

Strength and microscopic characteristics of alkali-activated fly ash-cement

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(Received 9 May 2005 • accepted 6 December 2005)

Abstract—The effects of different activator concentration, liquid/fly ash ratio, and curing temperature and time on the compressive strength of specimens prepared from low-calcium fly ash activated with sodium hydroxide without the use of Portland cement were investigated. SEM, XRD and mercury intrusion porosimetry (MIP) were used to observed the structural feature, reaction products, and porosity and pore-size distribution of the specimens from alkali-activated fly ash, respectively. It was found that the degree of reactivity, as shown by the compressive strength, the activator concentration and the ratio of liquid/fly ash, and the curing temperature always result to be significative factors. The 7, 14, and 28-day compressive strengths of specimens prepared from alkali-activated fly ash by 5 M NaOH solution at 50 °C are 152, 219, and 263 kgf/cm², while those from 6 M solution are 184, 225, and 267 kgf/cm², respectively. In SEM observation, the fly ash activated by the 5 M NaOH solution shows a more continuous matrix with solid and non porous due to subsequent gel restructuring by amorphous alkaline aluminosilicate produced from alkali-activated fly ash.

Key words: Fly Ash, Alkali-Activation, Compressive Strength, Reaction Products, Porosity

INTRODUCTION

The demand for industrial and domestic energy results in the production of a large volume of fly ash from solid coal fuel, which will increase in the world on an unprecedented scale during the next years (800×10⁶ tons by the year 2010). Therefore, fly ash should not only be disposed of safely to prevent environmental pollution, but should be treated as a valuable resource [Guerrero et al., 1999]. Traditionally, fly ash have been used as pozzolanic material to enhance physical, chemical and mechanical properties of cements and concretes. However, only amounts of 20-30% of fly ash are currently used for this purpose while the remaining 80-70% is landfilled or surface-imprisoned. The enormous amount of fly ash that is disposed of each year represents the waste of a valuable engineering material.

Luxan et al. [1989] determined that most fly ashes from the combustion of coal are made up of aluminosilicate and silica glasses with small amounts of crystalline materials, including mullite, quartz, hematite, and magnetite. In case of partial substitution of fly ash for Portland cement, the glasses in these ashes are sometimes pozzolanic, consuming calcium hydroxide upon hydration [Williams et al., 2002] and lead to the formation of additional calcium silicate hydrate (C-S-H) in the system, resulting in density and strengths equal to or greater than that for pure Portland cement. These new cements produced from partial substitution of fly ash are also advantage of reducing energy cost and environmental deterioration (almost exclusively usage of secondary raw materials instead of surface-excavated natural resources) [Puertas and Fernández-Jiménez, 2003].

In spite of these advantages, its strength gain and slow reactivity have been considered as obstacles in field application. To improve

these problems, researches have focused on developing the methods to gain the higher strength and reactivity of fly ash. The reactivity of a fly ash depends upon many parameters, most important of which are chemical and mineralogical composition of fly ash [Arjunan et al., 2001], curing temperature [Bakharev et al., 1999; Ma et al., 1995; Pietersen et al., 1990] and the use of chemical activators [Fernández-Jiménez et al., 1999; Shi and Day, 2000].

The reaction temperature plays an important role in this activation. The reaction rate was very low at ambient temperature while the reaction rate notably increased at the temperatures between 40 °C and 85 °C, which was making it possible to obtain mortars with 20 MPa at 2 h of curing. The reaction product obtained was, in this case, an inorganic polymer of amorphous nature formed by hydrated aluminosilicates chains. This polymer structure depends on the activator used. A strongly alkaline solution to 'activate' aluminosilicate solid dissolution is required as chemical activators [Palomo et al., 1999].

Puertas et al. [2000] studied the strength behaviour and hydration products of fly ash/slag pastes activated with activator concentration (NaOH 2 and 10 M) and curing temperature (25 °C and 65 °C). At 28 days of reaction, the mixture 50% fly ash/50% slag activated with 10 M NaOH and cured at 25 °C, developed compressive mechanical strengths of about 50 MPa. The main reaction products in these pastes are a hydrated calcium silicate, like C-S-H gel, with high amounts of tetracoordinated Al in its structure, as well as Na ions in the interlayer spaces. To utilization higher quantities fly ash as valuable material, in a recent study the conversion of low-calcium fly ash into cementitious material without Portland cement has generated considerable interest. Majiling and Roy [1993] have suggested a method to convert fly ash into cementitious material without the use of Portland cement.

