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Compressive strength and microstructural characteristics of class C fly ash geopolymer

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ABSTRACT

Geopolymers prepared from a class C fly ash (CFA) and a mixed alkali activator of sodium hydroxide and sodium silicate solution were investigated. A high compressive strength was obtained when the modulus of the activator viz., molar ratio of SiO_2/Na_2O was 1.5, and the proper content of this activator as evaluated by the mass proportion of Na_2O to CFA was 10%. The compressive strength of these samples was 63.4 MPa when they were cured at 75 °C for 8 h followed by curing at 23 °C for 28 d. In FTIR spectroscopy, the main peaks at 1036 and 1400 cm^{-1} have been attributed to asymmetric stretching of Al–O/Si–O bonds, while those at 747 cm^{-1} are due to the Si–O–Si/Si–O–Al bending band. The main geopolymeric gel and calcium silicate hydrate (C–S–H) gel co-exist and bond some remaining unreacted CFA spheres as observed in XRD and SEM–EXDA. The presence of gismondine (zeolite) was also observed in the XRD pattern.

1. Introduction

Manufacturing of Portland cement is a resource exhausting, energy intensive process that releases large amounts of the green house gas CO_2 into the atmosphere. Production of 1 ton of Portland cement requires about 2.8 tons of raw materials, including fuel and other materials. As a result of de-carbonation of lime, manufacturing of 1 ton of cement generates about 1 ton of green house gas [1]. At present, efforts have been made to promote the use of pozzolans to partially replace Portland cement. Recently, another class of cementitious materials, produced from an alumino-silicate precursor activated in a high alkali solution, has been developed. This cementitious material is termed geopolymer. The mortar and concrete made from geopolymers possess similar mechanical performance and appearance properties to those from Portland cement. It is also well known that geopolymers have excellent performance with respect to fire resistance, acid resistance, and stabilization/solidification of heavy metals wastes, etc. [2].

The precursors for geopolymers are developed from various sources including the natural resource kaolin and industrial wastes or byproducts, such as fly ash and ground granulated blast furnace slag (GGBFS). Thus far, the work on fly ash geopolymer has been based on a precursor derived from class F fly ash (FFA) produced by burning bituminous coals [3–6]. Recently, the increased use of lignite and subbituminous coals has substantially increased the available quantities of class C fly ash (CFA). However, there have been very few published papers on class C fly ash geopolymer (CFAG). CFA has not been subjected to the same level of analysis in the literature as FFA [7,8].

From a compositional viewpoint, the main difference between CFA and FFA is the calcium content, while both typically contain substantial amounts of silica and alumina. Compositionally, CFA can be viewed as resting somewhere in between FFA and GGBFS [9]. The fact that the mixes of FFA and GGBFS are often preferred in the production of geopolymer also provides an indication of the potential of CFA for producing geopolymer. The technology, for using a CFA in producing geopolymer would be beneficial to the understanding and to the future applications of this byproduct.

2. Experimental procedures

2.1. Materials

A CFA used in this study came from the First Energy Corporation in Ohio, USA. It conforms to the requirements of a class C mineral admixture as defined by ASTM specifications for fly ash and raw or calcined natural pozzolan for use as a mineral admixture in Portland cement concrete (C618-80). The chemical analysis of this fly ash is given in Table 1. Sodium hydroxide (99.2% NaOH) and sodium silicate (9.1% Na_2O, 29.2% SiO_2, and 61.7% H_2O) were acquired from the Fisher Scientific Company.

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Table 1
Chemical composition of a class C fly ash (CFA).

	Chemical composition of a CFA									
	SiO_2	$A1_2O_3$	Fe_2O_3	MgO	CaO	SO_3	K_2O	Na_2O	LOI	Total
Percentage (%)	38.0	19.0	9.0	5.0	20.0	3.0	0.4	1.0	3.5	98.9

2.2. Experimental design and sample preparations

Several series of samples were prepared to test the influential variables on the compressive strength of hardened geopolymer. The variables include modulus and content of the mixed alkali activator and the sample curing conditions.

