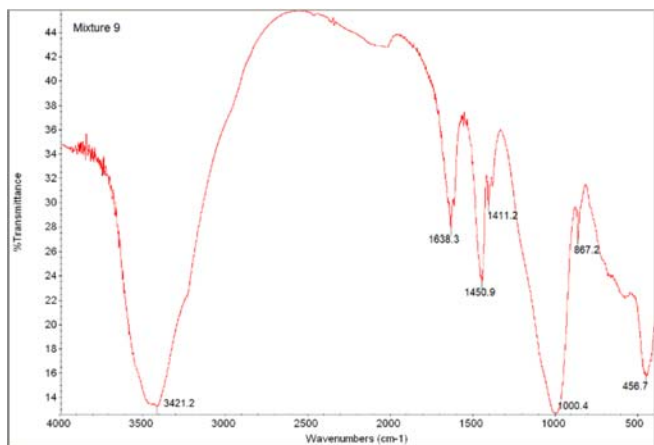


Fig. 4 FTIR Spectra of Mixture 9 synthesized from Metakaolinite (MK1) and Metabentonite.

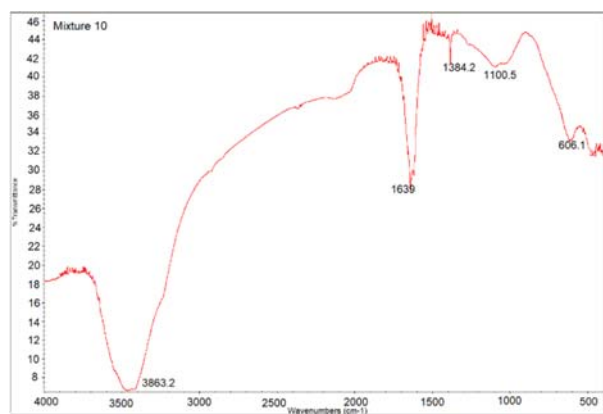


Fig. 5 FTIR Spectra of Mixture 10 synthesized from Metabentonite.

C. The Effect of the Rate of Kaolinite Calcination on the Compressive Strength

Two different rates of calcination were applied on kaolinite to study its effect on the reactivity of produced metakaolinite and how it could affect the strength of the geopolymer. The effect was reflected in the different compressive strength of geopolymer synthesized using the same mixture proportions as highlighted in Fig. 6.

There is a direct relationship between the rate of calcination and the compressive strength of geopolymer pastes. At NaOH/Metakaolinite ratio of 0.54 in Mixtures 3 and 7, decreasing the rate of calcination of kaolinite from 5 °C/min to 2.27 °C/min almost doubled the compressive strength of geopolymer paste. This can be explained that at higher calcination rate (when 750 °C was reached in 2.5 hours), the time was not sufficient for the removal of OH groups. Accordingly, the silica and particularly the alumina were not reactive enough to be easily activated by the relatively small amount of sodium hydroxide.

At higher NaOH/Metakaolinite ratio of 0.57 in Mixtures 4 and 8, the improvement in the compressive strength is about 30%. This is because the silicate and aluminate were less reactive and required higher percentage of alkaline solution to be released from the source material. Therefore, the excess presence of OH group favored the geopolymerization process at low reactivity source material.

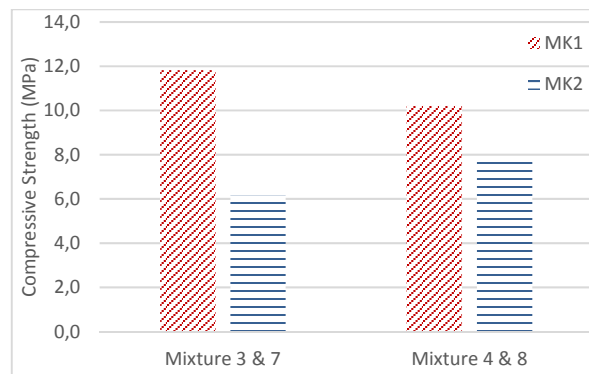


Fig. 6 Relationship between the rate of calcination of Kaolinite and the compressive strength of geopolymer paste

To conclude, the low strength obtained at higher rate of calcination is an indication of an incomplete calcination process which results in less efficient geopolymerization process. This complies with the study on the effect of the rate of calcination where the 28-day compressive strength which dropped from 49.4 MPa to 20.8 MPa when the rate of calcination was increased from 1 °C/min to 20 °C/min [6]. Also, in another study on geopolymer produced from kaolin source that was not calcinated, the highest compressive strength obtained after 180 days of curing was only 6 MPa [15]. This highlights the importance of the proper calcination of kaolinite prior geopolymerization.

