



The evaluation with anova of the effect of lime admixture and thermal cure time on fly ash paste activated with sodium silicate solution



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HIGHLIGHTS

- Fly ash was activated with sodium silicate solution and quenched calcium lime.
- The samples were subjected to thermal curing at +105 °C for 12, 24 and 48 h.
- The curing time had no statistically significant effect on water absorption.
- The lime rate had no statistically significant effect on the apparent density.

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ABSTRACT

Class F fly ash obtained from Kütahya Seyitömer (Turkey) thermal power plant was used as raw material in this study. Fly ash was activated with sodium silicate solution (water glass) and quenched calcium lime was substituted to the mixture at rates ranging from 10% to 30%. Then, samples were formed in $4 \times 4 \times 16$ cm metal molds by means of the vibration method. The samples were subjected to thermal curing in a laboratory type oven at +105 °C for 12, 24 and 48 h in order for them to acquire resistance. After the curing process, the physical properties such as apparent porosity, apparent density, bulk density, water absorption by weight and linear shrinkage, and the mechanical properties such as flexural strength and compressive strength of the samples were determined by the three samples for each mixture. After the physical and mechanical tests, a two-factor analysis of variance (ANOVA) and a comparative analysis of the mean values were carried out in order to investigate the effects of the variables used in the production and the preparation of the samples on the values. Consequently, it was determined that in the fly ash–lime mixtures, 24 h curing for the samples with 20% lime substitution is very efficient and that these samples yield the best compressive strength.

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1. Introduction

The geopolymerization process which generates new materials with cement characteristics is a complex process and has been poorly understood in detail so far [1,2]. Compared to ordinary portland cement (OPC), geopolymers have excellent properties such as high compressive strength [3,4], low shrinkage [4], acid resistance [4,5], fire endurance and no toxic fume emission, low thermal conductivity [4], excellent heavy metal immobilization and high temperature stability [1].

The geopolymerization process begins with the reaction of alkali silicate and alkali salts at low temperatures with industrial waste such as fly ash, coal slag, blast furnace slag, silica fume or with dehydrated aluminosilicate soil solids such as volcanic tuffs,

crumbled natural metamorphic rocks or kaolinite clay (metakaolinite) calcined at 750 °C [6]. A geopolymer reaction starting at room temperature is extended by subjecting it to heat treatment in an oven or in a steam room between the temperatures of 40 and 100 °C after achieving the desired mixture. Thus, the properties of the desired material are improved [7].

Alkaline activators have an important role in the dissolution of Al and Si oxides in the raw material structure which will be activated. Hence, sodium hydroxide (NaOH), potassium hydroxide (KOH), potassium silicate or sodium silicate is the most widely used activators for the synthesis of aluminosilicate reactive material in the activation process [8–15]. The characteristics of the product obtained in the activation process can vary depending on the temperature and time of the applied heat curing or calcination process. Alkali activation is generally described as geopolymerization [16].

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Geopolymer maintains its internal reactions after it takes form similarly to portland cement. The exothermic reaction of geopolymer continues until it forms the three-dimensional oligomer macromolecular structure. Water glass (sodium silicate) (Na_2SiO_3) or gibbsite (sodium aluminate) ($\text{Na}_2\text{O}-\text{Al}_2\text{O}_3$) is added so that the main components in the fly ash dissolve together with alkaline salt in a more regular fashion and this way, the properties of the geopolymer material improve. Solutions to which these chemicals are added are called solid-gel solutions. The use of a great amount of water in such solutions for the dissolution of chemicals reduces the compressive strength properties of the material. The concentration of precipitate sodium ion is high in such compositions. Nuclear magnetic resonance (NMR) shows that sodium aluminate tetrahedral containing Al and Si, which are the main elements of fly ash, facilitates the formation of the aluminum bond structure and that water glass increases the cross-linked silicate molecules [6].

Geopolymers are generally prepared at low temperatures using fly ash [2]. Fly ash, which is a thermal power plant solid waste, contains silicon and aluminium which can be activated by alkaline solutions [17]. Since fly ash particles are formed at high temperatures, they have a chemically stable and glass-like structure. Therefore, the reactivity in the process of hydration of fly ash is low. Alkali activations are used to increase the pozzolanic reactivity of fly ash, and alkalis such as NaOH, $\text{Ca}(\text{OH})_2$ etc. are used in these processes [18].

Liquid solution which will be used in the activation process can be either a single material or formed by mixing a number of different materials [17]. In the activation process, a strong alkaline medium is needed to increase the surface hydrolysis of the raw material particles with the dissolution of an amount of silica and alumina particles in the material. This medium can be obtained by using a single or combined alkaline solutions referred to as activators [16].