All samples were made from CFA. The mixed alkali activator of sodium silicate solution and sodium hydroxide was used. The modulus of the mixed alkali activator was investigated at 1.0, 1.5, and 2.0 M ratios of SiO_2/Na_2O, respectively. The content of this mixed activator was evaluated by the mass proportion of Na_2O to CFA (5–15%). For all samples, the mass ratio of water to CFA was 0.4. The water value includes both the water in the mixed alkali activator and the extra distilled water added to CFA. The water content of the mixed alkali activator was changed with its modulus. Additionally, the content of this mixed activator was also changed from 5% to 15%. Thus, the extra distilled water was to adjust all samples to obtain the same ratio of water to CFA. Details of mixture ratios are shown in Table 2. The mixing was done in an air-conditioned room at approximately 23 °C. Directly after mixing, the fresh paste was poured in the 20 × 20 × 20 mm cubic moulds. The samples and moulds were covered with a vinyl sheet to prevent moisture loss and the carbonation of the surface. One batch of these samples was placed in an air-conditioned room at 23 °C for 3, 7, and 28 d. The other batch was put in the oven at the elevated temperatures of 60–90 °C for 4, 8, and 24 h.

The compressive strength results of CFAG were measured on the cubic samples of dimension 20 × 20 × 20 mm. Values are the averages of four separate tests. Data that deviated more than 10% were eliminated. Also the microstructural characteristics of CFAG, which was made at the optimum condition and had a high compressive strength, was analyzed using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope–energy dispersive X-ray analysis (SEM–EDXA). The hardened geopolymer samples were crushed, ground, and passed through a 325 mesh screen. The resulting powder samples were analyzed directly by the above-mentioned XRD method using CuKa radiation and a Philips PW 1316/90 diffractometer equipped with a diffracted-beam monochromator and a 1° divergence slit. For FTIR analysis, the powder samples were mixed with KBr at a concentration of 0.2–1.0 wt.% to make the KBr disks. Then the disks were tested using a Perkin Elmer FTIR microscope. For SEM–EDXA, the crushed geopolymer samples were mounted on Al-stubs with double-sided carbon tabs and then coated with a thin layer of platinum in a Hummer 6.2 sputter coater. Magnification for the SEM–EDXA analysis was 3000 times and working voltage was 20 kV. The SEM–EDXA data were collected with the Hitachi S-3500N and Noran EDXA instruments.

3. Results and discussion

3.1. Compressive strength

3.1.1. Effect of modulus and content of alkali activator on compressive strength

Compressive strength of CFAG activated by the mixed alkali activator is given in Fig. 1. The modulus and content of this activator are the two crucial variables affecting the compressive strength of CFAG. The modulus is an important parameter for the mixed

Table 2
Mix ratios of class C fly ash geopolymer (CFAG).

Samples[a] (%)	CFA (g)	The mixed alkali activator of sodium silicate and sodium hydroxide		Water to CFA ratio [d]
		Modulus[b]	Content[c]	
CFA-M1.0-C6	350	1.0	6	0.4
CFA-M1.0-C7	350	1.0	7	0.4
CFA-M1.0-C8	350	1.0	8	0.4
CFA-M1.0-C9	350	1.0	9	0.4
CFA-M1.0-C10	350	1.0	10	0.4
CFA-M1.0-C15	350	1.0	15	0.4
CFA-M1.5-C6	350	1.5	6	0.4
CFA-M1.5-C7	350	1.5	7	0.4
CFA-M1.5-C8	350	1.5	8	0.4
CFA-M1.5-C9	350	1.5	9	0.4
CFA-M1.5-C10	350	1.5	10	0.4
CFA-M1.5-C15	350	1.5	15	0.4
CFA-M2.0-C6	350	2.0	6	0.4
CFA-M2.0-C7	350	2.0	7	0.4
CFA-M2.0-C8	350	2.0	8	0.4
CFA-M2.0-C9	350	2.0	9	0.4
CFA-M2.0-C10	350	2.0	10	0.4
CFA-M2.0-C15	350	2.0	15	0.4

[a] In the samples codes: CFA is a class C fly ash, *M* and the following number is the modulus of the mixed alkali activator, and *C* and the following number is the content of this activator.

[b] The modulus is the molar ratio of SiO_2/Na_2O in the mixed alkali activator.

[c] The content of this activator is evaluated by the proportion of Na_2O to CFA.

[d] All geopolymers were made with a mass ratio of water to CFA of 0.4. This water includes both the water in the mixed alkali activator and the extra distilled water added to CFA.

