



Mechanical properties and setting time of ferrochrome slag based geopolymer paste and mortar



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HIGHLIGHTS

- The objective of work is to learn utilization of FS in the production of geopolymer.
- This paper presents the effects of alkali dosage on the geopolymerization of FS.
- This paper presents the effects of silica modulus on the geopolymerization of FS.
- The setting time and hydration heat of geopolymer paste samples were investigated.
- The compressive strength of geopolymer mortar samples were investigated.

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ABSTRACT

Many researches have been done to investigate using raw materials in the production of geopolymer cements. This paper presents the effects of alkali dosage and silica modulus when using sodium metasilicate solution at different curing conditions on the geopolymerization of ferrochrome slag (FS). As alkali activation for geopolymerization, NaOH and Na₂SiO₃ solution were used. Geopolymer cement was produced using FS as raw material with 3 different silica modulus (0.50, 0.60 and 0.70) and 4 different Na₂O concentrations (4%, 7%, 10% and 12%). The setting time, hydration heat and compressive strength of geopolymer paste samples and compressive strength of geopolymer mortar samples were obtained. The setting time varied between 120 and 870 min, it showed variability depending on Na₂O content. The highest 28 day compressive strength of the geopolymer paste samples was obtained from one with Na₂O concentration of 7% and silica modulus of 0.70. Geopolymer mortars were prepared for the determination of compressive strength by adding FS:sand:alkali activator ratio 1:2:0.30, 0.35 and 0.40. The specimens were cured at 60 °C and 80 °C kept for 20 h and the other mortar samples were stored under laboratory conditions. Compressive strength of the material decreased, when w/b (water/binder) ratio increased. The highest 28 day strength of the geopolymer mortar was obtained at 0.30 w/b ratio and laboratory temperature curing conditions. The hydration heat of geopolymer paste samples was found to be less than normal Portland cements. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were investigated to study the microstructural properties of the geopolymers.

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1. Introduction

Ferrochrome slag (FS) is residue from the combustion of chromite which leads to waste management proposal. Apart from the

main product many by-products are produced in this factory. The disposal, removal and storage of by-products is a big problem. One of these by-products is FS. Many studies [1–3] have been done over FS used in the construction industry as road construction, cement additive and aggregate.

Practices in the delivery of build infrastructure, there are specific concerns over atmospheric CO₂ concentrations which at 390 ppm

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reached record breaking levels. Major CO₂ producing sectors, such as power generation, transportation, oil refining and manufacturing of steel and concrete are under pressure to adopt measures that would drastically reduce the global CO₂ emission rate by 2030. Within the concrete industry, cement manufacturing is the main culprit. Each year, the concrete industry produces approximately 12 billion tonnes of concrete and uses about 1.6 billion tonnes of PC worldwide. The production of cement is increasing about 3% annually. Indeed, with the manufacture of 1 tonne of cement approximately 0.94 tonnes of CO₂ are launched into the atmosphere. The International Energy Authority holds the cement industry responsible for emitting between 6% and 7% of all the CO₂ emission into the atmosphere. Among the green house gases, CO₂ contributes about 65% of global warming. Beside the emission of CO₂, cement industry launches SO₃ and NO_x which can cause the greenhouse effect and acid rain. This is particularly serious in the current context of climate change caused by CO₂ emissions worldwide, causing a rise in sea level and the occurrence of natural disasters and being responsible for future meltdown in the world economy [4]. In recent years, there is an increasing awareness on the quantity and diversity of hazardous solid waste generation and its impact on the human health. Increasing concern about the environmental consequences of waste disposal has led researchers to investigate the utilization of the wastes as potential construction materials. Among the researches, the successful one was through the development of geopolymer concrete to eliminate the use of cement [5].

Aluminosilicate inorganic polymers, also called geopolymers, were firstly described by Davidovits as materials formed under high alkali condition from aluminosilicate solid and alkali silicate solutions [6,7]. Geopolymer concrete is well-suited to manufacture precast concrete products that can be used in infrastructure developments [5,8]. Several materials containing silica and alumina can be used as starting source materials. These include the conventional starting materials viz., slag, metakaolin and class C fly ash [7,9,10]. According to previous research, when pozzolanic materials such as fly ash and metakaolin, which are rich in Si and Al, react with an alkali activator, good mechanical and physical properties can be obtained [11]. Geopolymer has diverse advantages, such as a high mechanical strength after curing at 60–75 °C for 24 h [12], good resistance to high temperatures [13] and chemical resistance to sulfates [14]. Based on these factors, alkali-activated geopolymer can be considered as a suitable material to replace ordinary Portland cement (OPC) due to its good environmental friendliness and use of advanced technology [15]. Bilim and Atiş [16] reported that many valuable research results have been reported on alkali activated slag binders which have recently received much attention from the academic field. However, the activation of FS with alkali and the properties of paste and mortar produced with geopolymer binding substance as a result of activation with alkali and FS is still unknown. Besides the properties of binding occurring via alkali activation method is unknown due to FS having low Si and Al content. The objective of the present work is to learn the possibility of utilizing FS in the production of geopolymer cements. For this purpose, geopolymer cement was produced using FS as raw material with 3 different silica modulus (0.50, 0.60 and 0.70) and 4 different Na₂O concentrations (4%, 7%, 10% and 12%). The setting time, hydration heat and compressive strength of geopolymer paste samples and compressive strength of geopolymer mortar samples were investigated experimentally.

