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Effect of Solid/liquid ratio during curing time fly ash based geopolymer on mechanical property

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Abstract. Geopolymer is produced from the alkali activation of materials rich in Si and Al such as fly ash. Based on the experimental and characterization result, solid to liquid ratio influenced the setting time and compressive strength of geopolymer in order to have good mechanical property. The optimum setting time and compressive strength were obtained at 3 : 1 solid to liquid ratio. Optimum curing time reach at 14 days.

Introduction

Geopolymers is a new family of synthetic aluminosilicate materials formed by alkali activation of solid aluminosilicate raw materials [1]. The geopolymeric systems are composed from two phases, the solid one and the aqueous one which is called activator. The solid phase is an aluminosilicate material, which contains easily dissolved silicon and aluminium in a strongly alkaline aqueous solution. This material could be a typical aluminosilicate from the solid industrial by-product such as fly ash, metallurgical slags, building demolition materials, etc. The term of alkali activation refers to the chemical dissolution of aluminoslicate raw materials in a strongly alkaline environment caused by an aqueous solution of sodium or potassium hydroxyde. Geopolymers belong to the family of inorganic polymers, which are macromolecules linked by covalent bonds and having –Si-O-M-O-backbone, where M denotes principally aluminum and secondarily other metals such as iron [1,2]. The difference between geopolymers and the other inorganic polymer lies in the kind of silicon and the aluminium precursors used for their synthesis. Geopolymers are synthesized by alkali activation of solid aluminosilicate raw materials utilizing as activator a strong alkaline aqueous solution of sodium or potassium hydroxide.

Dissolution of Si and Al from the solid aluminosilicate materials in the strongly alkaline aqueous solution. In the presence of water the surface metal ions of the aluminosilicates oxides may coordinate H_2O molecules and form hydroxylated surface sites that are well known as Silanol (>Si-OH) and Aluminol (>Al-OH) groups. These groups comprise the surface active sites, where the hydroxide ions of the alkaline solution act chemically to form surface chemical species. Under a complicated mechanism, silicon and aluminium ions are released from the surface species into solution, where they form aqueous species through the complexing action of hydroxide ions completing in this way the dissolution process[4]. In aqueous solution, the chemical dissolution of Al-Si minerals and generally of materials of aluminosilicate composition is favoured in the range of high pH values, given that the dissolution rate of these materials increases significantly as the solution pH is increased [5].

As Si and Al concentrations in the aqueous phase increase gradually, certain chemical reactions take place between the formed hydroxy-complexes. Reactions result in the formation of the geopolymers precursors that are oligomer spesies consisting of polymeric bonds of Si-O-Si and Si-O-Al type[6]. The existance of soluble silicates in the alkaline aqueous phase of the geopolymeric system enhances the formation of oligomer species. Soluble silicates in the aqueous phase increase essentially the concentration of Si, thus, alkaline silicate solutions used in the synthesis of geopolymers provide the system with the necessary silicate oligomers for the development of geopolymeric framework [7].

The increase of oligomers concentration in the aqueous phase involves their polycondensation, which in turn lead to the development of a three dimensional framework consisted of SiO_4 and/or AlO_4 tetrahedra linked alternately by sharing common oxygen ions. Polycondensation reaction involves the chemical bonding of geopolymers precursors (oligomers) by simultaneous removal of water molecules[8]. This procedure is well known as polymerization. Oligomers may react in every hydroxyl ion site, forming macromolecular chains and/or rings that reult in a three dimensional framework. Since the geopolymeric framework is developed in the aqueous phase, it comes across the active surface sites of the solid particles, where it is possible to react bonding the undissolved particles in the final geopolymeric structure[9]. There in after, hardening of polymeric matrix, which occurs as the excess of water is removed from the geopolymeric matrices during the curing procedure, may lead to durable and tough materials [10].

This paper presents the effect of solid to liquid ratio during curing time on mechanical property and microstructure of fly ash based geopolymer.

Materials and Experimental Details

Raw Materials

Class F fly ash from industrial waste was used as raw material. Laboratory grade potassium hydroxide (KOH) and distilled water was used to prepare alkaline solution. Table 1 shows the synthesis parameter of the geopolymer work.