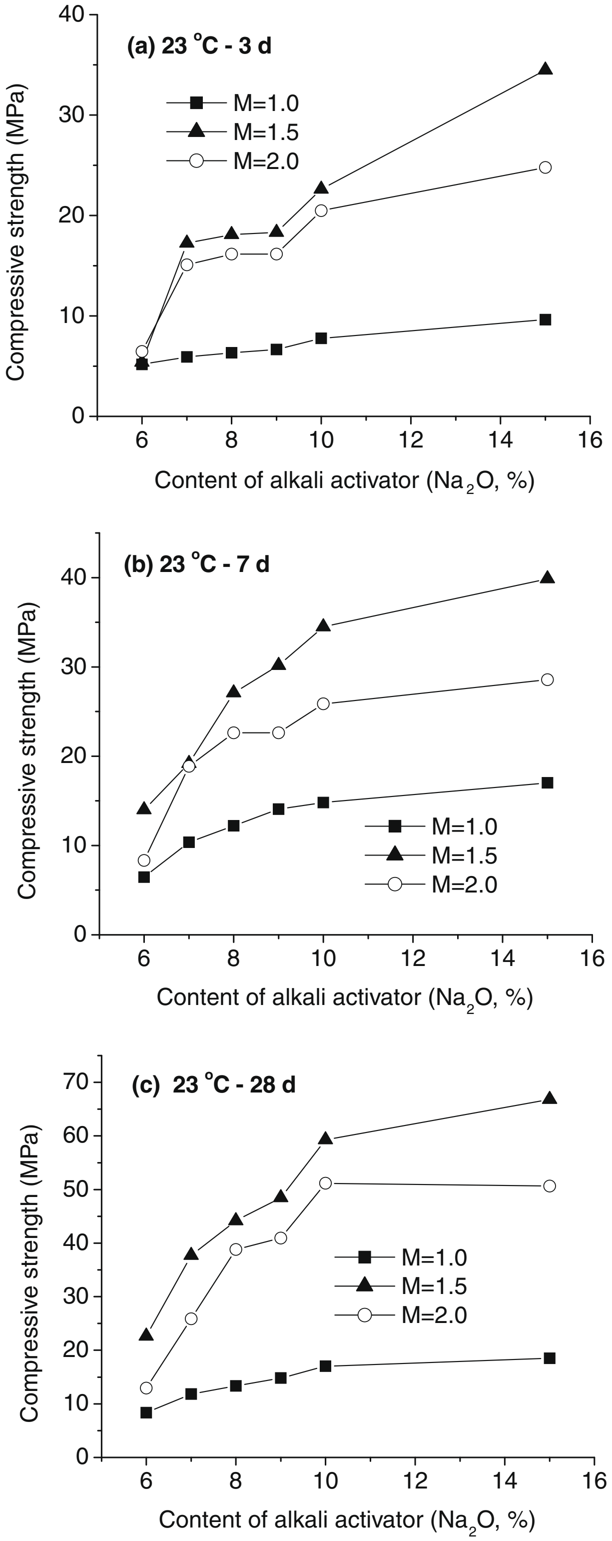

Fig. 1. Effects of modulus (M) and content of the mixed alkali activator on the compressive strength of the class C fly ash geopolymer (CFAG) cured at room temperature of 23 °C for 3, 7, and 28 d. The modulus of the activator is the molar ratio of SiO_2/Na_2O. The content of this activator was evaluated by the proportion of Na_2O to class C fly ash (CFA).

