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Accelerating the Polymerization Process of Fly Ash by Microwave

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Abstract: Fly ash is the solid waste from thermal power plants. It is a big problem with ecology. Using fly ash in addition to the Portland cement is the beneficial resolution, the most rest have been landfilled. On other hand, fly ash is an aluminosilicate containing amorphous silica with fine sizes, it can react very well in alkaline solution and then condense to form new bonds. In this study, fly ash was used in a chemical reaction with alkaline activators to produce a new form of concrete, known as geopolymer. The reaction time has been shortened by using a technique of semi – dry pressing and microwave oven heating. The new binder formed by this method has been tested by modern analytical methods such as Röntghen diffraction analysis (XRD), Infrared Spectrum (FTIR), and Scanning Electron Microscope (SEM). The results indicated that the geopolymer products with a relatively high compressive strength of about 50 MPa are suitable for use as backfill materials, reclamation materials, and unbaked building materials.

Keywords: Fly Ash, Geopolymer, Microwave Oven

1. Introduction

Fly ash is a form of solid waste generated from power plants. In many developing countries, fly ash is considered a major environmental and social issue since thermoelectricity is still produced in high proportion in these countries. Currently, the fly ash is mainly used as an active mineral additive for Portland cement. However, lost in the ignition (L.O.I.) of fly ash must be less than 6% by weight to be used as an additive for cement. Another important application of fly ash is in the building material industry. Fly ash, in combination with Portland cement as an adhesive, is used to produce non-baked materials.

From the perspectives of many environmental protectionists, in order to control climate change, it is necessary to limit the emission of CO and CO_2 into the air. In fact, for every ton of Portland cement produced, there will be 1 ton of CO_2 emitted into the environment [1]. Thus, the production of Portland cement also needs to be reduced to minimize the greenhouse effect. One possible solution to reduce the use Portland cement is substituting from Portland cement to geopolymer-based concretes. The fly ash can be created new bonds to successfully used in construction. The bonds could be produced by a polymerization reaction of alkali solutions with aluminosilicate available in the fly ash. These bonds are named "geopolymer" [2, 3].

When alkaline compounds (ex: NaOH, KOH, water glass, etc.…) react with aluminosilicate (Si – Al) compounds, hydrated aluminosilicate compounds will be formed. Next, the hydrated aluminosilicate compounds will dehydrate to form polymer chains of silicon and aluminum oxides, Si – O – Al – O. According to Davidovits (1994) [4], the reaction process is as follows:

$$\underset{(Si-Al\ material)}{n(Si_2O_5, Al_2O_2)} + 2nSiO_2 + 4nH_2O + NaOH(or KOH) \rightarrow Na^+(or\, K^+) + n(OH)_3 - Si - O - \underset{\underset{(OH)_2}{|}}{Al^-} - O - Si - (OH)_3 \quad (1)$$

(Geopolymer precursor)

$$n(OH)_3 - Si - O - \underset{\underset{(OH)_2}{|}}{Al^-} - O - Si - (OH)_3 + NaOH(or\, KOH) \rightarrow (Na^+, K^+) - (\overset{|}{\underset{\underset{|}{O}}{Si}} - O - \overset{|}{\underset{\underset{|}{O}}{Al^-}} - O - \overset{|}{\underset{\underset{|}{O}}{Si}} - O -) + 4H_2O \quad (2)$$

(Geopolymer backbone)

Raw materials used in the aluminosilicate compounds are often solid industrial wastes such as: fly ash [5, 6], clay and kaolin [7], red mud and diatomite [8], etc.… Referring to equation (1) above, it is clearly that water plays a very crucial role in the formation of the alkaline solutions that break the bonds of the aluminosilicates, or SiO_2, to provide good workability of geopolymer. After that, water will escape from the solution to form a chain-shaped or ring-shaped polymer structure, which finally results in the formation of the solid linkage of the geopolymers (referring to equation (2)). This breaking process of water is slower than that of the original materials. In this case, water can be separated by curing and drying under relatively high temperatures (60 – 80°C) for a long period of time. High water content will make the alkaline easy to dissolve, but it will reduce the intensity of the material [9]. Microwave treatment is a new technique to reduce geopolymerization time [10].

