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Optimization of heat cured fly ash/slag blend geopolymer mortars designed by "Combined Design" method: Part 1

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HIGHLIGHTS

- Experimental study on heat cured FA/GGBFS blend geopolymers activated with NaOH.
- 'Combined Design' method is introduced for simultaneous evaluation of parameters.
- Excellent correlations prove the method's accuracy for the design of geopolymers.

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ABSTRACT

In the current study, heat cured fly ash (FA)/ground granulated blast furnace slag (GGBFS) blend geopolymer mortars activated with sodium hydroxide (NaOH) were investigated. For this reason, 'Combined Design' method of Experimental Design has been introduced for the first time as an exceptional tool for the evaluation of the simultaneous effect of the parameters affecting different properties of the mortars. Empirical models were developed to predict the mortars flow workability, compressive strength and flexural strength as well as to find the optimum levels for the most relevant conditions. For this purpose, 40 design points with different FA/GGBFS ratios ranging from 0/450 g to 450/0g; water 180–225 g, NaOH 50–150 g and curing temperature 50–100 °C were generated. For each of the 40 design points, mortars flow workability, flexural and compressive strength were tested and investigated. In the end, Scanning Electron Microscopy (SEM) analysis of three optimal mortar samples, representative of 100% FA, ~55/45 FA/GGBFS and 100% GGBFS was performed and analysed for the physical properties such as unit weight, water absorption and porosity. The generated models shows excellent correlation coefficients such as R^2 = 0.95 for flow workability and R^2 = 0.98 for compressive strength. The SEM analysis and physical test results comply with the model results, which show that "Combined Design" method is a very effective and timesaving method to reach very accurate results in the design of geopolymer mortars.

1. Introduction

Portland cement concrete has become an indispensable necessity for the construction sector, and for the moment, based on its extensive use, it seems to be unbeatable in its category. It is the second most extensively used material by humankind after water [1], and its production is estimated to exceed 10 billion tons per year [2]. Beside this, Portland cement production is an energy-intensive process that consumes enormous amounts of energy [3], approximately 2–3% of global primary energy [4]. Moreover, the cement industry is considered responsible for more than 7% of the CO_2 released in the atmosphere, nearly 1 ton of CO_2 for every ton of cement produced [2,3].

On the other hand, cementitious compounds are not durable enough against many external influences. The abovementioned technical and environmental problems make the production of alternative binders an attractive option. One of the most interesting work towards the production of a binder without the use of Portland cement is the alkali-activation of industrial wastes such as GGBFS and FA [5].

Ground granulated slag and fly ash storages constitute a significant environmental problem. Despite that their use in blended cement production or as pozzolanic materials is well known, only 20–30% of GGBFS [6], and about 53% of fly ashes [7] are being used, and the remaining part is stored in large extensions. Utilizing these types of by-products by activating with different alkalis has come

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up as an attractive substitute to the PC (Portland Cement) based binders [8]. Also known as geopolymers, they form when alumina and silica containing materials react of with alkaline solutions, resulting in an aluminosilicate structure [9]. The importance of the alkali-activation is not restricted in converting waste materials to useful products, but also in its ability to produce a high-performance binder from materials such as fly ash or blast furnace slag [10].

Davidovits defines geopolymer as amorphous three-dimensional alumino-silicate materials with ceramic-like properties which are formed by mixing solid silicate-aluminate raw materials with alkali or alkali silicate solutions [11,12].

The primary inputs are usually sources of amorphous alumina silicates with $SiO_2 + Al_2O_3 > 80$ wt%. The geopolymer structure consists of a chain, sheet-like and three-dimensional networks made of various monomeric or polymeric structures formed after the geopolymerization referred as Q unit types of connected SiO_4 (S) and AlO_4 (A) tetrahedral. In contact with a high pH alkaline solution, the input materials (amorphous or semi-crystalline alumino-silicates) dissolve progressively to form oligomers; geopolymers are then precipitated [13].

GGBFS is a glassy granular material formed when molten blast-furnace slag is rapidly chilled by immersion in water. The fast cooling of the slag minimizes the formation of crystal structures and transforms the molten slag into fine aggregate sized particles composed of mainly amorphous material. Because of its high silica and alumina content in an amorphous state, GGBFS shows pozzolanic behaviour similar to that of natural pozzolans [14,15].

Class F fly ash is a by-product of thermal power plants. It contains large quantities of amorphous alumina and silica. Therefore, it is a suitable and good source of material for producing geopolymeric binder owing to its chemical composition [16].

The mechanical properties and outer appearance of geopolymer mortars or concrete are very similar to ordinary Portland cement mortars or concrete. At the same time, geopolymers are known to have a very good performance when exposed to high temperatures or acidic environment [17,18].