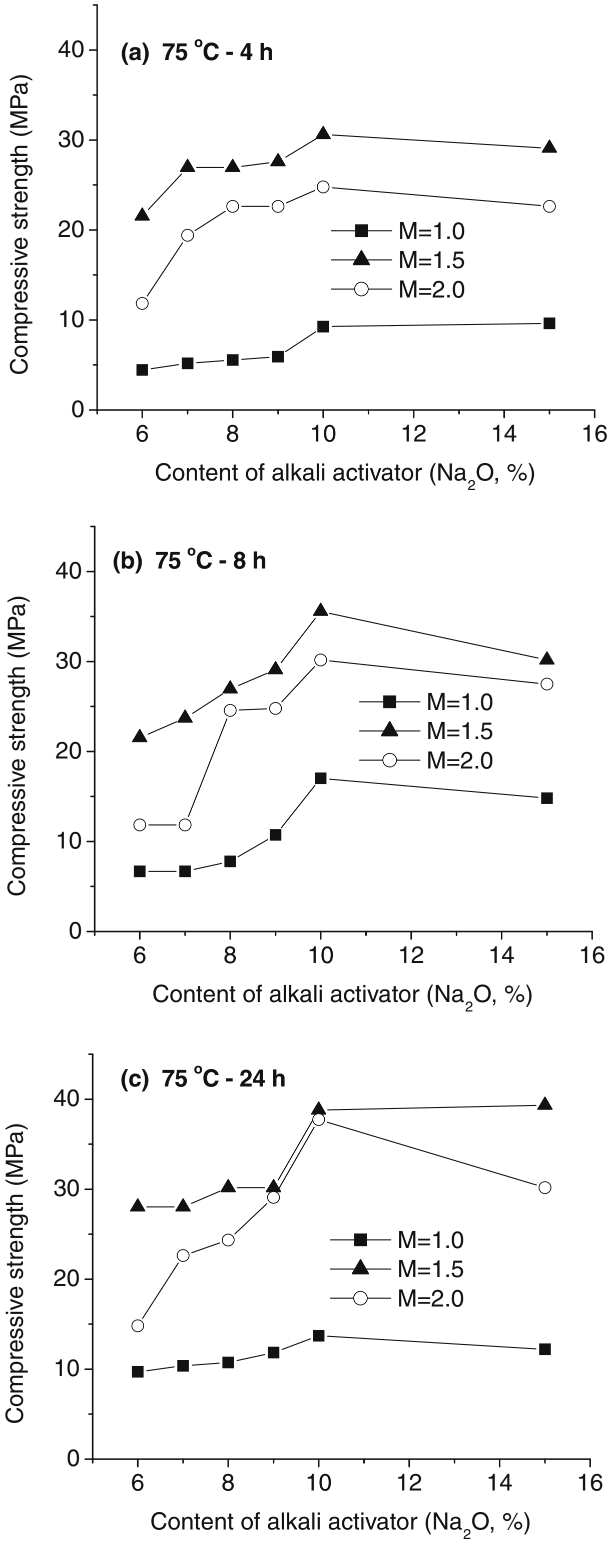

Fig. 2. Compressive strength of the class C fly ash geopolymer (CFAG) cured at room an elevated temperature of 75 °C for 4, 8, and 24 h. M is the modulus of the mixed alkali activator viz., molar ratio of SiO_2/Na_2O. The content of this activator was evaluated by the proportion of Na_2O to class C fly ash (CFA).

activator and it also presents the mixture ratio of sodium hydroxide and sodium silicate solution. In the geopolymerization process, strong alkalis are required to activate the silicon and aluminum present in the fly ash to allow the glassy structure to partially or totally dissolve and then transform into a very compacted composite. Alkali concentration is the most significant factor for geopolymerization. Thus, the content of the mixed activator is another critical parameter.

Table 3
Compressive strength of the class C fly ash geopolymer (CFAG) curing at elevated temperatures of 60–90 °C.

Sample[a]	Curing temperature (°C)	Compressive strength (MPa)		
		4 h	8 h	24 h
CFA-M1.5-C10%	60	14.4	20.7	20.7
	70	15.6	20.0	20.7
	75	30.6	35.6	38.8
	80	15.6	20.0	25.6
	90	20.0	28.1	29.6

[a] Sample, sample code of CFA-M1.5-C10%, see in Table 2.

For CFAG cured at 23 °C for 3, 7 and 28 d, the compressive strengths indicate that the modulus of the mixed alkali activator plays an important role in the development of the system's compressive strength (Fig. 1). The highest compressive strength is obtained when the modulus is 1.5.

The content of the mixed alkali activator also plays an important role in determining compressive strength of CFAG. The content in this work ranged between 6% and 15% in mass (Table 2). With increasing alkali concentration, the compressive strength can also be improved upon because the solubility of alumino-silicate increases with increasing Na_2O concentration. Higher concentrations of Na_2O yielded higher compressive strength. However, the increase of compressive strength is no longer evident when the concentration of Na_2O is higher than 10% (Fig. 1).

