



Effect of Kaolinite Mass Ratio on Compressive Strength of Kaolinite-Calcite Based Geopolymer

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Abstract: This study focuses on the preparation of geopolymers using various proportions of kaolinite and calcite mixture, and the effects of kaolinite mass ratio to calcite on compressive strength of kaolinite/calcite-based geopolymer. Several geopolymer samples were formed using pure kaolinite, pure calcite, and various mass ratio of kaolinite to calcite to examine properties of the geopolymers. The impact of NaOH concentration on the compressive strength of the geopolymers was also examined. The results of the laboratory analyses indicate that the compressive strength of geopolymers did not increase steadily with increasing concentration of NaOH. The optimum workability of geopolymers was obtained at 10 M NaOH and 30 % kaolinite content in the mixture. Several sensitivity experiments were carried out using the parameters at cure temperatures of 22, 45, 65 and 85°C. The best compressive strength of 12.68 MPa was obtained on the 28th day of curing process at 85°C. The structural evolution of the geopolymers prepared was investigated using FTIR spectroscopy. They were also characterized by X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

1.Introduction

Geopolymers based on alumina silicates such as natural raw materials (kaolinite, zeolite, calcite) and industrial wastes (fly ash, granulated blast-furnace slag, silica fume) are a favorable alternative for replacing Portland cement (PC). Specially, the use of waste materials in the construction sector has increased in recent years due to environmental and economic reasons [1,2]. For geopolymerization process, raw materials do not need to be burned at high temperatures as in the case of Portland clinker while alkali active binders are being prepared. For that reason, geopolymers are environmentally friendly materials that do not release CO₂ [3]. In addition, geopolymers are of great interested by researchers due to their superior mechanical strength, low shrinkage, acid resistance, fire resistance and low thermal conductivity [4]. The primary step of geopolymerization is the dissolution of the solid alumina silicate oxide in a KOH or NaOH solution to form free SiO₄ and AlO₄

tetrahedral units. Alkali activation of these raw materials results in three-dimensional alumina silicate structure which display better mechanical strength properties such as compression strength [5]. The main product formed in the geopolymeric system is either sodium alumina silicate gel N-A-S-H (Na₂O-Al₂O₃-SiO₂-H₂O) or calcium alumina silicate hydrate gel type C-A-S-H (CaO-Al₂O₃-SiO₂-H₂O) as depend on the content of the raw material [6,7]. Polymer formation rate during geopolymerization is affected by such parameters as curing temperature, curing day, alkali concentration, water content and initial solids content [4]. As useful construction materials such as kaolinite could be hardened and transformed into geopolymers therefore kaolinite is an alternative alumina silicate source for geopolymerization [8,9]. Herein, we aimed to prepare the most suitable geopolymers using various proportions of kaolinite and calcite mixture for building industry. The effects of curing time, curing temperature and NaOH concentration and kaolinite mass ratio on the

compression strength of prepared geopolymers were investigated.

2. Experimental

NaOH and kaolinite were mixed in a beaker for 5 min. Then calcite was added and mixed for 5 min. Finally, Na_2SiO_3 was added and mixed for 15 min and then the fresh cement was rapidly poured into a cubic steel mold of $40 \times 40 \times 40 \text{ mm}^3$. In order to investigate the effect of the activator on the properties of the geopolymer material, a series of NaOH solution with concentrations of 5, 10, 15 and 20 M and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio of 0.5, 1, 1.5 and 2 were used. Varying curing temperatures were applied on the prepared geopolymers such as 22, 45, 65 and 85°C .

3. Results and Discussion

NaOH concentration, $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio, curing temperature and curing day are the most important parameters in geopolymer production [4]. The effect of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio on the compressive strength is plotted in Fig 1. It was observed that compressive strength of the geopolymer increases as the $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio increases. The best compressive strength was reached (9.12 Mpa) when $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio was 2.

