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Effect of synthesis parameters on compressive strength of fly ash-slag blended geopolymer

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HIGHLIGHTS

- A comprehensive study on synthesis and characterization of geopolymers.
- An experimental investigation on influential parameters on mechanical strength.
- A correlation between microstructure and mechanical strength.
- Statistical analysis and development of a mathematical model for strength.

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ABSTRACT

This article outlines the influence of synthesis parameters on mechanical properties of geopolymer synthesized under ambient environmental conditions. Geopolymer binders are prepared from binary blended pozzolanic materials, i.e. the fly ash and slag as source material and sodium hydroxide as alkali activator. The dissolution of alumino-silicates from the source materials under different alkali condition is studied. The compressive strength of blended fly ash-slag geopolymer; synthesized under different conditions is investigated. The relevance of development in compressive strength with the amount of leached alumino-silicate ions is discussed. Further, the mineralogical, chemical and microstructural characterizations are done for the geopolymers. The test results reveal that the dissolution and compressive strength are highly affected by the alkali content and the reactive component of the source material. The reaction products, chemical bonds and microstructural development are different for different raw materials; depending upon the reactive phase present. Statistical analyses are carried out to check the significance of the factors affecting the synthesis process. Also, mathematical relationships are established for factors influencing mechanical behaviour of synthesized geopolymer.

1. Introduction

The production and consumption of conventional cement is growing at a faster rate. However, the production processes of traditional cementitious binders are energy intensive and emit a huge amount of CO_2, which constrains its use as an eco-efficient material. Also, the industrial processes generate a huge quantity of by-products which have a lethal effect on the environment, thus require effective utilization. Geopolymerization is a very favourable innovative technology that transforms unusable industrial solid wastes of alumino-silicate composition into useful products competitive with many known civil infrastructure construction materials. French materials scientist Joseph Davidovits invented the term "Geopolymer" in 1979, attributed this term to the amorphous to semi-crystalline tri-dimensional alumino-silicates that can be formed at low temperature and short time by alkali reaction with naturally occurring alumino-silicates solid materials [1]. The mechanism of this activation involves three major reactions (a) Dissolution of aluminate and silicate monomers when OH^- in the alkaline activator breaks the bond of alumino-silicates bonded with oxygen in the source material (b) Polycondensation of silica and alumina monomers to form dimers, which in turn react with another monomer to build polymer (c) Crystallization occurs by precipitation of reaction products [2]. Geopolymers are the inorganic polymers possess low density, micro/nano-porosity, negligible shrinkage, high mechanical strength, notable surface hardness, thermal stability, fire, and chemical resistance [3]. Also, they are of low cost having low energy consumption and least greenhouse gas emission (about 80–90% less compared to cement) during synthesis [4]. Due to these properties, geopolymers are considered as

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potential alternative cementitious materials which can find a place in the industrial application like construction and building materials [5,6].

Three components are essential for making of geopolymers; those are the raw materials containing reactive alumino-silicates e.g. fly ash, ground granulated blast furnace slag, metakaolin, red mud etc., inactive filler like kaolin or metakaolin as an additional source of aluminium ions and alkali reagent in the form of alkali hydroxide or alkali silicate solution [7]. Fly ash is a pozzolanic material bearing good physical, chemical, and mechanical properties which is used for cement [8]. Early high strength geopolymeric binders can be developed from geopolymeric and hydraulic cement. Incorporation of a latent hydraulic cementitious product like ground granulated blast furnace slag with geopolymer shows an improved compressive strength of the final binder [1]. The most commonly used alkalis are hydroxide or silicates of sodium and potassium. Minerals show a higher extent of dissolution in sodium hydroxide than potassium hydroxide [9,10]. Also, considering durability aspect, sodium hydroxide shows better resistance to acids in comparison to potassium hydroxide and alkali silicates [11,12]. Moreover, the low cost of sodium hydroxide over alkali silicates and potassium hydroxide encourages its use in the production of geopolymers.