Therefore, high compressive strength of CFAG was obtained when the modulus of the activator was 1.5, and the content of this activator was 10% (sample CFA-M1.5-C10% in Table 2). When these samples were cured at 23 °C for 3, 7, and 28 d, the compressive strength values were 22.6, 34.5, and 59.3 MPa, respectively.

3.1.2. CFAG cured at elevated temperatures

In order to study the effects of curing temperatures on the compressive strength of CFAG, these matrices are usually subjected to elevated curing temperatures. Most research has been conducted with curing temperatures ranging from 30 to 85 °C [3,10,11]. According to research, curing times may vary from several hours to several days.

Compressive strength of CFAG samples cured at elevated temperature of 75 °C for 4, 8, and 24 h (Fig. 2) also confirms that the optimum modulus of the mixed alkali activator was 1.5, and the content of the activator was 10%, viz., the mix ratio of sample CFA-M1.5-C10% in Table 2. Further, the compressive strength of the sample CFA-M1.5-C10% cured at elevated temperatures of 60–90 °C is given in Table 3. Fig. 2 and Table 3 show that the optimum curing condition for CFAG is at 75 °C. Curing temperature had a significant effect on the compressive strength development because it affects CFAG setting and hardening. Synthesized products are known to be very sensitive to experimental conditions [12]. However, compressive strength begins to decrease after curing for a certain period of time at higher temperature. Prolonged curing at higher temperatures can break down the granular structure of geopolymer mixture. This results in dehydration and excessive shrinkage due to contraction of the gel, which does not transform into a more semi-crystalline form [13]. After curing at 75 °C for 4, 8, and 24 h, compressive strengths of these samples were 30.6, 35.6, and 38.8 MPa, respectively.

3.2. Microstructural analysis

The CFAG, sample CFA-M1.5-C10% in Table 2, obtained a high compressive strength of 63.4 MPa when it was cured at 75 °C for 8 h followed by curing at 23 °C for 28 d. Its microstructural characteristics were further studied using XRD, SEM–EDXA and FTIR.

3.2.1. XRD

The XRD patterns of the raw material of CFA (Fig. 3a) and the geopolymer of CFAG (Fig. 3b and c) were obtained. The main mineral components of CFA were mullite, quartz, anhydrite, and f-CaO. In the XRD pattern of CFAG, a large part of the structure was of amorphous content. The degree of disorder can be interpreted by the way it diffracts X-rays to form a pattern. In a non-crystalline

