



## Full Length Article

## Influence of graphene oxide in a chemically activated fly ash

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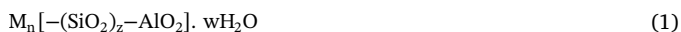
## ABSTRACT

To divert fly ash from hazardous waste stream to beneficial uses, this work aims to improve the solidification of fly ash as a geopolymer material by using graphene oxide (GO). The hydration precursors, morphology, elemental composition, mineralogy, chemical structure and ordering of GO-modified fly ash geopolymer were investigated by means of Raman spectroscopy, SEM/BSE, EMPA, XRD/TGA and <sup>29</sup>Si/<sup>27</sup>Al MAS-NMR, respectively, to unravel the role of GO. The experimental results suggest that GO regulated the Ca/Si, Si/Al and Ca/(Si + Al) mole ratios to facilitate the formation of fly ash hydrates with improved mechanical strength, as GO showed the ability to selectively affect the distribution of different hydration precursors. GO also promoted the formation of low quartz and jennite-like hydrates. Overall, the 28-day compressive strength of fly ash geopolymer (*w/b* = 0.35) in this study was improved by 23% (from 33.6 MPa to 41.4 MPa) with GO admixed at 0.02% by mass of fly ash. The NMR study showed that GO improved the polymerization degree of fly ash geopolymer by increasing the total Q<sup>3</sup> and Q<sup>4</sup> Si-tetrahedrons, which suggests potential for improving the immobilization of heavy metals in fly ash.

## 1. Introduction

In the context of recycling fly ash from hazardous waste stream and reducing CO<sub>2</sub> emissions, alkali-activated fly ash binder or geopolymer is being considered as one of the main treatments to solidify fly ash and as a sustainable construction material [1–5]. Palomo et al. [6] showed the great potential of alkali-activated fly ash as an alternative to cement for the future. The glassy silica and alumina contents in the fly ash were transformed by alkali activation into well-cemented composites. Some researchers [7–10] further demonstrated that certain high-reactivity fly ashes can be used as the sole binder to produce mortar or concrete with mechanical and durability performance comparable to those using Portland cement.

Davidovits described the alkali activation of metakaolin, which is similar to the alkali activation of fly ash, in a general polymeric model as follows [11]:



where M is the alkaline element; n is the degree of polymerization and z is 1, 2, or 3. Fernández-Jiménez et al. also developed a descriptive model for alkali-activated fly ash cement [3]. In these two models, the fly ash binder system involves the dissolution of aluminosilicates from

fly ash in the alkali medium. Then a 3D network of silico-aluminate structure which consists of [SiO<sub>2</sub>] unit, [AlO<sub>2</sub>]<sup>-</sup> unit, cations (Ca<sup>2+</sup>, Na<sup>+</sup> or K<sup>+</sup>) and other elements is formed by a polycondensation of reaction products.

Recent years have seen a growth in innovative research on the application of graphene oxide (GO) in construction materials [12–15]. GO shows potential as an admixture for cementitious materials because it is a two-dimensional carbon sheet with an aspect ratio up to 30,000 or higher [16]. It features a Young's modulus of ~0.3 TPa and an intrinsic strength of ~112 GPa, and is highly hydrophilic [17,18]. Studies have suggested that GO can significantly improve the overall performance of cementitious mix by regulating cement hydration and providing a crack branching and bridging mechanism, in addition to acting as nanofillers. Gong et al. [12] indicated that the addition of GO at 0.03% by weight of plain cement resulted in 13.5% lower total porosity and 27.7% fewer capillary pores in a cement paste. The compressive strength and tensile strength of the cement paste were both increased by over 40%. Lv et al. [13] observed that different amounts of GO (0.01–0.06% by weight of cement) promoted flower-like and polyhedron-like cement hydration products, which formed much more compact and ordered microstructure. As a result, the flexural strength and compressive strength of the cement paste were considerably improved by the incorporation of

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0.03% GO. Ranjbar et al. [15] found that GO absorbed more energy when under crack-bridging or pull-out and thus led to improved toughness.

Up to date, research carried out to study the GO's function in conventional cementitious binders has showed this new nanomaterial has a significant potential to become an outstanding admixture [12–15]. However, little research has been conducted to investigate the performance and compatibility of GO in an activated fly ash binder or geopolymer. The alkali-activated fly ash binder or geopolymer is distinguished from the cement by its higher initial alkalinity and less availability of  $\text{Ca}^{2+}$  ions [19]. Also the hydration products tend to form Ca(Na, K)-A-S-H (aluminosilicate hydrate) gels instead of C-(A)-S-H (aluminato-substituted calcium silicate hydrate) gels [20]. As such, the predications of GO's performance in the alkali-activated fly ash binder or geopolymer made based on the ordinary cement binder may be inappropriate.

In this respect, research is needed to obtain an enhanced understanding of the GO's reaction mechanism in the fly ash hydration system and to improve the solidification of fly ash as a geopolymer material. This work examines the possible role of GO in a chemically activated fly ash binder based on experimental results. The authors hypothesized that GO dispersed in the chemically activated fly ash binder can be employed as growth points and templates to facilitate the fly ash hydration and modify the structure of hydration products at molecular level, which in turn improves the mechanical performance of fly ash geopolymer. The following sections detail the investigation of GO's function through the fabrication, testing and microstructural characterization of a chemically activated fly ash geopolymer (without the use of strong alkali or heat).

