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Effect of Iron Oxide (Fe_2O_3) on the Properties of Fly Ash Based Geopolymer

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Abstract. Geopolymer is an attractive construction binder owing to its ability to improve the properties of the concrete and preserves the environment from the high CO_2 emission. Geopolymer technology will convert the potential hazardous industrial waste such as fly ash into valuable construction materials. However, there is a need of studying the properties of iron-based geopolymer in order to enhance the fundamental and knowledge of the geopolymer research also development in this study area. Fly ash which contains a significant amount of iron oxide (Fe_2O_3) was used as a precursor and tested at different curing duration (1, 3, 7, 14 and 28 days). Crystallization of iron oxide (Fe_2O_3) contained in the fly ash under geopolymerization process will be able to turn waste fly ash into a strong concrete materials, simultaneously creating a waste-to-wealth economy. Furthermore, the formation of fayalite detected from the microstructure characterization is mainly contribute to the strength development of the fly ash after 28 days curing.

1. Introduction

Geopolymer is an inorganic polymer which made up of two main constituent namely aluminosilicate-source materials and alkaline activator solutions [1]. Generally, aluminosilicate source materials that contain high silica, alumina, and calcium oxide minerals are commonly used in the geopolymer production. The combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) has been used widely as the alkaline activator in liquid form [2].

Fly ash, slag, kaolin and metakaolin are examples of aluminosilicate source materials which gives different behavior, properties and specialities depends on many factors [3-5]. One of the factors affecting the properties of geopolymer final product is the composition of aluminosilicate source materials. Fly ash is by far the most commonly used as a precursor for geopolymer concrete due to its commercial and performance reasons. It is an industrial by product which residue resulting from the combustion of coal [6]. Fly ash generally can be categorized into two classes which are class F and class C and the content of calcium in the chemical composition is one of the indicator to make the classification. Silicon oxide (SiO_2) and aluminium oxide (Al_2O_3)



can be found mostly in fly ash thus making it a suitable source of aluminum and silicon for geopolymerization [7].

Geopolymer becomes an attractive research due to its ability to improve the concrete properties and preserve the environment from the high CO₂ emission from the concrete production. In addition, the geopolymer technology will convert the potential hazardous industrial waste such as fly ash, bottom ash, slag and etc. into the valuable construction materials [8]. However, it is a very limited study that reported on the performance of high iron based geopolymer. The current research and development of geopolymer usually focussing on high silica and alumina based geopolymer due to its high resistance against temperature and high durability.

Over the past few years, iron based geopolymer have attracted increasing attention and have been considered for applications such as repair materials and etc. Dimitros et al. (2009) produces geopolymer from red mud and achieved very high strength and high durability when exposed to high temperature [9] and also excellent applied as flue gas desulfurization sorbent [10]. While Pacheco et al. (2008) explored tungsten mine waste based geopolymer and obtained very good mechanical properties of geopolymer [11]. However, the effect of iron oxide is not thoroughly discussed and reported. Hence, there is a clear need of searching and studying the properties of high iron based geopolymer to enhance the current state of the geopolymer research and development in this study area. Thus, this study focuses on the producing a geopolymer by using high iron content in the selected fly ash and mixed with alkali activator and left hardened under geopolymerization process at room temperature. The effects of the iron oxide to the microstructure and mechanical properties of geopolymer were assessed.

2. Experimental details

Fly ash has been used as the main aluminosilicate source materials and mixed with the alkali activator solution to produce geopolymer. The combination of sodium silicate (Na₂SiO₃) solution and 12 Molar sodium hydroxide (NaOH) solution at ratio 2.5 was used as the alkali activator solution [12]. The physical and chemical characteristics of the fly ash have a significant role in determining the properties of the fly ash based geopolymer. Thus, compressive strength and microstructure characteristics were carried out to explore the properties of high iron based geopolymer.