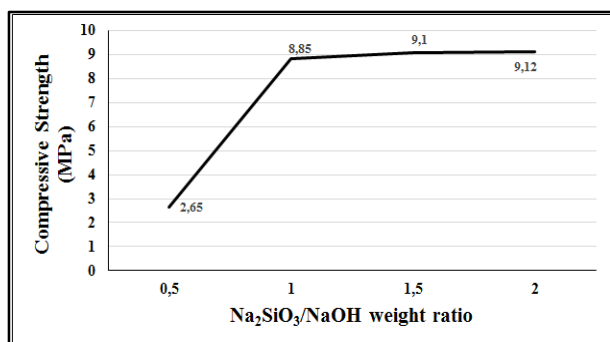


Figure 1. The effect of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio on the compressive strength.

Fig 2. shows the effect of percent of kaolinite in the mixture with various NaOH concentration and curing temperature on the compressive strength. It is seen from the figures that the highest compressive strength of the geopolymers was at 80% kaolinite in the mixture at 20 M NaOH concentration (Fig 2a). Although the compressive strength increased with the NaOH concentration, because of the deterioration and hard workability of

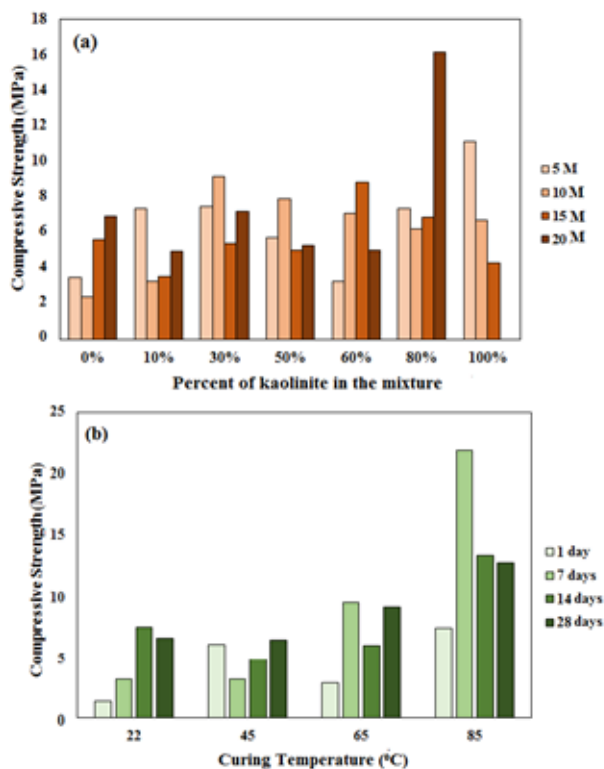


Figure 2. (a) The effect of percent by weight of kaolinite in the mixture (b) curing temperature on the compressive strength

the samples at high NaOH concentration, 30% kaolinite content in the mixture and 10 M NaOH concentration was chosen as the best experimental conditions for preparation of geopolymers (Fig 2a). Compressive strength of geopolymer samples increases along with increase curing temperature and days (Fig 2b). Curing temperature at 85°C was used for preparing for all geopolymer samples because the highest compressive strength reached on the 28th day of curing process at 85°C . The highest compressive strength was obtained as 12, 68 MPa at the 28th day of curing process at 85°C (30% kaolinite content and 10 M NaOH concentration, Fig 2b).

Fig. 3a illustrates the FTIR spectra of calcite, kaolinite and geopolymers at different curing temperature on the 28th day of curing process. The band at 3686 cm^{-1} corresponds to Si-O/Al-O vibrations which belongs to kaolinite [4]. In addition, the characteristic calcite peaks observed at 1393 , 871 and 711 cm^{-1} indicate CO_3^{2-} asymmetric stretching, CO_3^{2-} out-of-plane, and CO_3^{2-} in-plane bending, respectively [10]. The characteristic peaks of kaolinite and calcite were also observed from FTIR spectra of geopolymers as seen in Fig. 3a. These results clearly show the presence of unreacted kaolinite and calcite particles in the geopolymer.