In this study, therefore, the effects of activator concentration, liquid/fly ash ratio, and curing temperature and time on the compressive

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Table 1. Chemical composition of fly ash (%)

Element	Content (%)	Element	Content (%)
SiO ₂	62.6	K ₂ O	1.8
Al ₂ O ₃	22.4	SO ₃	0.6
Fe ₂ O ₃	6.2	TiO ₂	1.2
CaO	2.8	P ₂ O ₅	0.4
MgO	0.7	MnO	0.1
Na ₂ O	0.6	Ig. loss	6.1

strength of specimens prepared from low-calcium fly ash activated with sodium hydroxide without the use of Portland cement were investigated. Ultimately, the research identified the proper alkali-activation conditions of low-calcium fly ash by analyzing compressive strength, and converted low-calcium fly ash into cementitious material without Portland cement. We also identified the reaction products, structural features, and porosity of specimens prepared from alkali-activated fly ash.

EXPERIMENTAL

1. Materials

Fly ash produced from coal-fired electric power stations in Korea were used as cementitious materials in this work. This fly ash is a low-calcium type and is designated as a Class F fly ash in accordance with ASTM standard C 618. The chemical composition, as determined by analysis using a Philips PW2404 sequential X-ray fluorescence (XRF) spectrometer, is shown in Table 1.

2. Sample Preparation

The cement pastes consist of 40-70 wt% solid, and the rest is liquid. Sodium hydroxide (NaOH) was used as alkali activator. The solid portion was low-calcium Class F fly ash (less than 2.8% CaO). The liquid part contains sodium hydroxide and water. The liquid (solution of sodium hydroxide) was prepared by using water and the amount of NaOH needed for the desired concentration. Mixing of fly ash and liquids was done by a small HJ-1150 mortar mixer at a low speed. The cement pastes were prepared in cubic moulds of 5×5×5 cm and placed in an oven at 20 °C. After the mix had hardened sufficiently (after one day) it was demolded and placed again in the oven at the curing temperature for activation time. The compressive strength of all specimens prepared from the liquid/fly

ash ratio of 0.3-0.6 at 50 °C for 7 days by using eight sodium hydroxide concentration (1-8 M) was tested in the first part of the experimental program. On considering the results, it was decided to test the effects of the curing temperature and time on 5 M and 6 M with fixed the liquid/fly ash ratio of 0.4. A summary of the testing program can be seen in Table 2.

3. Analysis Methods

The compressive strength of all specimens prepared from the testing program given in Table 2 was determined by KS L 5105. All the values collected were the average of three separate tests. In order to determine the influence of activation conditions on reaction products, microstructure and porosity of fly ash-cement pastes, the specimens were stored in a sealed container. A JSM-6400 scanning electron microscope (SEM) was used to observe the structural features of the specimens. X-ray diffraction patterns were obtained from several specimens by using a Rigaku D/MAX-III B X-Ray diffractometer. Porosity and pore-size distribution of several specimens were investigated by mercury intrusion porosimetry conducted with a Micromeritics AutoPore IV 9510.

RESULTS AND DISCUSSION

1. Mechanical Strength Behaviour

The compressive strength of material is defined as the ability to resist stress without failure. In cementitious materials, compressive strength is of primary importance [Rostami and Brendley, 2003]. Optimization of sodium hydroxide concentrations and liquid/fly ash ratios for alkali-activation was carried out using the low-calcium fly ash at 50 °C for 7 days. The 7 day compressive strength obtained for various alkali concentrations and liquid/fly ash ratios is given in Fig. 1. The analysis of Fig. 1 indicates that, in all liquid-to-fly ash ratios, the compressive strength increased with the concentration of the sodium hydroxide solution. As can be observed in the Fig. 1, a low concentration of 1 M and 2 M has no positive effect in strength development. It was due to the relatively lower concentration of OH⁻. After 7 days in all liquid-to-fly ash ratio, the compressive strength was very low (9 and 13 kgf/cm² of 1 M and 2 M, respectively), due probably to weak van-der-Waals forces between the small fly ash particles and not to the formation of hydration products [Katz, 1998]. Solution concentration of 3 M and 4 M yielded compressive strength values between those produced by 2 M and 5 M solutions (see Fig. 1), which indicates that the degree of reac-

Table 2. Summary of the testing program for alkali-activated fly ash-cement

NaOH concentration	1 M	2 M	3 M	4 M	5 M	6 M	7 M	8 M
Liquid/FA (wt%)	0.3 : 1.0	0.3 : 1.0	0.3 : 1.0	0.3 : 1.0	0.3 : 1.0	0.3 : 1.0	0.3 : 1.0	0.3 : 1.0
	0.4 : 1.0	0.4 : 1.0	0.4 : 1.0	0.4 : 1.0	0.4 : 1.0	0.4 : 1.0	0.4 : 1.0	0.4 : 1.0
	0.5 : 1.0	0.5 : 1.0	0.5 : 1.0	0.5 : 1.0	0.5 : 1.0	0.5 : 1.0	0.5 : 1.0	0.5 : 1.0
	0.6 : 1.0	0.6 : 1.0	0.6 : 1.0	0.6 : 1.0	0.6 : 1.0	0.6 : 1.0	0.6 : 1.0	0.6 : 1.0
Curing temp. (°C)	50	50	50	50	20	20	50	50
					50	50		
					80	80		
Curing time (day)	7	7	7	7	7	7	7	7
					14	14		
					28	28		