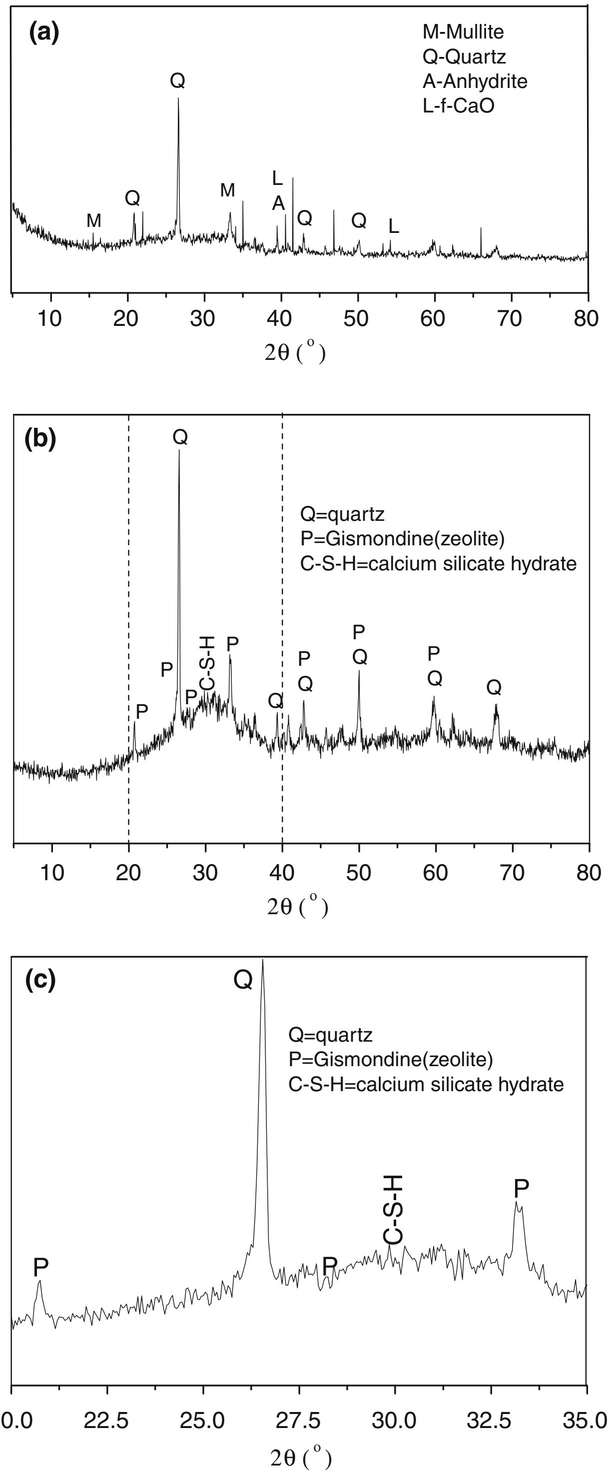

Fig. 3. X-ray diffraction (XRD) of the class C fly ash (CFA) (graph (a)), and the CFA geopolymer (CFAG) cured at 75 °C for 8 h followed by curing at 23 °C for 28 d (graphs (b) and (c)). 2θ is 5–80° in graph (a) and (b) and 20–35° in graph (c).

state, diffraction of X-rays resulted in a broad diffuse halo rather than sharp diffraction peaks. The broad and amorphous hump from 20° to 40° (2θ) was the characteristic peak of amorphous gels including geopolymeric gels and calcium silicate hydrate (C–S–H) gels (Fig. 3b and c). It indicated that the geopolymeric reaction and the hydrate reaction occurred at the same time within the single system of CFAG.

Additionally, peaks of a zeolite of gismondine were also discovered in the XRD pattern of CFAG. Peaks due to the crystalline components of quartz from the unactivated fly ash have been determined.

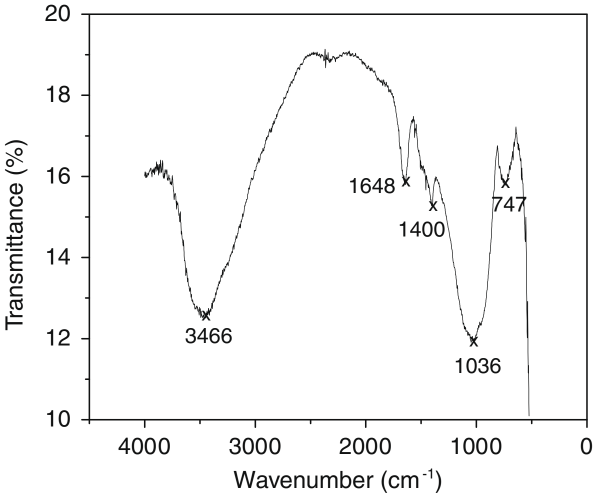

Fig. 4. Fourier transform infrared spectroscopy (FTIR) of the class C fly ash geopolymer (CFAG) cured at 75 °C for 8 h followed by curing at 23 °C for 28 d.

3.2.2. FTIR

FTIR absorption spectroscopy is well known for its sensitivity in characterizing materials with short-range structural order, and has been useful for characterizing CFAG. It shows the main adsorption bands of CFAG as follows: 747, 1036, 1400, 1648, and 3466 cm^{-1} (Fig. 4). The peak at 1036 cm^{-1} and the relatively weak peak at 1400 cm^{-1} are attributed to asymmetric stretching vibrations of Al–O/Si–O bonds, while the Si–O–Si/Si–O–Al bending band can be seen at 747 cm^{-1}. These bands are common in ring silicates and provide an indication of the degree of amorphisation of the material, since its intensity does not depend on the degree of crystallization [4]. The relationship between Al–O, Si–O, Si–O–Si, or Si–O–Al asymmetric stretch peak positions and the extent of the geopolymerzation process is complex, but helpful towards the study of the mechanism of geopolymerization. The broad bands in the region of 1648–3466 cm^{-1} characterized the spectrum of stretching and deformation vibrations of OH and H–O–H groups from the weakly bound water molecules which were adsorbed on the surface or trapped in the large cavities between the rings of geopolymeric products [14].