Some relevant literature on the effect of synthesizing parameters on properties of geopolymers is summarised in this part. Puertas et al. [8] synthesized fly ash/slag geopolymer and concluded that the fly ash to slag ratio and the concentration of alkali activator are the most significant factors. A compressive strength of about 50 MPa is reached for a mixture of 50% fly ash and 50% slag when activated with 10 M sodium hydroxide after 28 days of curing at 25 °C. De Silva et al. [13] studied the kinetics in geopolymerization reaction. The role of Al_2O_3 and SiO_2 is studied and identified that Si^{+4} and Al^{+3} ions concentration influences the properties of geopolymer. Duxon et al. [14] specified that neither silicon nor aluminium alone is sufficient for formation of strong chemically bonded geopolymer structure. It depends on the amount of amorphous alumino-silicate compounds formed; which in turn influenced by the temperature, alkali concentration and leachable alumino-silicates present in the source material. Rattanasak and Chindaprasirt [15] found when fly ash comes into contact with an aqueous solution of sodium hydroxide; leaching of Si, Al and other minor ions occurs. The amount of leaching is dependent on the concentration of sodium hydroxide solution and leaching time. The results indicate leaching time about 5–10 min is sufficient with 10 M sodium hydroxide solution. Dimas et al. [16] observed that the solubility of materials is closely related to the amorphous alumino-silicate phase, which generally dissolves easily in sodium hydroxide solution. Silicon dissolution is always greater than aluminium, which is invariable with respect to the alkali concentration. Chindaprasirt et al. [17] synthesized high strength (86 MPa at 28 days) geopolymer with excellent stability and studied the effect of fineness of fly ash on physical and mechanical properties of high calcium fly ash geopolymer. Deb et al. [4] reported that the inclusion of ground granulated blast furnace slag upto 20% in fly ash activated with a mixture of sodium hydroxide and sodium silicate can form a geopolymeric product with excellent compressive strength comparable to ordinary Portland cement. Topark-Ngarm et al. [18] prepared high calcium geopolymer concrete by activating high calcium fly ash with a mixture of sodium silicate and sodium hydroxide solution of 15 M concentration. A compressive strength of 54.4 MPa is attained after 28 days of curing. The strength continues to increase with time similar to ordinary Portland cement concrete. Zhang et al. [19] opined that ambient curing condition is more practical for synthesizing red mud-fly ash geopolymers. The strength of geopolymer increases with increase in silicon/aluminium and sodium/aluminium ratios. Also, a prolonged curing period of upto 180 days shows a continuous increase in strength. Cihangir et al. [20] studied the effect of alkali type and their concentration and slag content on compressive strength and stability of alkali-activated slag. Higher unconfined compressive strength in short-term is obtained when activated with sodium hydroxide and with liquid sodium silicate in long-term curing. Junaid et al. [21] considered various parameters like alkaline liquid/fly ash, water/geopolymer solid, alkaline liquid/water for development of fly ash geopolymer and identified that alkali concentration, sodium silicate composition, silicate to hydroxide ratio, temperature and curing time are the important parameters.

The present work deals with the synthesis and characterization geopolymers prepared from blending of fly ash and blast-furnace slag, activated with sodium hydroxide under ambient environment conditions. The variables investigated are alkali concentration, alkali content, slag content, and curing duration. The dissolution mechanism and the strength development of geopolymers are investigated. Further, the microstructural and mineralogical studies are made on the geopolymer products. Statistical analysis is performed to quantify the significance of the influential factors and a mathematical model is developed for factors influencing mechanical behaviour of synthesized geopolymer.

2. Materials and methodology

2.1. Raw materials characterization

Class-F type fly ash (FA) and granulated blast-furnace slag (GBS) were collected from captive power plant and slag granulation plant of Rourkela Steel Plant, Odisha, India. These materials were sundried first and mixed thoroughly to bring homogeneity in the sample. These materials were kept in the oven within a temperature range of 105 °C to 110 °C for 24 h for drying and then stored separately in air tight containers for subsequent use. GBS contains mostly sand-sized particles, so it is ground in a ball mill to increase its fineness for better reactivity. Consequently, ground granulated blast-furnace slag (GGBS) is obtained. The physical properties of the source materials are listed in Table 1. Sodium hydroxide (NaOH) flakes with 98% purity were used as the alkaline activator and the same is procured from Loba Chemie Pvt. Ltd., Mumbai, India. The NaOH solutions with desired concentration expressed in molarity (M) were prepared before 24 h of its use to ensure proper dissolution and to completely dissipate the heat of hydration.

The chemical compositions of raw materials were analysed by X-ray fluorescence (XRF) spectroscopy and summarized in Table 2. The amount of Al_2O_3+SiO_2+ Fe_2O_3, SO_3 and CaO in the fly ash sample are 91.353%, 0.903% and 2.043% respectively, which indicates that the fly ash is class-F type as described in ASTM C618-08a [22]. The major constituents of slag are dioxide of silicon, aluminium and calcium. The Scanning Electron Microscopic (SEM) images (Fig. 1) shows that most of the fly ash particles are spherical with few irregular structures and the slag particles are rough and angular shaped. The X-ray diffraction (XRD) patterns (Fig. 2) indicates that fly ash contains quartz, mullite, hematite, corundum and calcium oxide and slag is found to be a glassy phase with some

Table 1
Physical properties of source materials.

Physical properties	FA	GGBS
Colour	Grey	White
Shape	Spherical	Angular
Average particle size (μm)	2.43	3.71
Specific gravity	2.3	2.94
BET surface area (m^2/g)	28.07	22.73