Many studies on geopolymer have been conducted in many countries around the world. However, many issues such as high cost of caustic soda (NaOH, KOH, etc….) and water glass, unified technique of manufacturing unburned material, content of alkaline residue, duration of long term development, problem of longevity, etc.…still remain unsolved. As a result, the real world application of geopolymer based concrete is still limited.

In this study, the geopolymer samples based on the fly ash were obtained from a coal power plant in South of Viet Nam. To shorten the curing and drying time, a semi-dry pressing and heating techniques using a special mixer and a microwave were applied to minimize the amount of water in the substance. Using this technique, the time to produce geopolymer material from fly ash was shorten to only a few minutes. The quality tests of the product show that the materials were geopolymerized and suitable for using as building aggregates and building materials.

2. Experimental Procedure

First, fly ash was mixed with alkaline activators. The alkaline activators include sodium silicate (Na_2O 15%, SiO_2 43% H_2O 42%) and sodium hydroxide flakes (98% Na_2O). The ratio of alkaline activators to fly ash was 12% by weight. The substance was then pressurized at 20 MPa. The two processes were carried out in a mixing and pressing equipment.

After shaping, the samples were put into a microwave oven. The heating time in the microwave oven was 4 minutes at a temperature up to 160°C. The formation process of geopolymerization products is summarized in the Figure 1.

***Figure 1.** The Formation Process of Geopolymerization Products.*

The quality of the geopolymerization product was tested by modern analytical instruments such as Röntghen Diffraction Analytic (XRD), Infrared Spectroscope (FTIR), and Scanning Electron Microscope (SEM). The compressive strength of geopolymerization product was determined by Hydraulic Compression Testing Machine STYE-2000.

***Table 1.** The XRF analysis results of fly ash.*

Oxides	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	K_2O	CaO	TiO_2	-	L.O.I
% weight	52.64	21.36	5.31	0.69	0.41	4.88	0,43	0,30	12,85

3. Results and Discussion

3.1. Chemical Composition of Raw Materials

The average particle size, which was determined by Lazer analysis, was about 48.2 mm. The chemical composition of raw material was determined by using X – ray fluorescence. The results are shown in Table 1. Accordingly, the results demonstrate that fly ash is mainly composed of SiO_2, Al_2O_3, Fe_2O_3, and CaO. The composition of oxides of aluminum and silica is suitable for geopolymerization process.

3.2. Results of XRD Analysis

XRD – spectra of fly ash and geopolymer concrete are showed in figure 2 (a and b). The result of XRD spectrum analysis of fly ash sample (Figure 2a) indicates that crystal compositions of fly ash are quartz, cristobalite, and mullite.