D. The Effect of the Molar Ratios of Source Material on the Compressive Strength

One of the crucial synthesis ratios is the silica to alumina ($\text{SiO}_2/\text{Al}_2\text{O}_3$). It was calculated from the XRF analysis of the dehydroxylated Kaolinite and Bentonite 1.43 and 2.56, respectively. It is obvious that both have high percentage of silica and alumina and the $\text{SiO}_2/\text{Al}_2\text{O}_3$ falls within the range (1.5-3) which most of the researchers have agreed upon. The optimum $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio was reported by [20] to be 2.5 when metakaolinite, silica fume, and sodium hydroxide were used to produce geopolymer paste strength of about 64 MPa. When the $\text{SiO}_2/\text{Al}_2\text{O}_3$ was further increased beyond this value, cracks were noticed, and the strength was noticeably decreased. Another study reported optimum strength of 33 MPa at the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of 2, clarifying that this ratio triggered the maximum dissolution of silicate and aluminate species from the source material forming strong crosslinked Si-O-Al bonds [17].

Geopolymer paste was synthesized using two aluminosilicate sources to study the effect of the variation of $\text{SiO}_2/\text{Al}_2\text{O}_3$ on the obtained strength. Mixtures 3, 9 and 10 were produced using the same NaOH/source ratio except that the source material was 100% MK1, 50% MK1 + 50% bentonite, 100% Metabentonite. This study excluded the utilization of soluble silicates which possibly explains the relatively low compressive strength. Previous research highlighted that the presence of soluble silicates especially at the early stage enhance the polymerization process [17].

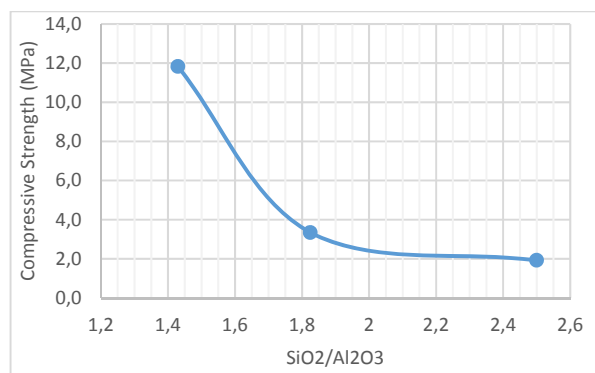


Fig. 7 Relationship between the SiO₂/Al₂O₃ ratio and the compressive strength of geopolymer paste

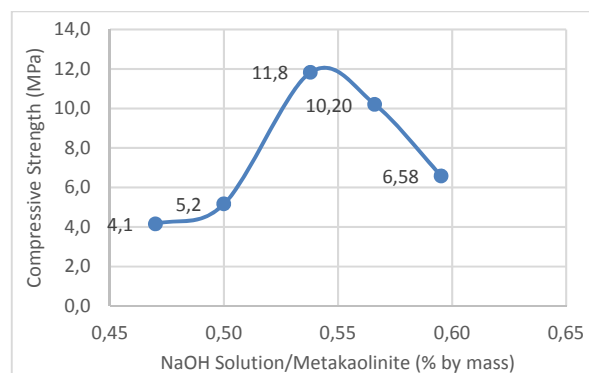


Fig. 8 The relationship between the ratio of NaOH/Metakaolinite and the 7-day compressive strength of geopolymer paste