2. Materials and methods

The raw material employed in the paper was FS. In this study, FS obtained from Elazığ Ferrochrome plant in the west of Elazığ, Turkey, was used as the basic aluminosilicate material to manufacture geopolymers. The common materials used as alkaline solution in producing geopolymer are sodium metasilicate (Na₂SiO₃) and

Table 1
Chemical composition of FS (%).

| Component, (%) | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | Cr ₂ O ₃ |
|----------------|------------------|--------------------------------|--------------------------------|------|-------|--------------------------------|
| FS | 33.80 | 25.48 | 0.61 | 1.10 | 35.88 | 2.12 |

sodium hydroxide (NaOH). Bakri et al. [17] noted that the compressive strength of geopolymers also mainly depended on the content of pozzolan fine particles (smaller than 43 µm). Therefore, about 95% of the particles was smaller than 45 µm. The chemical materials used in this study were also Na₂SiO₃ and NaOH. The chemical composition of FS, NaOH and Na₂SiO₃ solution used were given in Tables 1 and 2, respectively. As seen in Table 1, FS has a low content of SiO₂ and Al₂O₃. The NaOH solution was prepared by dissolving NaOH pellets (with 98% purity) in water and Na₂SiO₃ (Na₂O = 8.9%, SiO₂ = 28.7% and water = 64.8% by mass) with silicate modulus was 3.22. The mass of NaOH solids in a solution was varied depending on the required concentration of the solution. Enough NaOH was added to Na₂SiO₃ solution for preparing an alkali-activator having silica modulus of 0.50, 0.60 ve 0.70. The different ratios were obtained by adding NaOH to the liquid sodium silicate. The Na₂O contents of the designed geopolymer cement mixes coming from both sources of NaOH and Na₂SiO₃ of the activator were adjusted at four different values of 4%, 7%, 10% and 12% (by weight of dry FS). After adding activators to the dry binders and enough mixing, the pastes were cast into molds of 50 × 50 × 50 mm in size. The molds were held at ambient conditions (22 °C and 60% relative humidity) until the specimens were hardened to be removed (1 day). After the curing period, the test specimens were left in the molds for at least 6 h and demoulded. The specimens were then stored at ambient condition without any curing process until the time of testing.

In fresh pastes, the initial and final setting time was determined using the Vicat apparatus in accordance with the ASTM C191 [18] standard test method. The hydration heat of geopolymer paste samples was also measured by isothermal calorimetry at 25 °C over the first 96 h of reaction. The hydration heat of paste mixtures was examined for different silica modulus and Na₂O content using isothermal calorimetry instrument. It was utilized scanning electron microscopy to investigate the microstructural properties of geopolymer samples and X-ray diffractometry technique was used to determine the crystalline mineral phases in FS. Then, 7 and 28-day compressive strength of geopolymer paste samples were performed according to ASTM C109 [19] using a 50 mm size cubic sample. For each mix, three specimens were used and the average of these three values was reported.

Initially, the effect of silica modulus and Na₂O content of the geopolymer paste mixes was investigated. The Na₂O content was changed from 4 to 12 by varying quantity of NaOH solids in activator solution. Later on, the effect of curing regime for geopolymer mortar samples was studied. Other parameters of mix such as quantity of Na₂SiO₃ and NaOH was kept constant for all samples.

Mortar samples were produced by using results on the mixture ratio that have the highest compressive strength for the geopolymer paste samples. For mortar samples, extra water was added in the activator solution so as to result a water to FS ratio of 0.30, 0.35 and 0.40. The amount of water in the activator was taken into account. All samples were formulated with a binder/sand ratio of 1:2. Quartz sand was used as sand in all of geopolymer mortars samples.

The mixture was then cast into 50-mm cubic mold. 3 different curing conditions were used. First curing regime, the specimens were cured at 60 °C for 20 h, then hold on laboratory conditions at 20 ± 2 °C and relative humidity 60%, second curing regime, the specimens were cured at 80 °C for 20 h, then hold on laboratory conditions at 20 ± 2 °C and relative humidity 60%. Last curing regime, mortar samples were stored under laboratory conditions (20 ± 2 °C and relative humidity 60%). The compressive strength values of the mortar samples were examined in accordance with the ASTM C109/C109 M [19] standard test method at the age of 3, 7, 28 and 90 days.

Table 2
Chemical properties of sodium hydroxide and sodium metasilicate solution.

| Properties | Sodium hydroxide | Sodium metasilicate solution |
|---------------------------------------|------------------|----------------------------------|
| Molecular formula | NaOH | Na ₂ SiO ₃ |
| Molecular weight (g/mol) | 40.00 | 122.06 |
| Color | White | White |
| pH | 13–14 | – |
| Relative density (g/cm ³) | 2.13 | 1.38 |
| Na ₂ O (%) | – | 8.9 |
| SiO ₂ (%) | – | 28.7 |
| H ₂ O (%) | – | 64.8 |

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