Enhanced characteristics of alkali activated slag/ grog geopolymer bricks

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Abstract: The development of alternatives to conventional Portland cement produced with more ecoefficient processes (lower energy consumption and carbon foot print) is of great importance to the industry and world climate. Alkaline cements and concretes are an effective alternative to these traditional cements. Geopolymer bricks that are prepared by partial binder substitution, water cooled slag, with clay brick waste (grog) in a ratio ranging from 0 up to 100%, and with a fine sand ratio of 15% of the total weight. Also, sodium hydroxide activator was used in a ratio of 8% of the total weight. The properties of the produced Geopolymer bricks have been investigated through measurement of compressive strength, water absorption, FTIR, XRD and SEM imaging. The results revealed the feasibility of substituting water cooled amorphous slag with grog in synthesization of Geopolymer bricks. Resulting in superior quality, where the compressive strength values, with a substitution of slag with up to 40% grog, exceeds 40 MPa and so can be used in production of heavy duty bricks that can withstand harsh weathering conditions. However, further substitution leads to the lowering of mechanical properties of the brick, as a result of increased crystalline content and deficiency of activator to dissolve all crystalline fractures.

Keywords: slag, grog, heavy duty, eco-friendly, brick.

1. Introduction

Clay bricks are the oldest and the most commonly used building materials in masonry construction. However, the production process of clay brick consumes high amounts of energy and releases enormous amounts of dangerous emissions (greenhouse gases) into the atmosphere of our planet. In addition, there are diminishing supplies in natural resources of raw materials (natural clay, silt, sand, etc.) used to fabricate these clay bricks. Accordingly, there is a worldwide need to find an effective alternative that is also environmentally friendly. The recycling of waste products, by incorporating them into building materials, is considered an appropriate solution to this pollution crisis. Many attempts have been made to incorporate waste in the production of bricks, including rubber, limestone dust, sawdust, processed waste tea, fly ash, polystyrene and sludge [1]. Nevertheless, the utilization of these waste products is still limited due to the lack of consistent quality and type of waste products used. Thus, impacting the final physical and mechanical performance of fabricated brick.

Recently research attempts have been done on the utilization of waste materials to manufacture bricks, composed of 100% of recycled materials using Geopolymerization process, which utilizes one source of waste or blended waste in production [coal fly ash, slag, mining tailings and cement kiln dust (CKD)] [2-6].

The Geopolymerization process is a series of reactions which interact with each other: (1) dissolution of amorphous aluminosilicate parent materials in an alkaline media, (2) formation of Si and/or Si-Al oligomers in the aqueous phase, and (3) polycondensation of the oligomers to form a three-dimensional aluminosilicate framework. In principle a wide range of materials, both natural minerals and industrial

waste, can be used to make Geopolymer, only if they contain sufficient amorphous silica and alumina [7].

The production of traditional brick consists of the following steps: 1. Quarrying of natural raw materials. 2. Processing and screening of the quarried material. 3. Mixing raw materials with water, forming/extruding, and cutting into standard-sized blocks. 4. Drying at room temperature or in sunlight for one to two days. 5. Kiln firing where the dried blocks are heated up to 1300 °C.

In the production of Geopolymer bricks, steps 1 and 5 are eliminated due to waste materials being used, rather than the natural materials and therefore the Geopolymerization process is initiated at an ambient temperature of around 90 °C, dependent upon the components of the used waste and its reactivity **[8]**.

Ferone, et al. [2], utilized coal fly ash to produce Geopolymer bricks and studied its mechanical performance. He found that using sodium based alkali activation (sodium hydroxide (NaOH) + sodium silicate) solution in the activation of coal fly ash and curing the bricks at 60 °C, even for just 24 hours (rather than the typical 7 days), increases the mechanical strength up to 3.5 times.

Bashar et al. [3], investigated the possibility of using steel slag in production of Geopolymer bricks, using 8M sodium hydroxide and sodium silicate solution as an alkali activator and curing at 60 $^{\circ}$ C for 48 hours. They were able to develop steel slag Geopolymer with a compressive strength of 10.5 MPa.

Radhakrishna et al. [4], investigated the production of Geopolymer bricks based on a blend of fly ash and blast furnace slag. He used sodium based alkali activation (NaOH + sodium silicate), water content (15-25%) and cured both at room temperature and 60° C. The results indicated that the final strength of the produced Geopolymeric brick is controlled by the following: quality of coal fly ash, binder to aggregate ratio, molarity of activator solution, fine aggregate type and curing conditions. The presence of calcium in slag contributes to the improvement noted in the physical and mechanical properties of Geopolymer bricks, owing to the enhancement of Geopolymerization and formation of Calcium Silicate Hydrate (CSH) gel.

Ahmari & Zhang [5], used copper mine tailing to make Geopolymer bricks and studied the effects of four major factors on their physical and mechanical properties; sodium hydroxide solution (with a concentration of 10 and 15 M), water content (8-18%), forming pressure (0-35 MPa) and curing temperature (60-120 °C). Based on the test results, they suggest an alkali activation of 15 M sodium hydroxide and an optimum curing temperature of around 90 °C and with adequate forming pressure, resulted in an eco-friendly Geopolymer brick produced from copper mine tailings.