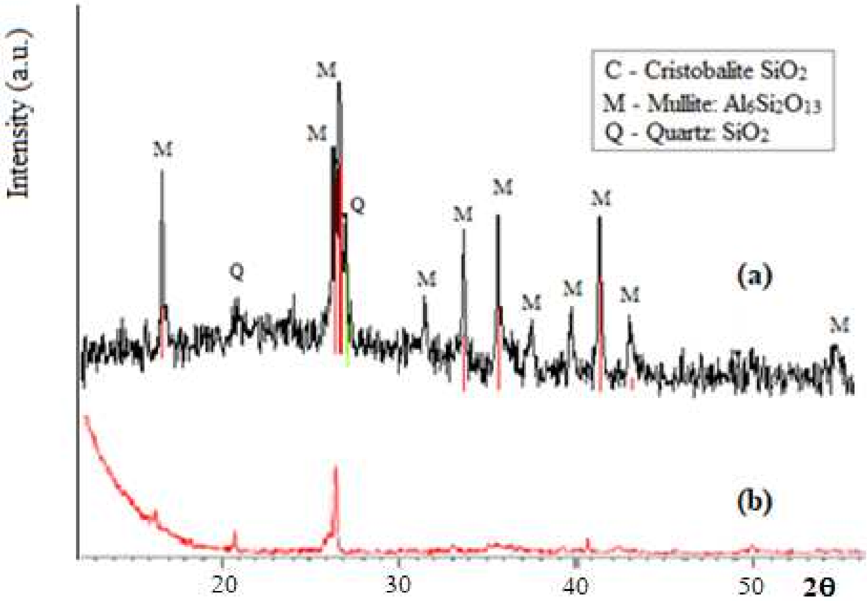

Figure 2. *The XRD spectra of fly ash (a) and geopolymer concrete (b).*

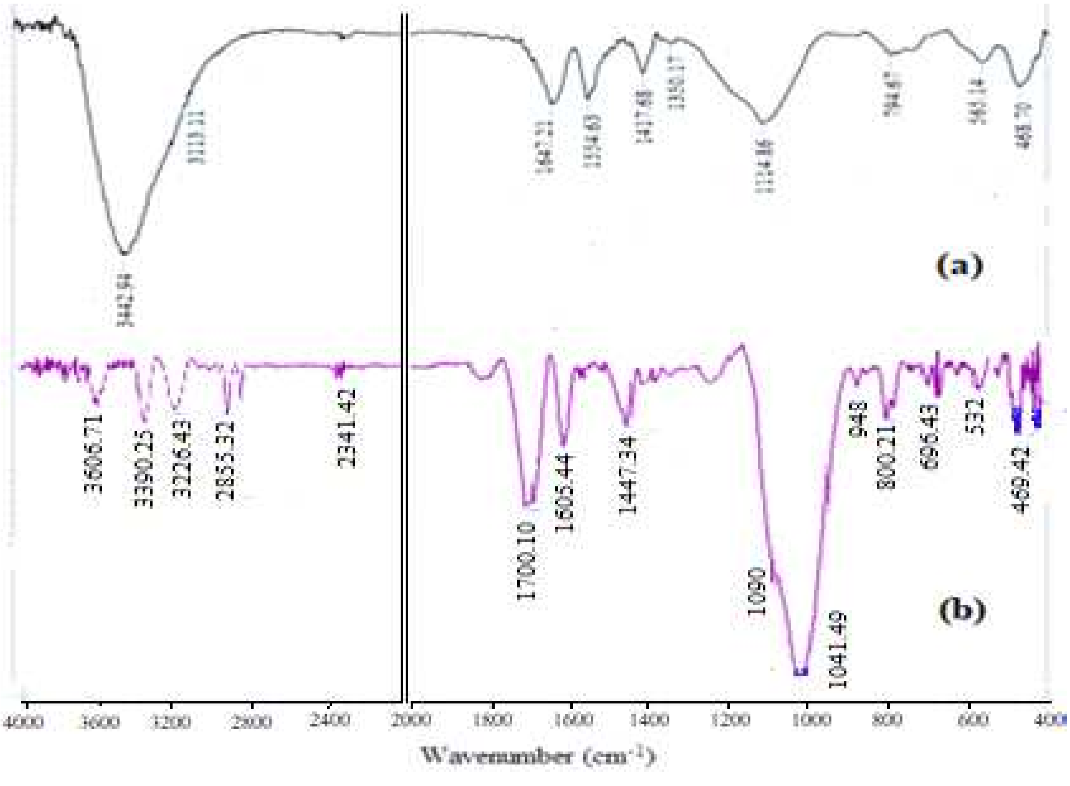

Figure 3. *FTIR spectra of fly ash (a) and geopolymer concrete (b).*

According to the XRD spectrum of the polymerized concrete sample (Figure 2b), the characteristic peaks of the crystals are almost invisible. There is only one peak at 2θ= 26,7°, which is the characteristic peak of crystalline quartz. The XRD spectrum of polymerized concrete exhibits its amorphous structure. This is also a signal that the sample has been polymerized quite well.