2.1 Chemical Composition Characterization

The chemical composition characterization of fly ash was determined by using a bench top X-ray fluorescence (XRF) spectrometer with a brand name PAN analytic PW4030, MiniPAL 4, X-Ray Fluorescence (XRF) spectrometer. The chemical composition was reported in the percentage by weight of the sample.

2.2 Microstructure Characterization

The microstructure of the fly ash and fly ash based geopolymer were obtained using JSM-6460LA model Scanning Electron Microscope (JEOL) utilizing the secondary electron detectors. All specimens were coated with palladium by using Auto Fine Coater JEOL JFC 1600 model before test. At the same time, the surface elemental composition was taken by Energy Dispersive X-ray (EDX) spectroscopy using the same procedure.

2.3 Compression Test

The compressive strength was evaluated using Instron machine series 5569 Mechanical Tester with a loading capacity of 3000 kN using a loading rate of 0.5 MPa/s. Compressive strength was performed on cubes with the dimensions of 50 mm × 50 mm × 50 mm according to ASTM C109 [13]. The average of three specimens was tested to determine the compressive strength of geopolymer mortar and the mean value of three samples was reported.

3. Physical and chemical composition analysis of fly ash

3.1. Physical analysis of fly ash

Morphological studies of fly ash and sand have been conducted using Scanning Electron Microscope (SEM) as shown in Figure 1. The spherical particles structure of raw fly ash can be seen clearly in the Figure 1. Existing of the ferrosialate and cennosphere with small particle fly ash is from the special source of coal which able to improve geopolymerisation rate and shows high reactivity. Thus, it is believed that different structure of fly ash will have impact to the mechanical properties.

The ferrosphere (dendritic type) as shown in the figure was identified. Ferrosphere contains very high iron oxide and this kind of particles structure was rarely observed in the fly ash. The ferrosphere has a significant impact in the formation of geopolymer gel. Needle crystallites on the surface of ferrosphere indicate that crystallizes from amorphous aluminosilicate melt during combustion of fly ash but not having transformed directly from fly ash [14].

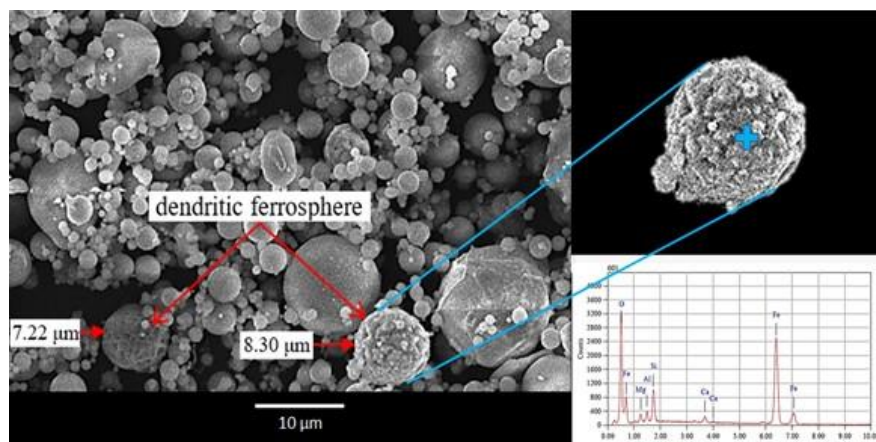


Figure 1. Ferrosphere shape of fly ash.

Needle crystallites of ferrosphere were capable of raising the strength of fly ash based geopolymer which was good for the long term durability.

3.2. Chemical composition analysis of fly ash

X-ray fluorescence (XRF) was used to identify and measure the chemical composition of fly ash as shown in Table 1. The main chemical composition of fly ash is silica oxide SiO_2 , followed by Fe_2O_3 , Al_2O_3 , and CaO . The content of SiO_2 , Fe_2O_3 and Al_2O_3 are 40%, 23% and 14% and sum of these oxide minerals are more than 70 %. Fly ash is classified as Class F and suitable to be used as raw material for geopolymer as referred to American Society of Testing and Materials (ASTM C618) as shown in Table 2 [15]. It also indicates that this fly ash classified as pozzolanic material and has cementitious properties [16]. Those SiO_2 and Al_2O_3 will undergoes geopolymerization to form aluminosilicate gel or known as (N-A-S-H) gels.