The raw material of alkaline lime is CaO and is obtained by the sintering of CaCO_3 , also known as limestone, between 850 and 900 °C. Limes are not used in the form of calcium oxide (CaO). They have to be quenched, and the quenching of the lime is a hydration process. When quenched, the size of the lime increases 2–2.5 times. Deformation and cracks may occur in the material if the lime containing material is not thoroughly quenched. When hydrated lime–calcium hydroxide $\text{Ca}(\text{OH})_2$ –(CH) is treated with plenty of water, CH particles are dispersed in suspension in the water [19].

Information regarding the surface of CH particles in an environment where CH is abundant is scarce in the literature. However, one study shows that the consumption rate of CH increased in the CH and fly ash mixtures with an increase in the content of CH during the first mixture. The increase in the consumption rate was accounted for by the increase in the calcium ions in parallel with the increase of CH surfaces in the solution. Another study states that in the case of an increase in the CH/fly ash ratio in the starting material, the consumption rate of CH increases [20].

It is observed that in general studies on the activation of fly ash prefer to use two different alkaline activator materials, and different curing times and temperatures in the activation process. In one of these studies, Thokchom et al. cured the samples produced by using fly ash and sand at 85 °C for 48 h [21]. Rickard et al. used sodium silicate and sodium aluminate solutions for the synthesis of fly ash based geopolymer and cured the samples at 70 °C for 24 h [22]. Sarker used sodium hydroxide and sodium silicate in the production of fly ash based geopolymer concrete and cured the samples at 60 °C for 24 h. He suggested that the samples yielded satisfactory results [23]. In addition, there are some studies in the literature in which fly ash content geopolymer materials are produced [24–29]. Furthermore, NaOH is a good activator for the

activation of fly ash. It is also stated that, due to the high Si content, the use of water glass during geopolymerization leads to a high mechanical resistance in samples [30]. However, the use of water glass in the form of powder is observed to lead to lower performance than when the liquid form is used [10].

There is little information in the literature on fly ash activated with alkaline solution–calcium hydroxide pastes. It is pointed out that the thermal curing of fly ash–CH containing systems at 100 °C, increases hydrate products to higher levels [20]. Therefore, different from the literature, in this study, fly ash–lime pastes activated only with sodium silicate solution were subjected to thermal curing at high temperatures. $\text{Ca}(\text{OH})_2$ substitution was carried out for the activation process of the fly ash to be more effective during the preparation of the pastes and then the properties of the samples were investigated. Two-factor ANOVA tests were applied in order to determine the effects of the factors (curing time and lime rate of substitution) on the data obtained. The two-factor ANOVA test is a statistical method which analyses the main effects of factors and the joint effect of two factors on a dependent variable instead of analysing separately the effect of two factors between groups on a dependent variable [31]. The literature includes studies in which ANOVA tests are applied [32–38].

2. Material and method

2.1. Materials

Fly ash with low levels of calcium oxide (CaO) and a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of 2.41 by mass was used in this study as raw material (Table 1) and it was obtained from Kütahya Seyitömer thermal power plant. The chemical composition of the fly ash was determined by X-ray fluorescence (XRF) analysis and it is presented in Table 1. According to the data obtained from the XRF analysis, the fly ash was of Class F according to ASTM C-618 [39].

Fig. 1 presents the XRD graphic of the mineralogical analysis of the fly ash. According to the XRD analysis, the main minerals in the fly ash are quartz, magnetite, anhydrite, anorthite and hematite.

S-KK 80 T quenched calcium lime, which conforms to TS 4022 [41], was used as replacement material during the production of the samples (Table 2).

2.2. Preparation of samples

In the preparation of the samples, fly ash and lime were first dry mixed until reaching a homogeneous state. Then, mixing water which was equal to the sum of the mass weight of the mixture was prepared. Accordingly, one unit of dry material was mixed with one unit of mixing water. The mixing water was prepared in a way that $\frac{1}{4}$ of it was water and $\frac{3}{4}$ of it was sodium silicate solution (water glass) ($\text{Na}_2\text{O}-\text{SiO}_2$) by weight for all the mixtures. Table 3 presents the prepared mixtures and the ingredients they contain.

Through substitution, lime admixture was added to the fly ash based mixtures activated with sodium silicate solution. After the mixtures were homogeneously mixed, they were placed into cement mortar molds ($4 \times 4 \times 16$ cm) with the help of a vibrating table. Then, the samples were subjected to thermal curing in a laboratory type oven at +105 °C. Three samples for each sample group were used in the physical and mechanical tests. Three different curing times (12, 24 and 48 h) were used in the curing of the samples.

2.3. Tests

After the curing processes, the samples were placed in a water tank for 24 h to determine their physical properties. Water

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