3.2.3. SEM–EDXA

The SEM images presented in this paper depict the characteristic morphology of the original CFA, the reactive CFA sphere, and the reaction products of CFAG. The original CFA consists of a series of spherical vitreous particles of different sizes (Fig. 5a). These particles are usually hollow, and some spheres may contain other particles of a smaller size in their interiors [15]. Fig. 5b shows the morphology changes of a reactive CFA sphere as a consequence of the alkali activated dissolution and mild thermal curing. The sphere seems to be broken in the high alkaline condition and part of the alumino-silicate dissolves from the fly ash. Further, the inner area of the broken fly ash seems to be filled with a large quantity of micro particles of the reaction products (Fig. 5c).

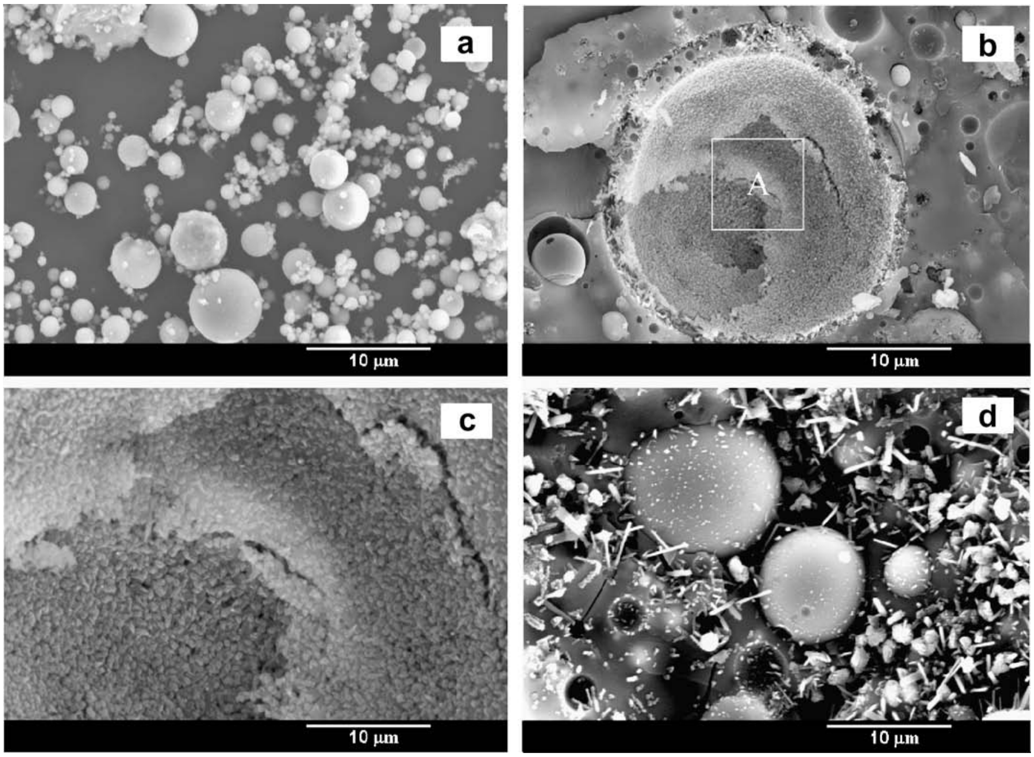

Fig. 5. Scanning electron microscope (SEM) of (a) the original class C fly ash (CFA), (b) the reactive CFA sphere, (c) the reactive "A" area of CFA sphere in graph (b), and (d) CFA geopolymer (CFAG) cured at 75 °C for 8 h followed by curing at 23 °C for 28 d.

Table 4
The most common types of geopolymers [16,17].

Types of species	Abbreviations
1. Poly(sialate): R_n–(–Si–O–Al–O–)$_n$–	R-PS
2. Poly(sialate-siloxo-): R_n–(–Si–O–Al–O–Si–O–)$_n$–	R-PSS
3. Poly(sialate-disiloxo-): R_n–(–Si–O–Al–O–Si–O–Si–O–)$_n$–	R-PSDS

Table 5
Atom percentages and ratios of the original class C fly ash (CFA), the reactive CFA sphere, and the class C fly ash geopolymer (CFAG) using energy dispersive X-ray analysis (EDXA).