The relation between the SiO₂/Al₂O₃ ratio and the 7-day compressive strength is presented in Fig. 7. Increasing the SiO₂/Al₂O₃ ratio from 1.43 to 1.82 and then to 2.56 causes the decline of compressive strength remarkably from 11.8 MPa to 3.3 MPa and 1.3 MPa, respectively. The decrease in strength is possibly attributed to the increased silica content which might have prevented the proper polycondensation of silicate and aluminate to the geopolymer matrix especially that the setting of metabentonite samples is noticeably prolonged compared to that of metakaolinite ones. The metabentonite samples were still wet when they were tested on the 7th day. Another explanation is that silica and alumina in metabentonite were not reactive to participate in geopolymerization process or the amount of alkaline solution was more than required. Still, the tested range of SiO₂/Al₂O₃ ratio was inconclusive to pinpoint the optimum value and more ratios need to be tried.

E. The Effect of the Alkaline Solution to Source Material Ratio on the Compressive Strength

The alkaline solution has a major role in the geopolymerization process. It provides the OH group required for the dissolution and then conversion of silica and alumina oxide into soluble species ready for polymerization. It is inferred that the dissolution of silicate takes place when the hydroxyl groups (OH⁻) break the siloxane bond (Si-O-Si) in a nucleophilic attack [21]. Several studies have confirmed that increasing the alkalinity to a certain extent enhances the geopolymerization resulting in higher mechanical properties [16], [22]. This indicates a strong correlation between the amount of hydroxyl group and the strength. However, there is an optimum amount beyond which any further increase possibly hinders the process and affects the strength.

Fig. 8 shows how the increase in the amount of NaOH, at a constant amount of MK1, favored the geopolymer formation till an optimum value of 0.54 was reached. This is because the more the amount of OH⁻ group, the better the dissolution of silicate and aluminate from the source. However, the high percentage of OH⁻ group can hinder the polycondensation process and negatively affect the geopolymer formation.

The results of the compressive strength match with FTIR spectra of geopolymer samples. Increasing the amount of alkaline solution resulted in clear broad band appearing at 3534 cm⁻¹ due to the presence of OH⁻ group in excess and in different orientations that are not utilized in geopolymerization process. The problem with the excess alkaline solution links to its inhibition of the polycondensation of silicate and aluminate.

F. The Effect of the Curing Conditions on the Compressive Strength

Curing presents the medium for the geopolymerization process. The absence of the required conditions whether humidity or temperature prevents the development of geopolymer structure and strength gain. Mixtures 2 and 6 were produced using the same materials and synthesis ratios except that the former samples were let to cure in room temperature at 21 °C and latter were put in oven for 24 hours at 60 °C after 24 hours from being poured. The 7-day compressive strength was observed to increase from 5.2 MPa to 8 MPa due to high temperature curing. This complies with the findings Aredes et al. on the influence of the curing temperature. The compressive strength of Metakaolinite geopolymer was improved from 7 MPa to 12 MPa when the curing temperature was raised from 55 °C to 65 °C [8].

IV. CONCLUSIONS

Geopolymer is an environmentally friendly building material that is synthesized from metakaolinite with reasonable compressive strength. Based on the study and experimental results herein, the following findings can be inferred as:

1. There are several factors that affect the geopolymerization initiation and completion. These factors are the SiO₂/Al₂O₃ ratio, type and dosage of alkaline solution, and the curing conditions. However, these factors can slightly vary depending on the aluminosilicate source material that will be used.
2. In case of kaolinite, the calcination process plays a major role in addition to the other factors. The slower the rate of calcination, the higher the strength obtained.
3. Metakaolinite-based geopolymer can be a potential building material with relatively low density as compared to OPC paste.

4. Increasing the NaOH/Metakaolinite ratio causes reduction in the 7-day compressive strength of pastes.
5. The use of metabentonite to increase the $\text{SiO}_2/\text{Al}_2\text{O}_3$ 1.43 to 1.82 negatively affects the 7-day compressive strength of pastes. Lower $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios should be considered in future research.
6. Heat curing improves the geopolymerization process, resulting in higher mechanical properties of geopolymer. However, future research should focus on the determination of the optimum curing duration and temperature especially its effect on mechanical properties on the long term.

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