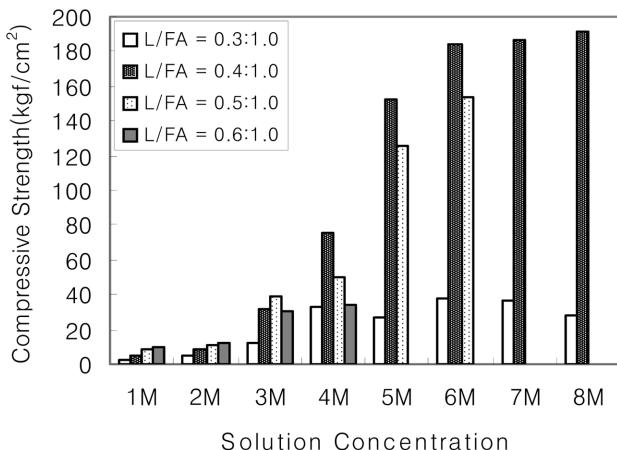


Fig. 1. Compressive strength of specimens prepared from alkali-activated fly ash with different activator solution concentration and liquid/fly ash (L/FA) ratio at 50 °C for 7 days.

tivity increases with increasing NaOH concentration.

Higher compressive strength was obtained when the experiments were conducted at equal or higher of 5 M activator concentration at liquid-to-fly ash ratio of 0.4. However, the 7-day compressive strength yielded from 7 M (187 kgf/cm²) and 8 M (192 kgf/cm²) in the liquid/fly ash ratio of 0.4 at 50 °C is slightly higher than that at 5 M (152 kgf/cm²) and 6 M (184 kgf/cm²) in same conditions. These results indicate that an alkali-activated system requires a strongly alkaline solution to 'activate' aluminosilicate solid dissolution [Davidovits, 1994; Palomo et al., 1999]. In addition, the sodium hydroxide concentrations of 5 M and 6 M were adequate for the alkali activation effect for the low calcium fly ash. Arjunan et al. [2001] explained for the alkaline activation of fly ash that NaOH attacked the surface of the fly ash particles at a high alkaline pH during the processing of the fly ash. The corroded surface provided more surface area for further attack by sodium hydroxide. As more surface of the fly ash particle was exposed by sodium hydroxide, the surface area became no longer smooth by chemical attack, and consequently, the formation of amorphous aluminosilicate was promoted [Puertas and Fernández-Jiménez, 2003; Davidovits, 1994; Van Jaarsveld and Van Deventer, 1997]. This is one of the reasons for the improved compressive strength of specimens produced from alkali-activated fly ash.

In this series of experiments, the effect of the liquid/fly ash ratios is also given in Fig. 1. At a low liquid/fly ash ratio of 0.3, the compressive strength was very low in all sodium hydroxide concentrations. It was estimated that these results produced due to no formation of activation products by lowering the water and sodium hydroxide to fly ash ratio in lowering the liquid/fly ash ratio. In higher liquid/fly ash ratio of 0.5 and 0.6, excess water except for necessary for the reactive process resulted in the formation of pore in specimens prepared from fly ash. The pores formed like this in a matrix had an adverse effect on the compressive strength of specimens, resulting in low compressive strength. In particular, the specimens produced from 5 M and 6 M at liquid/fly ash ratio of 0.6, and 7 M and 8 M at liquid/fly ash ratio of 0.5 and 0.6, did not cube and distort due to excessive sodium hydroxide. The results of 7-day compressive strength developed at 50 °C showed that the sodium hydro-