Table 1. Chemical composition of fly ash from different sources.

Chemical	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MnO	CuO	K_2O	Na_2O	SO_3	SrO
Fly ash (wt.%)	38.8	14.7	19.48	1.02	18.1	0.16	0.04	1.79	-	1.50	0.11

Table 2. Fly ash classification.

Chemical	wt.%	ASTM C618 (%)
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	72.98	70.00

Based on the chemical composition testing, iron (III) oxide content (Fe_2O_3) was the second highest mineral oxide that exist in fly ash which was 19.57 %. The content of Fe_2O_3 from the source material was probably have a positive impact on the durability in term of final compressive strength of geopolymer. The atomic diameter of iron was higher than silica and alumina, thus the high content of iron in the raw materials may longer the geopolymerization reaction with alkali solution to form iron silicate binder. Thus, the longer aging duration of the geopolymer sample left at room temperature help to enhance the strength development of geopolymer. The significant amount of iron oxide content in the raw materials gives a good indication to improve the overall properties of geopolymer [17-20].

3.3. Compressive strength of fly ash based geopolymer at different curing duration

Trends for compressive strengths against different curing duration were observed. The purpose of curing is to identify the appropriate period of sample to gain the desired strength and allow for the further development of its optimum. Figure 2 shows that the compressive strength of geopolymer strength increase as the curing time increased. Geopolymerisation is a time dependent process that may continuously.

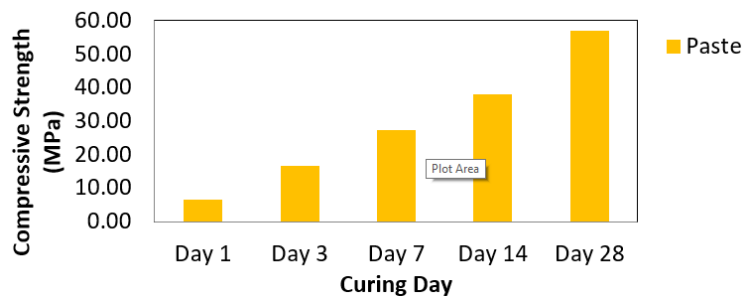


Figure 2. Compressive strength of geopolymer with different curing time.

The increment of the compressive strength between 14 days and 28 days of curing of fly ash based geopolymer was about 38 %. It was also noticed that the strength development of geopolymer differs significantly at others curing duration. This can be explained by the high percentage of iron (Fe_2O_3) available in the fly ash which is about 19.57 %.

Fly ash with high content of iron (19.57 %) takes time to react with the alkali solution in the geopolymer system in order to form the iron silicate binder gel. This was due to the high atomic mass and large atomic diameter of the iron compared to silica and alumina. Cannio et al. (2018) identified the ferrosialate in addition to the sodium aluminosilicate gel in the geopolymer which contribute to the durability of the geopolymer [20-22]. Thus, the longer curing duration led to increase the compressive strength which was contributed by the iron oxide.

3.4. Morphology of fly ash based geopolymer

The microstructure analysis for fly ash based geopolymer paste cured at different curing days are presented in Figure 3. The microstructure of selected samples were cured at different aging duration which presented by the 1 day and 14 days aging as shown in Figure 3 (a) and Figure 3 (b), respectively. It was found that a denser matrix has been obtained at 14 days of curing time compared to that obtained at 1 day. When compared between at 1 day with 14 days ageing period, compressive strength increased with sample age.

Ferrosphere are well known iron rich spheres with a chemical composition that rich in iron oxide (Fe_2O_3). The EDX analysis for the early curing ages (at 1 day) marked with 'X' at Figure 3 (a) has found that this partially reacted fly ash is rich in iron. This kind of fly ash microstructure is formed by

the conglutination after the oxidation of iron bearing minerals contains high amount of iron with more than 80 %. The white needle like structures shows the reaction on the surface and formed from the crystallization of iron oxide and from the geopolymerization between iron and alkali activator solution but requires time (Xue et al., 2008).