Table 1 : Various parameter to produce geopolymer

Raw Material : fly ash	
Alkaline Solution	KOH 4.5M
Solid/liquid ratio	1:1, 2:1, 3:1, 4:1, 5:1
Curing time	1 day, 7 days, 14 days, 21 days, 28 days
Curing temperature	Room temperature (25^0C), 60^0C

Mix Proportion and Mixing Process

Mixing was done in an air conditioned room at approximately 25^0C. The mixing procedure started with mixing of KOH solution and fly ash for 5 min. Well-mixed geopolymer was used for the measurement of setting time using the Vicat apparatus according to ASTM C191. Setting time is the time required by the fresh paste for hardening, which reflected the water content in the mixture. Geopolymer cubes were also prepared by casting the geopolymer mixes in the 50mm x 50mm mould and vibrated for 5-10 minutes to reduce entrained air for compressive strength tests according to ASTM C109. Characterization was done using RAMAN Spectroscopy and FESEM to investigate the chemical bonding, chemical composition and microstructure of geopolymer.

Result and Discussion

Figure 1,shows the setting time with respect to solid/liquid ratio. Higher solid/liquid ratio decreased with setting time. The role of water content and KOH concentration influence the results. The dissolution of SiO_2. Higher solid/liquid ratio, KOH concentration will get the amount of water content and KOH concentration.

Table 2: Setting time with various solid/liquid ratio of fly ash based geopolymer

Solid/Liquid Ratio	Setting Time (minutes)	
	RT	60^{o}
1 : 1	1440	536
2 : 1	1380	180
3 : 1	50	30
4 : 1	75	35
5 : 1	40	30

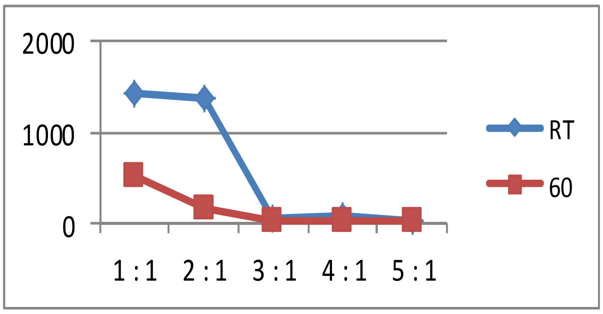

Fig. 1: Effect of solid/liquid ratio on the setting time of fly ash based geopolymer

Figure 2 shows the result of compressive stength of geopolymer samples with respect to solid/liquid ratio during curing time. The strength increased with increase of solid to liquid ratio between fly ash and alkali solution. For curing time, compressive strength increase until 14 days then decreased. In other words, the high K_2O/Al_2O_3 and K_2O/SiO_2 ratio promoted the strength enhancement. Alkali solution with higher concentration provided better dissolution of fly ash particles and generating more reactive bonds for monomer, which in turn increased geopolymerization of pastes.

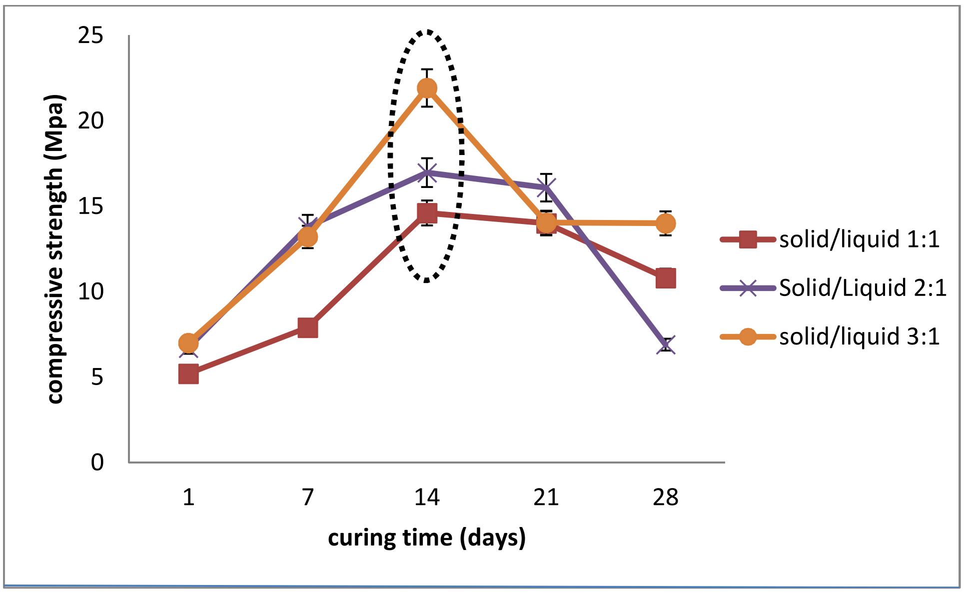

Fig. 2: Effect of solid/liquid ratio on mechanical properties of fly ash based geopolymer

Figure 3 shows the Raman pattern for geopolymer products that are produced at high compressive strength. The band between 1130-1140 cm $^{-1}$ as depicted in Figure 3 is referred to the streching mode of T-O-T (where T= Si or Al) bond.