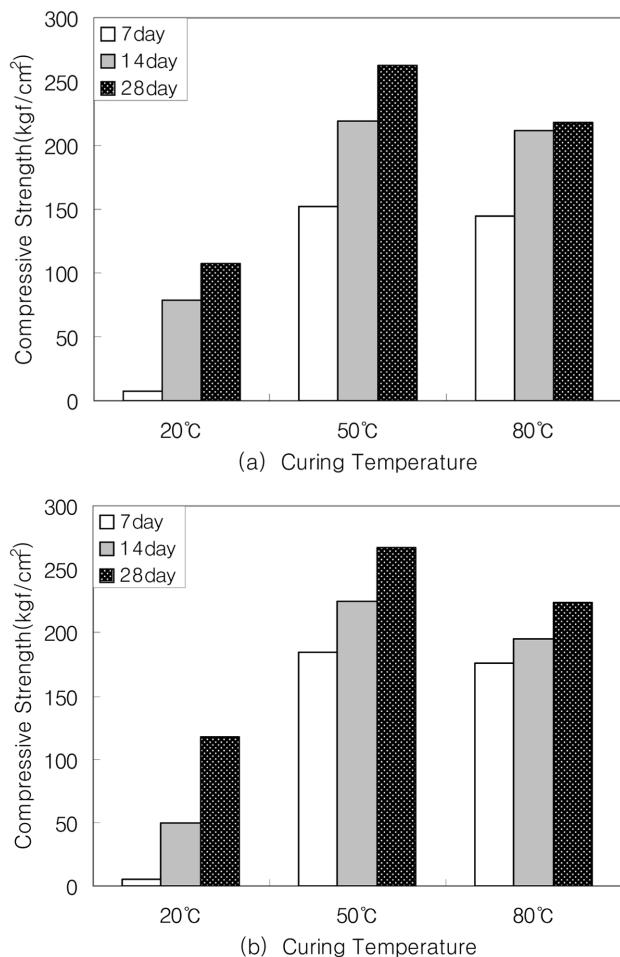


Fig. 2. Compressive strength of specimens prepared from alkali-activated fly ash with different curing temperature and time at liquid/fly ash ratio of 0.4 and activator solution concentration of (a) 5 M and (b) 6 M.

xide concentrations of 5 M and 6 M with liquid/fly ash ratio of 0.4 produced the proper alkali-activation effect for the low-calcium fly ash.

Fig. 2(a) and (b) shows the effect of curing temperature and time on compressive strength development of specimens prepared from alkali-activated fly ash with sodium hydroxide concentrations of 5 M and 6 M at liquid/fly ash ratio of 0.4, in which proper activation conditions determined from the above experiments results were used. In all curing temperature and time, the compressive strength of specimens prepared from 5 M solution, as shown in Fig. 2(a), was quite similar or slightly lower than those at 6 M solution, as showed in Fig. 2(b). In this activation, the curing temperature plays an important role for strength development. At the low temperature of 20 °C, alkali-activity reaction is very low, resulting in producing very low 7-day compressive strength. It should be noted that a negligible compressive strength was observed in the study of Katz, 1998, when alkali-activated fly ash was cured at 20 °C for the same period of time.

In Fig. 2(a) and (b), much higher compressive strength was observed at a higher curing temperature (50 °C), which indicates that a higher alkali-activity reaction of fly ash occurred at this tempera-

ture. The 7, 14, and 28-day compressive strength of specimens prepared from 5 M NaOH solution at 50 °C was 152, 219, and 263 kgf/cm², while those from 6 M solution were 184, 225, and 267 kgf/cm², respectively. At 80 °C, the compressive strength was slightly lower than those treated at 50 °C. From the factors considered on strength development, we identify that activator concentration, liquid/fly ash ratio, and curing temperature are always significative factors at 7, 14, and 28 days.

Table 3. Porosities and mean pore diameters of specimens prepared from alkali-activated fly ash with liquid/fly ash ratio of 0.4 at 50 °C: (a) different activator solution concentration at 7 day and (b) different curing time at 5 M solution

Activations	(a) 7 day			(b) 5 M		
	2 M	4 M	6 M	7 day	14 day	28 day
Items	0.028	0.022	0.021	0.023	0.024	0.028
Mean pore diameter (μm)	34.04	32.76	31.21	32.08	25.08	22.60