Researchers report the use of different alkali-activators such as liquid sodium silicate, sodium metasilicate, sodium hydroxide and sodium carbonate in the production of geopolymer mortars [17,19,20].

Atis & Bilim studied the behaviour of alkali activated Portland cement/GGBFS mortars. They investigated the carbonation, compressive and flexural strength of the mortars produced with 0%–100% GGBFS and Na_2SiO_3 as activator. They concluded that 100% GGBFS activated mortars showed better performance than the Portland cement/GGBFS mortars [21].

A study on the alkali activation of GGBFS with NaOH, Na_2CO_3 and Na_2SiO_3 has been reported. Their hydration process shows to be similar to that of OPC (Ordinary Portland Cement), thus producing hydrates similar to C-S-H. Their activation energy is approximately 57.6 KJ/mol. Non-continuous microcracks are visible in the microstructure of the activated GGBFS concrete, which is very important regarding the permeability of concrete. For that reason, the crack formation mechanism of alkali activated slag has been in the focus of many studies [11,12].

Bakharev et al. investigated the alkali activation of GGBFS, using different activators in different modules and concentrations. As a result, for same water/cement ratio, the Na_2SiO_3-activated GGBFS mortars demonstrated higher strength than OPC mortars. Early strength and setting time decreased as the activator's silica modules increase. On the other hand, the increase of alkaline activator concentration, increased the shrinkage of the alkali-activated mortars [19].

In the experimental work of Xie & Xi, Class F fly ash geopolymer mortars' hardening mechanism was investigated. When the water glass activated FA mortar samples were cured at 60 °C for 24 h, they observed the formations of amorphous or low order crystals of $Na_2O\text{-}Al_2O_3\text{-}SiO_2$ [22].

Olivia & Nikraz investigated the strength development, water absorption and permeability of alkali-activated Class F fly ash concrete. They reported that, when properly designed, Class F geopolymer concrete can reach to good strength and physical parameter. [23].

Tho-in et al. studied the characteristics of high calcium FA geopolymer concrete. Their investigation showed that activated high calcium FA concretes has satisfactory strength, density, porosity similar to that of OPC concretes [24].

According also to some of the researchers mentioned above, curing temperature shows to have a significant effect on the alkali activation reaction of ground granulated slag and fly ash [6,22].

Bakharev et al. investigated the effect of curing temperature on microstructure, shrinkage, and compressive strength of alkali-activated slag (AAS) concrete, activated with sodium silicate and sodium hydroxide. They concluded that heat treatment was very effective in reducing drying shrinkage of AAS concrete and promoting high early strength, but the strength of AAS concrete at later ages decreased. They suggested a pre-treatment at room temperature before elevated temperature curing for further improvement of early strength and decrease shrinkage of AAS concrete [25].

Kovalchuk et al. studied on the role of curing conditions in the development of mechanical strength of the alkali-activated fly ash. They recommended dry curing at 150 °C only for sodium hydroxide-based systems, since water glass-based ones tend to delay reaction rate. Steam curing was found to have an intermediate effect on strength development, between curing in covered mould and dry curing [8].

Shi & Day investigated the hydration and strength development of 50/50% slag/fly ash (both Class C and Class F) geopolymer mortars activated with NaOH and Na_2SiO_3. The type of fly ash resulted of no significant effect, whereas the highest strength values were obtained from the slag mortars activated with sodium silicate [25].

Luga & Atiş in their experimental work activated GGBFS/FA in different blend ratios with sodium meta-silicate or silica fume dissolved in sodium hydroxide and cured at room temperature. They reported that increasing the fly ash content decreases the compressive strength of the GGBFS/FA blend geopolymer mortars [26].

Puertas et al. studied on fly ash/slag geopolymers activated with sodium hydroxide. They used 2 M and10 M NaOH solution to activate the blended solid phase and cured them in 25 °C and 65 °C. They reported that the geopolymerization rate and strength development increase with the slag % and activator concentration increase, on the other hand the curing temperature didn't show any significant effect on the final strength [6].

Zhao et al. in their investigation reported about the very important effect of fly ash/slag on the strength of geopolymer mortars. They concluded that good strength and low-cost products could be obtained by controlling the fly ash percentage used in the blend [27].

Activated FA/GGBFS binders have been in the focus of many recent studies. Their coexistence in the same system and the optimum proportions are still subject of discussion [28], because different studies show different and contradictory results [6].

In order to obtain the most beneficial information in the most precise way, the experiment needs to be planned accurately. Also, different problems need to be examined systematically, and for that reason, experimental design and optimisation would be a useful tool [29]. Previous studies have used different methods to optimise concrete mixes such as mixture design of experiment and response surface methodology for polymer concrete, [30] or foam concrete [31], Taguchi method for self-compacting [32] and fly ash geopolymer concrete [33].