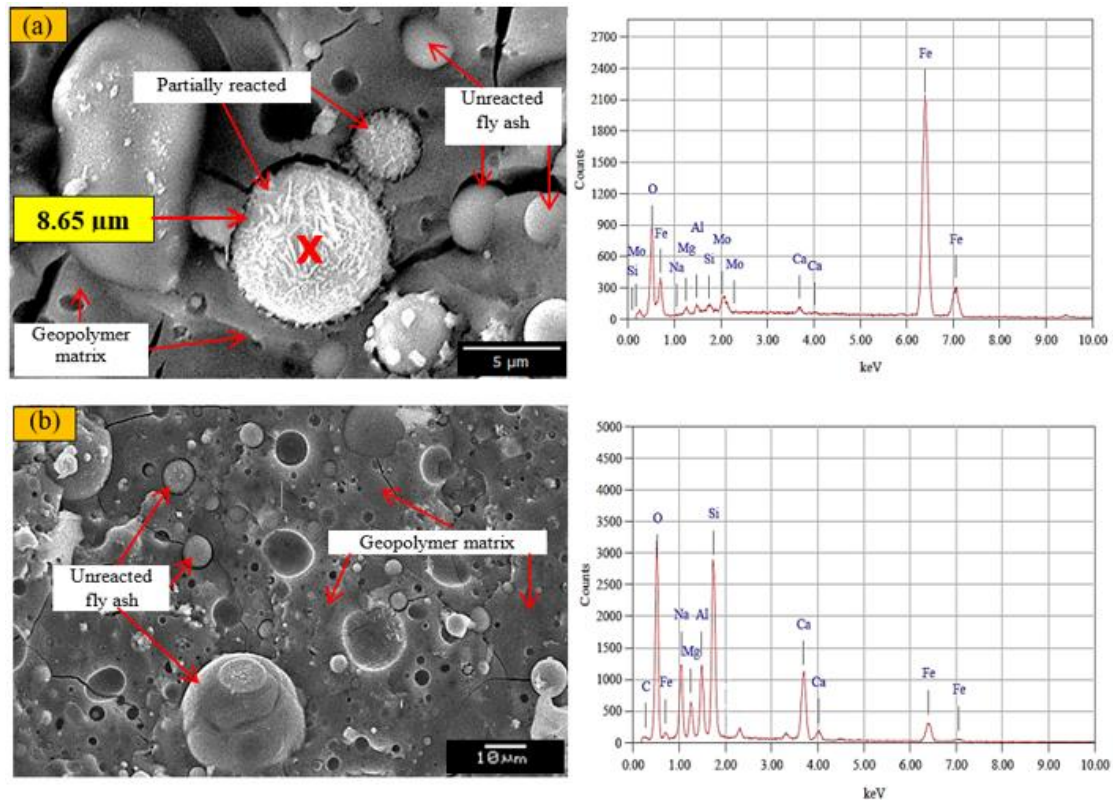


Figure 3. Microstructure of geopolymer samples (a) 1 day curing and (b) 14 days curing.

This EDX results confirmed that this shape is one of the ferrosphere particles which is partially reacted at early age according to the high percentage of iron element content.

4. Conclusion and perspectives

The properties requirement as grouting material led us to propose the optimum grout geopolymer. From the behaviour of the geopolymer, this clearly highlighted that the use of geopolymer is potentially to provide and increased stiffness under services condition and the high tensile strength of geopolymer. Thus, the main purpose of this paper is to give an overview on the most recent studies using polymer based materials and geopolymer with different raw material as an excellent repair material to developed and applied to solve civil engineering problems indeed. Geopolymer is compatible with the standard requirements and shows the potential to become as an excellent repair material. Geopolymer are good in durability and high mechanical strength thus they can be an alternative as an excellent repair materials.

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