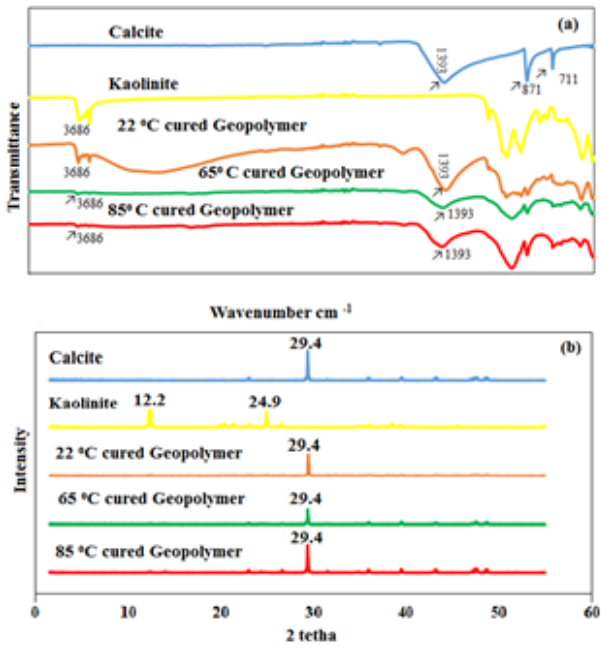


Figure 3. (a) FTIR spectrum and (b) XRD patterns of calcite, kaolinite and geopolymers with various curing temperature (30% kaolinite content in the mixture, 10 M NaOH and 28th day of curing process).

As seen in figure 3b, the characteristic diffraction peaks of kaolinite and calcite were located at 12.2, 24.9 and 29.4° (2θ). The characteristic diffraction peaks of kaolinite disappeared in all geopolymer samples after geopolymerization process [4]. The peaks of the prepared geopolymers were observed around 29° (2θ) [11].

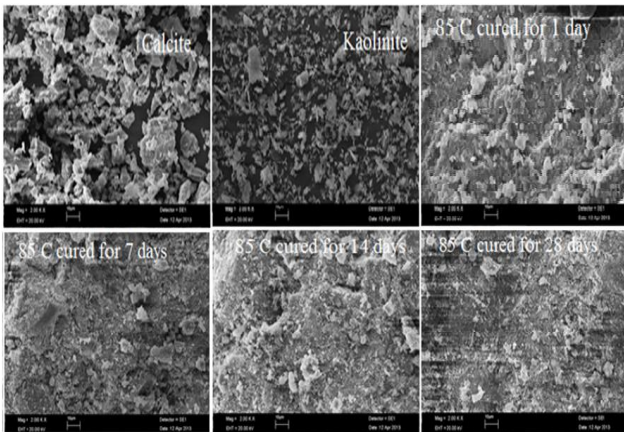


Figure 4. SEM micrographs of calcite, kaolinite and 85° C cured geopolymers

SEM micrographs of geopolymers exhibit significant change in the microstructure with increased curing time (Fig. 4). It is seen that the geopolymers are more dense and homogeneous as

curing day increases and have a more hardened structure at 28th day.

4. Conclusion

In this study, kaolin and calcite were used together to prepare geopolymer and the effect of kaolin on compressive strength was analyzed. Kaolinite-calcite based geopolymer materials have been successfully synthesized various mass ratio of kaolinite to calcite using aqueous solution of alkali silicate and NaOH. %. It was observed that when the amount of kaolin was 80% and the concentration of NaOH was 20 M, although the compression strength was the highest, there was deterioration in the structure of the sample. Therefore, the optimum compressive strength was reached at a Na₂SiO₃/NaOH mass ratio of 2, when the NaOH concentration was 10 M and at curing time at 85 °C on the 28th day. The results of this formulation were obtained when the amount of kaolin in the mixture was 30. The amount of kaolin in the geopolymer was found to be important in terms of compressive strength. Calcite material has been found to improve the roughness, rapid freezing and shaping of geopolymer samples

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