3.3. FTIR Analysis

The spectra of FTIR analysis are showed on Figure 3. The FTIR spectra of the fly ash (figure 3a) demonstrate the same crystalline patterns as the ones found in analysis of the XRD spectrum. These are crystalline quartz and mullite. Furthermore, the FTIR spectrum clearly shows amorphous links of - OH group, unburned carbon, and SiO_2 amorphous.

On the FTIR spectrum of the geopolymer concrete (Figure 3b), the characteristic peaks on the FTIR spectrum of the fly ash have disappeared, resulting in new peaks that characterize the links of the geopolymers. Those are the bonding vibrations, Si – O – Al, of geopolymers such as broad peaks at 1000 – 1010 cm^{-1} [16] and at 964 cm^{-1} [17]. The characteristic peaks of the Al(IV) – O – Si, Al – O – Si, and Si – O – O links of the geopolymers also appear at wavenumber 710, 600, and 450 cm^{-1}. The results are summarized in Table 2.

Table 2. *Wavenumber and characteristic bonds from FTIR spectra (Figure 2a).*

	Wavenumber (cm^{-1})	Bonds	References
	3442.94 and 1647.21	– O – H Asymmetric stretching of water and silanol group	[11]
	1137, 882, 751, 567	mullite	[11]
	713	Ca – O	[12]
(a)	1417, 866	C = O	[12]
	1055, 790, 696, 532, 469	Quartz crystal	[13]
	3390, 1690, 1190, 1090, 948, 800	SiO_2 amorphous	[14]
	1633, 1396, 1056	Armorphous carbon	[15]
	1000 – 1010 (broad peak)	Si – O – T (T: Si or Al) bonding vibrations of geopolymer	[16]
	964	Geopolymer	[17]
(b)	710	Al(IV) – O – Si Bending vibration of geopolymers	[18]
	600	Al – O – Si	[18]
	450	Si – O – Si	[18]

3.4. SEM Analysis

Figure 4 includes SEM images of fly ash (2a) and geopolymer concrete (3b). These images also show that fly ash particles have been reacted since their origin shape has been changed.

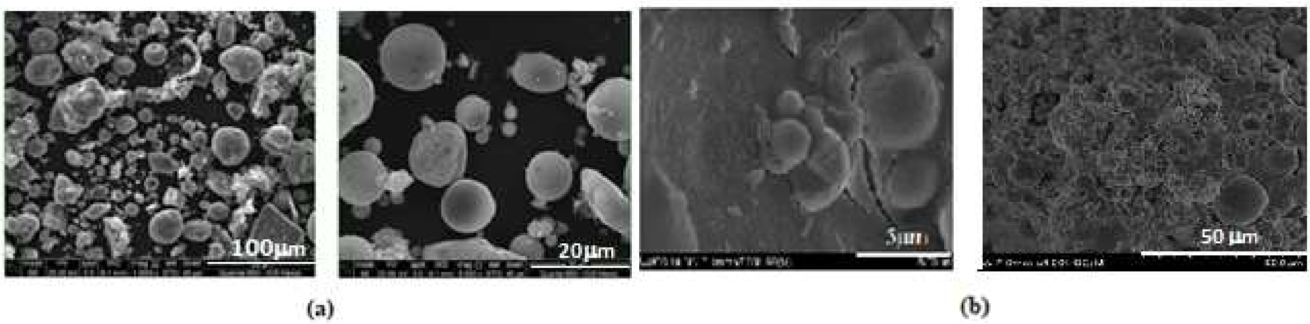

Figure 4. *SEM images of fly ash (a) and geopolymer concrete (b).*

3.5. Compressive Strength

The compressive strength of geopolymerization products, which was determined by Hydraulic Compression Testing Machine STYE-2000, reached approximately 50 MPa.

4. Conclusion

The geopolymerization process of fly ash occurs very fast due to the effect of microwave oven. Products with high compressive mechanical strength are suitable for using as backfill materials, reclamation materials, and unbaked building materials. In conclusion, this is a potential technique that can promote greater use of fly ash in construction industry.

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