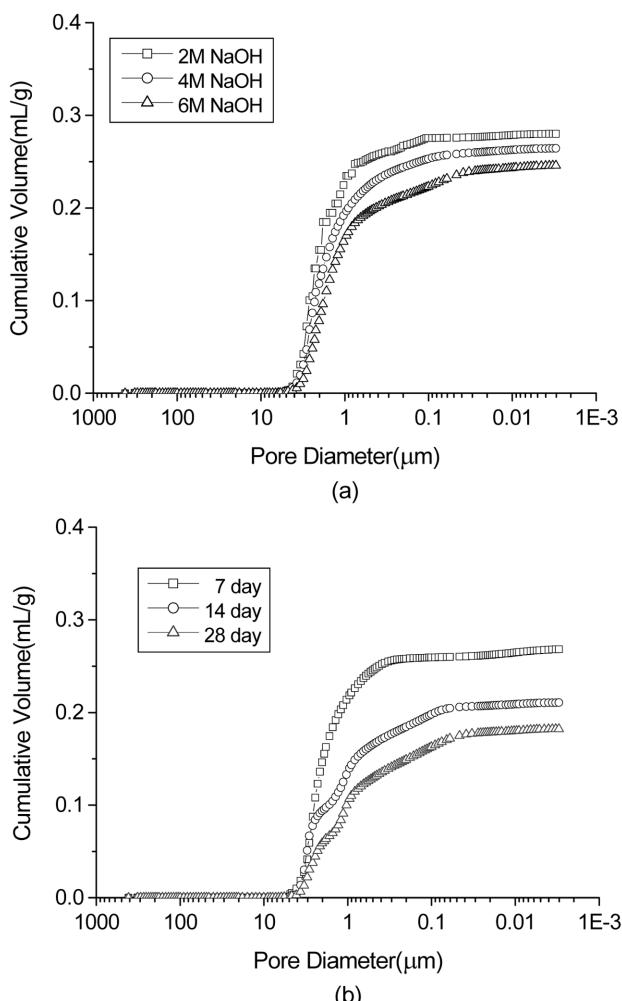


Fig. 3. Pore-size distribution curves of specimens prepared from alkali-activated fly ash with liquid/fly ash ratio of 0.4 at 50 °C: (a) different activator solution concentration at 7 day and (b) different curing time at 5 M solution.

2. Porosity and Pore Size Distribution

The pore-size distribution and accumulated porosity of several specimens were investigated by mercury intrusion porosimetry by using a Micromeritics AutoPore IV 9510. For porosity analysis, cylinder specimens about 0.8 mm diameter and 1 cm height were first degassed under vacuum. The porosities and pore-size distribution of 7-day alkali-activated fly ash with 2 M, 4 M and 6 M NaOH solution (8, 76 and 184 kgf/cm², respectively) and 7, 14 and 28-day alkali-activated fly ash with 5 M NaOH solution (152, 219 and 263 kgf/cm², respectively) at 50 °C and liquid/fly ash ratio of 0.4 were measured, and the results are shown in Table 3 and Fig. 3(a), (b).

As shown in Fig. 3 and Table 3, the specimen prepared from alkali-activated fly ash with 2 M NaOH, which has the lowest compressive strength in this series of experiments, has the highest cumulative pore volume with a diameter ranging from 0.01 to 5 μm and porosities. The porosities of specimens decreased with the higher sodium hydroxide concentration and prolonged curing time, which indicates higher denser pore refinement of the microstructure [Lorenzo et al., 2002] and, consequently, the compressive strength increased. The lowest values of both porosity and cumulative pore volume of the 28-day specimen from alkali-activated fly ash with 5 M NaOH indicated higher reactivity of this fly ash compared with other specimens, and result in the highest compressive strength (263 kgf/cm²). The mean pore diameter of specimens prepared from alkali-activated fly ash with different activator concentration and time was almost the same size with a diameter ranging from 0.021 to 0.028 μm.

3. Scanning Electron Microscopy

The morphological features of the unreacted fly ash and alkali-activated fly ash with the different sodium hydroxide concentration and temperature are showed in Figs. 4 through 6. The change in microstructural features caused by alkali-activation with the different sodium hydroxide concentration were clearly observed by SEM analysis.

It is clearly noticed that the micrograph of the raw fly ash (Fig. 4(a)) from power plants retained its round and smooth spherical shape of fly ash particles, and some of them are clusters of small particles. The fly ash particles activated by 2 M NaOH at 50 °C (Fig. 4(b)) and 5 M NaOH at 20 °C (Fig. 4(c)) were retained their round and smooth spherical shape, which suggests that large amounts of fly ash were unreacted. The unreacted fly ash in Fig. 4(b) was due to the low NaOH concentration in which the OH⁻ concentration was not high enough to react with the fly ash, while the unreacted fly ash in Fig. 4(c) was due to the low curing temperature which was not high enough to react with the fly ash.

SEM analysis of fly ash hydrated in the 3 M NaOH at 50 °C showed morphology suggesting crust of small spheres (Fig. 5(a)) and needle-like products (Fig. 5(b)) on the surface of large fly ash particles due to alkaline activation. The same type of crusts were seen in the work of Puertas and Fernández-Jiménez, 2003, and the crusts observed were believed to be alkaline aluminosilicate due to the alkaline activation of fly ash. Fig. 5(a) and (b), small spheres gels hydrated in the 3 M NaOH solution converted into needle like products due to interactions of increased gel concentration while it was cured from 7 day to 28 day.

Fig. 6(a) and (b) shows substantially different morphology of fly ash activated by 4 M and 5 M NaOH solution, respectively. The fly ash activated by the 4 M NaOH solution showed spots of erosion