Atom percentages and ratios	The original CFA (Fig. 5a)	The reactive CFA sphere (Fig. 5b)	CFAG (Fig. 5d)
Na, at.%	0.91	2.00	6.23
Al, at.%	9.93	17.9	6.63
Si, at.%	17.9	9.22	20.9
Ca, at.%	10.1	1.17	6.91
Na/Al	0.09	0.11	0.94
Si/Al	1.80	0.52	3.15
Ca/Al	1.02	0.07	1.04

Generally, the three-dimensional amorphous geopolymeric network with a general formula of R_n–[[–Si–O_2]$_z$–Al–O]$_n$·$w$$H_2O$ (R represents a cation such as sodium, potassium, or calcium; n is the degree of polycondensation; $z = 1$, 2, or 3; and w is the amount of binding water) is often argued to be the phase that gives the binding property to the geopolymeric gel. The most common types of geopolymers are listed in Table 4 [16,17]. The ratio of Si/Al should be 1–3, and the ratio of Na/Al should be 1. During the reaction process of this work, Si/Al ratios in the original CFA, the reactive CFA sphere, and the reaction products of CFAG are changing (data in Table 5, the quantities of Na, Al, Si, and Ca were obtained using SEM–EDXA, and then we calculated the ratios of Na/Al, Si/Al, and Ca/Al). The Si/Al ratio in the original CFA is 1.70, however, it changes to 0.51 in the reactive CFA sphere. This happens either because the dissolution speed of silicate is faster than that of the aluminum or the geopolymerization speed of aluminum is faster than that of silicate. Table 5 shows the EDXA data of CFAG. The ratio of Na/Al in this system was 0.94 and near to 1, and the Si/Al ratio was 3.15. Thus, the main geopolymeric gel was inferred to be (Na)-poly(sialate-disiloxo-), i.e. Na_n–(–Si–O–Al–O–Si–O–Si–O–)$_n$–. Additionally, in Fig. 5d, geopolymeric gel, the main reaction products of CFAG were observed co-existing with calcium silicate hydrate (C–S–H) gel and some remaining unreacted spheres. On the other hand, the Na/Al ratio in this system was slightly less than one, which indicates that only sodium as the cation is insufficient. In effect, a partial calcium participant in geopolymerization is needed to obtain charge balance. Furthermore, the excess silicate and calcium may participate in the formation of calcium silicate hydrate (C–S–H) gel. This was in agreement with result of the XRD pattern.

4. Conclusion

In this paper, compressive strength and micro structural characteristics of a class C fly ash geopolymer (CFAG) were studied.

A high compressive strength was obtained when the class C fly ash (CFA) was activated by the mixed alkali activator (sodium hydroxide and sodium silicate solution) with the optimum modulus viz., molar ratio of SiO_2/Na_2O of 1.5. The proper content of this mixed activator was 10% as evaluated by the mass proportion of Na_2O to CFA. The compressive strength of these samples was 63.4 MPa when they were cured at 75 °C for 8 h followed by curing at 23 °C for 28 d.

The main FTIR peaks of CFAG at 1036 and 1400 cm^{-1} have been attributed to asymmetric stretching of Al–O/Si–O bonds, and the Si–O–Si/Si–O–Al bending band can be seen at 747 cm^{-1}. The original CFA consists of a series of spherical vitreous particles of different sizes. When CFA is alkali activated the sphere seems to be attacked and broken due to the dissolution of alumino-silicate in the high pH alkali solution. Hereafter, geopolymeric gel (Na)-poly(sialate-disiloxo-), i.e. Na_n–(–Si–O–Al–O–Si–O–Si–O–)$_n$– is formed and observed co-existing with calcium silicate hydrate (C–S–H) gel and some remaining unreacted spheres. Peaks of a zeolite of gismondine were shown in the XRD pattern.

The increased use of lignite and subbituminous coals has substantially increased the available quantities of CFA. Utilization of this fly ash in geopolymer materials is a resource and energy saving process and it also indirectly reduces the emission of green house gas CO_2 released from cement manufacturing. This is beneficial for resource conservation and environmental protection.

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