## 2. Experimental

### 2.1. Materials

The GO (Fig. 1a) used in this study was produced from 325 mesh flake graphite following the Hummers method [21], featuring an extremely high specific surface area ( $\sim 2600 \text{ m}^2 \text{ g}^{-1}$ ) and a zeta potential of  $-30 \text{ mV}$  in neutral aqueous solution, due to the presence of negatively charged functional groups on this nanomaterial [22]. The as-produced GO was pasty, which was diluted with deionized water and then sonicated for 45 min by using a Branson digital sonifier (S-450D, 400W, 60% amplitude) to produce stable GO suspension (max. 3 g/L). The GO suspension with designed GO content was then ready for the production of GO-fly ash binder. The chemical composition of GO was investigated by energy-dispersive X-ray spectroscopy, and major elements were C = 71 wt% and O = 27 wt%. The functional groups in this GO were studied by X-ray photoelectron spectroscopy (XPS). As shown in Fig. 1b, the GO mainly contained “OH” functional groups (at 286.2 eV) and “C–C” bonds (at 284.6 eV). Major elements were also confirmed by XPS: C = 70.1 wt% and O = 28.1%.

The FTIR spectrum was also taken on the GO sample, as shown in Fig. 1(c). The strong peak at  $3436.92 \text{ cm}^{-1}$  is attributed to the O–H stretching vibration in hydroxyl [23]. The peak at  $2928.06 \text{ cm}^{-1}$  corresponds to the typical C–H stretching vibration in carboxyl [23]. The small peaks between  $1100$  and  $1400 \text{ cm}^{-1}$  indicate the typical C–O vibrations of epoxide [23]. The C=C bond vibration is shown at  $1624.35 \text{ cm}^{-1}$ . And the peak at  $1762.12 \text{ cm}^{-1}$  can be attributed to the C=O vibration [23].

The fly ash in this study was examined by X-ray fluorescence (XRF)

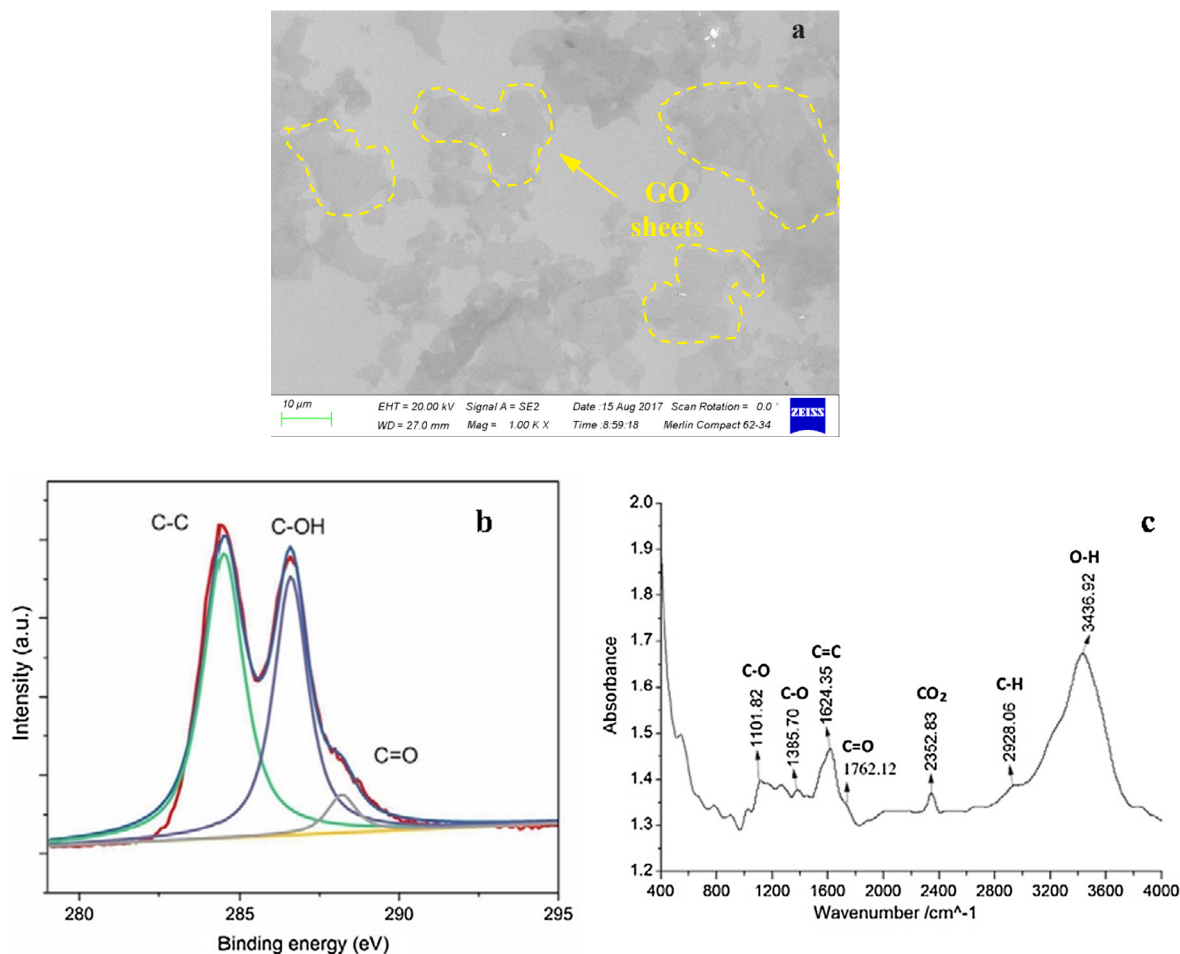


Fig. 1. (a) SEM image, (b) XPS spectra, and (c) FTIR spectra of GO used in this study.

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