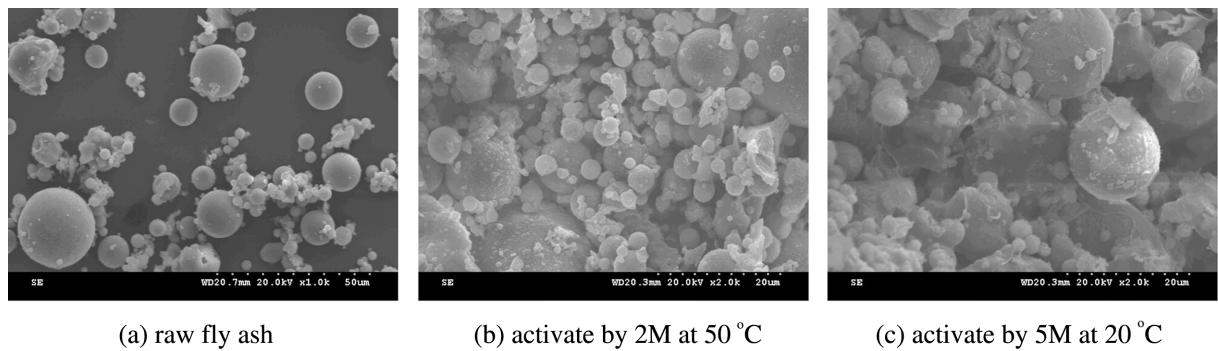


Fig. 4. SEM micrograph of (a) raw fly ash, (b) fly ash activated by 2 M NaOH at 50 °C and (c) fly ash activated by 5 M NaOH at 20 °C with liquid/fly ash ratio of 0.4 for 7 day.

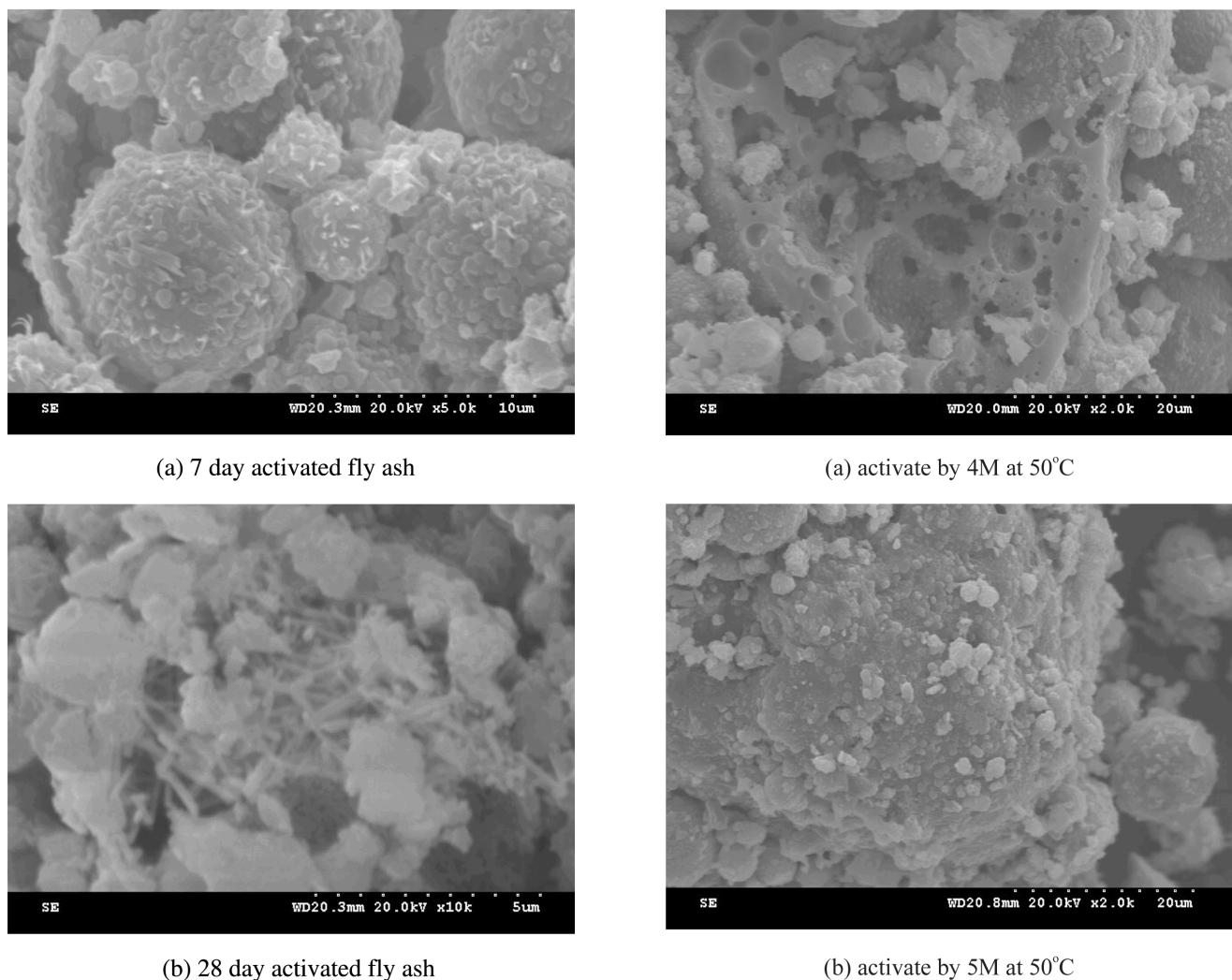


Fig. 5. SEM micrograph of (a) 7 day ($\times 5,000$ magnification) and (b) 28 day ($\times 10,000$ magnification) of fly ash activated by 3 M NaOH at 50 °C with liquid/fly ash ratio of 0.4.