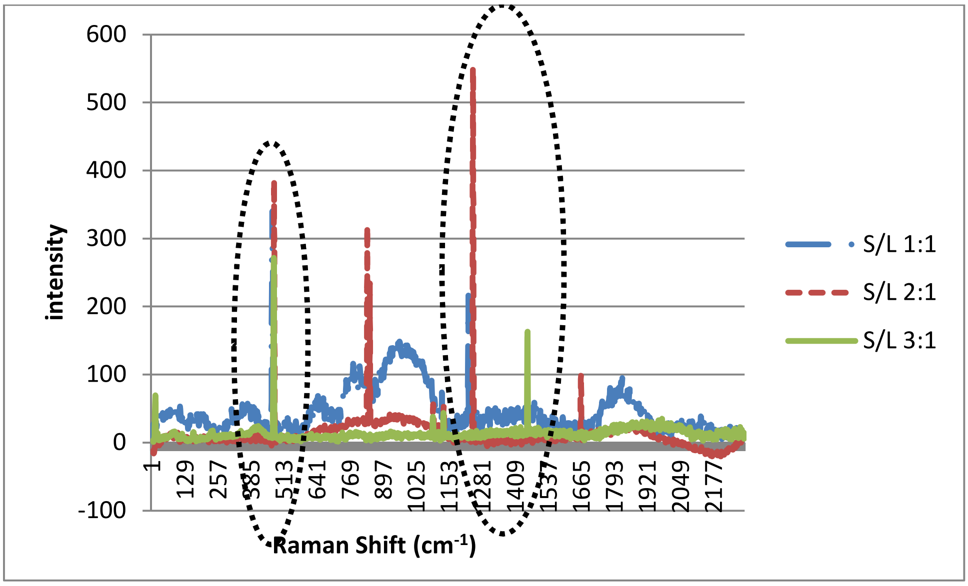

Fig. 3: Raman spectrocopy of fly ash based geopolymer with various solid/liquid ratio

With less water and KOH concentration, dissolution cannot occur completely resulting in some unreacted raw material and will decrease compressive strength.

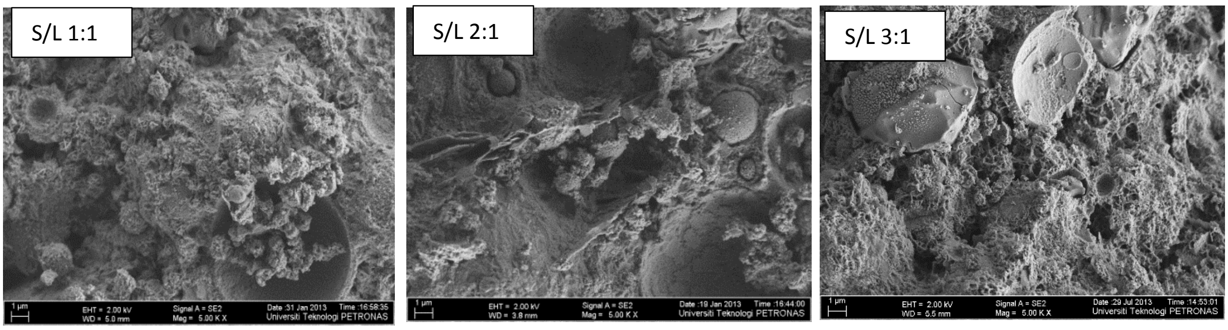

Fig. 4: FESEM result of fly ash based geopolymer with various solid/liquid ratio

Figure 4 shows the FESEM result of geopolymer with various solid to liquid ratio. Reaction between fly ash and KOH increase with increasing solid to liquid ratio. The starting materials for the geopolymeric synthesis were K_2O:Al_2O_3 in distilled water, which follow hydrothermal process for generating the Al_2O_3-$2SiO_2$-K_2O geopolymer. Upon further inspection, our analysis revealed that geopolymers contained unreacted starting materials. Setting time was too fast, causing raw materials do not have enough time to react with the activator.

Conclusion

Based on the experimental and characterization result, solid to liquid ratio influenced the setting time and compressive strength of geopolymer in order to have good mechanical property. The optimum setting time and compressive strength were obtained at 3 : 1 solid to liquid ratio. Optimum curing time reach at 14 days.

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