on the spherical surface of the fly ash particles as can be Fig. 6(a). The surface erosion of the fly ash particles provides more reactive surface [Arjunan et al., 2001] and exposing the mullite crystals [Wesche, 1991], resulting in more aluminosilicate solid dissolution. As a result,

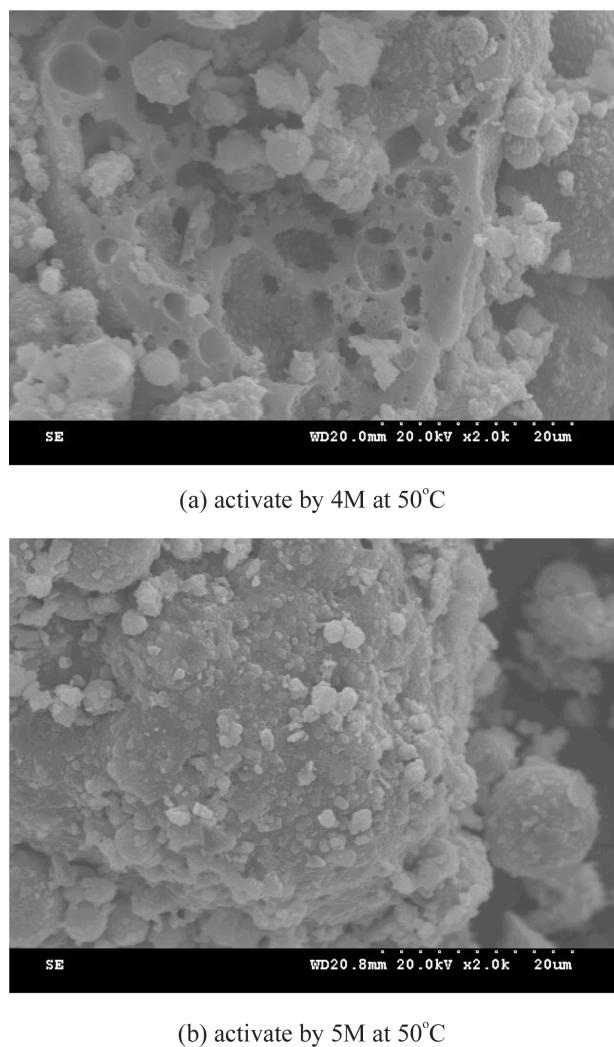


Fig. 6. SEM micrograph of (a) fly ash activated by 4 M NaOH and (b) fly ash activated by 5 M NaOH at 50 °C with liquid/fly ash ratio of 0.4 for 7 day.

a series of polysialation, coagulation, colloidal formation, gelation and subsequent gel restructuring took place and lead to the final reaction product [Davidovits, 1994; Van Jaarsveld, 1997]. Puertas and Fernández-Jiménez [2003] determined that this reaction product cor-

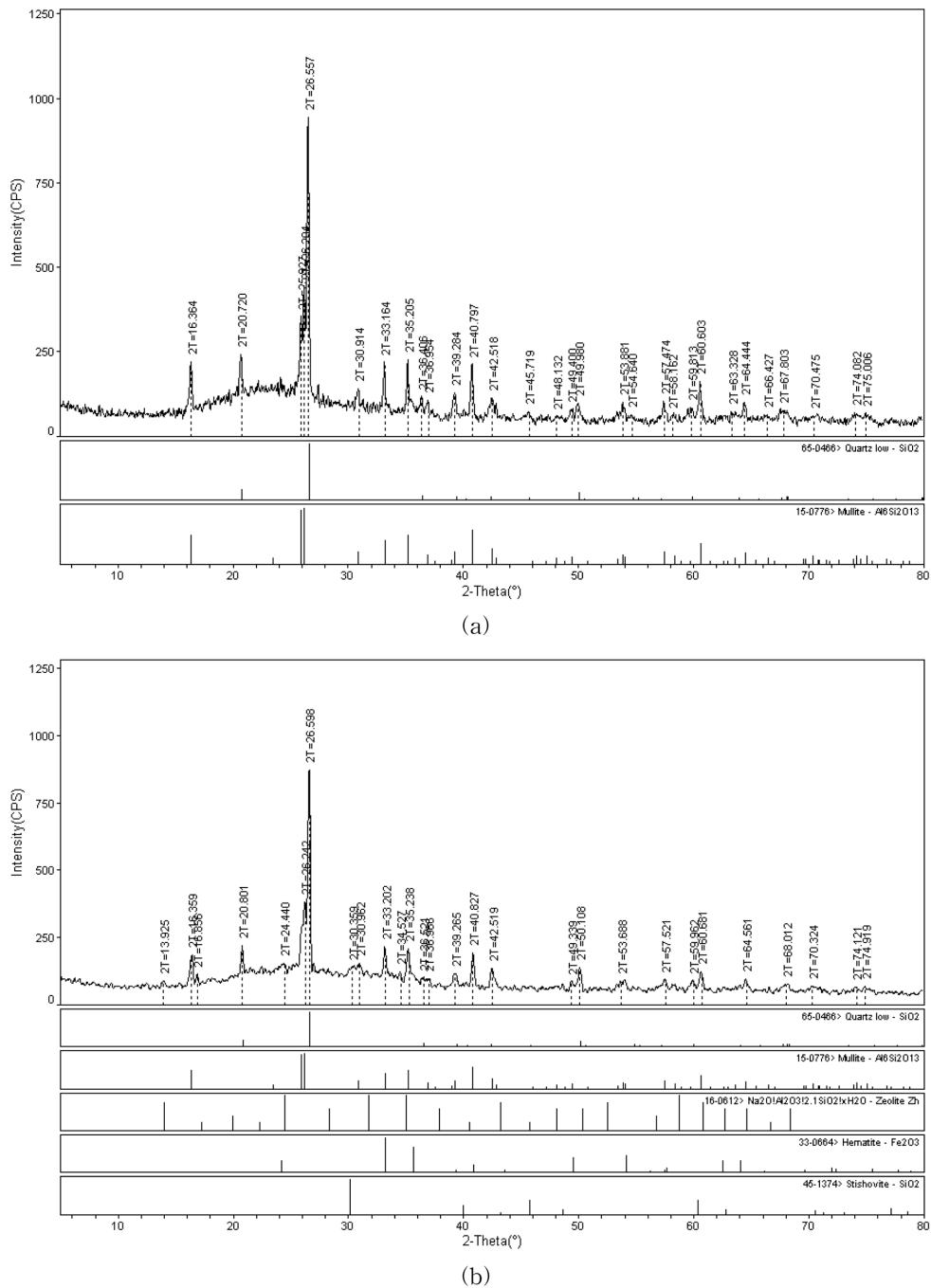


Fig. 7. X-ray diffraction data showing the phases present in a unreacted fly ash (a) and alkali-activated fly ash by the 5 M NaOH solution at 50 °C for 28 days (b).

responds to an amorphous alkaline aluminosilicate with a 3D structure as a zeolitic gel type. In Fig. 6(b), the fly ash activated by the 5 M NaOH solution shows a more continuous matrix due to subsequent gel restructuring and lead to reaction products with more solid and non porous.

4. XRD Analysis of Reaction Products

Fig. 7(a) and (b) show the XRD patterns for the unreacted fly ash and alkali-activated fly ash by the 5 M NaOH solution at 50 °C for 28 day, respectively. In Fig. 7(a), XRD patterns show that the crystalline components of unreacted fly ash mainly consist of two major phases, mullite ($3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) and quartz (SiO_2). In Fig. 7(b),

XRD diffraction patterns show that alkali-activated fly ash consists of quartz and mullite as the major crystalline phase, and hematite (Fe_2O_3) as the minor crystalline phases. The hydro-aluminosilicates of zeolite formula ($\text{Na}_2\text{O}\cdot x\text{Al}_2\text{O}_3\cdot y\text{SiO}_2\cdot n\text{H}_2\text{O}$) and stishovite (SiO_2) were reaction products of alkaline activation of fly ash. It was known that stishovite was generally synthesized from restructuring of amorphous silica at higher reaction pressure and temperature.

CONCLUSION

The activator concentration, liquid/fly ash ratio, and temperature

were always significative factors on the strength development of alkali-activated fly ash-cements. The sodium hydroxide concentration between 5.0 and 6.0 M, and liquid/fly ash ratio of 0.4 at 50 °C produces the best alkali activation effect for the low calcium fly ash, and results in higher strength. SEM observations showed that no reaction took place at lower sodium hydroxide concentration and room temperature, while higher reactivity took place at higher solution concentration and temperature. Fly ash activated by the 5 M NaOH solution at 50 °C shows a more continuous matrix with solid and nonporous due to subsequent gel restructuring by amorphous alkaline aluminosilicate produced from alkali-activated fly ash. The amorphous aluminosilicate produced from alkali-activation of fly ash bound the particles of fly ash and resulted in a decrease of cumulative pore volume and porosities of the specimens. Consequently, this is one of the major causes for the enhancement of the compressive strength of specimens prepared from fly ash with alkali-activation.

ACKNOWLEDGMENT

This work was supported by grant No. R01-2004-000-10480-0 from the Basic Research Program of the Korea Science & Engineering Foundation.

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