Ahmari & Zhang [6], investigated the effect of CKD content (0–10%), sodium hydroxide concentration (10 and 15 M) and initial water content (12–20%) on the physical and mechanical properties and the durability of copper mine tailing (MT)-based Geopolymer bricks with cement kiln dust. Based on the experimental results, it is indicated that the addition of CKD resulted in significant improvement of the physical and mechanical properties as well as improved durability of MT-based Geopolymer bricks.

The objective of this research is to investigate the possibility of using blended waste to make an eco-friendly Geopolymer brick. Different blends are produced by

partial substitution of blast furnace slag with a specified percentage of grog incorporating a fixed percentage of sand, followed by alkali activation, moulding and curing of brick specimens. The effect of these substitutions and their impact on the physical, mechanical and microstructural characteristics of the synthesized Geopolymer bricks will be inspected.

2. Experimental procedures

2.1. Materials

The materials used in this research were Water cooled slag, a type of ground granulate blast furnace slag (GGBFS), supplied by the Iron and Steel Factory-Helwan, Egypt; clay brick waste (grog) supplied from landfills located in 6th October, Egypt. Sodium hydroxide (NaOH) 99% purity (in pellet form) is used as an alkali activator, obtained from SHIDO Co., Egypt, The used sand dunes used for mortar preparation are sourced from fine sand (<1 mm) from Oases (Wahat)-Road, Egypt.

The chemical compositions of the used raw materials are given in Table (1). Mineralogical characterization of the raw materials was accomplished using X-ray diffraction analysis in powder form as represented in Fig. 1. Water cooled slag is defined as a rich aluminosilicate material composed from the prominent content of SiO_2 and MnO, Fe₃O₄, CaO and Al₂O₃ as illustrated from Table (1), while it is mineralogicaly composed of amorphous materials.

On the other hand, clay brick waste is composed of high percentages of silica and alumina and low percentages of calcium, magnesium and alkalis, which are considered the main component in the Geopolymer formation. This is also confirmed by XRD patterns where quartz and albite are predominant, in addition to lesser amounts of hematite (Fe_2O_3).

Material	SIO2	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO3	K ₂ 0	Na ₂ O	TIO2	MnO₂	P ₂ O ₅	Cŀ	L.O.I.	SrO	BaO	Total
Water – Cooled Slag (GGBFS)	36.67	10.31	0.50	38.82	1.70	2.17	1.03	0.48	0.57	4.04	0.04	0.050	0.12	0.18	3.28	99.96
Clay Bricks Waste (Grog)	60.00	14.00	8. 0 6	5.76	1.48	2.74	1.24	1.42	1.05	0.08	0.16	0.05	3.72	-	-	99.77
Fine Sand (Sand dunes)	89.91	2.00	1.45	1.56	1.91	0.87	0.37	0.06	0.03	0.04	0.03	0.12	1.65	-	-	99.98

Table (1): Chemical composition of starting raw materials (Mass, %)



Figure (1): XRD analysis of the starting raw materials.

2.2. Geopolymerization and Curing

Geopolymer mixes A, B, C, D, E and F as defined in Table (2) were made by combining raw materials, each mixture passing through 90 μ m sieve, with the alkaline activator (8% NaOH of the total weight) for 10 minutes, then an additional 5 minutes with an electronic mixer. The water-binder ratio (w/b) was 0.227: 0.339 by mass. The mixture was cast into 25×25×25 mm cubic-shaped moulds, vibrated for compaction and sealed with plastic sheets to minimize evaporation loss.

The mix design of the mixes is tabulated in Table (2) where the water content increases with additional grog content as the silica content in grog absorbs water. The table also illustrates the oxide ratios of the reacting raw materials, where the silica/alumina (Si/Al) ratio increases to 3.90 with 40% grog content then subjected to gradual increase up to 4.29 as more grog is added. It is worth mentioning that the optimum range of oxide molar ratios resulted in a three dimensional network with a more branched. homogeneous and compact structure are as follows: $0.2 < M_2 O/SiO_2 < 0.48$, $3.3 < SiO_2 / Al_2O_3 < 4.5$, H_2O/M_2O , 10-25 [9, 10] and M₂O/Al₂O₃, 0.8 to 1.6 [11].

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Mix no.	Water cooled slag	Grog	Sand (<1mm)	NaOH	Water/ binder	T.M ₂ O/ Al ₂ O ₃	SiO ₂ /Al ₂ O ₃	T.M ₂ O /SiO ₂
Α	100	-	15	8	0.227	0.922	3.56	0.242
В	80	20	15	8	0.236	0.892	3.74	0.220
С	60	40	15	8	0.247	0.856	3.90	0.202
D	40	60	15	8	0.254	0.824	4.05	0.188
E	20	80	15	8	0.273	0.796	4.17	0.176
F	-	100	15	8	0.339	0.771	4.29	0.165

Table (2): Composition and proportion of the studied mixes.

All mixes were left undisturbed to cure at an ambient temperature for 24 hours, and then cured at a temperature of 40 °C and 100% relative humidity. At the end of the curing process, the cubic specimens were subjected to compressive strength measurements, as defined in section 3.3, then further subjected to stopping of hydration process by drying the crushed specimens for 24 hours at 105 °C [12], and then preserved in a air tight container until the time of testing.

2.3. Methods of Investigation

The chemical analysis was conducted using Axios, Wave Length Dispersion X-ray Fluorescence (WD-XRF) Sequential Spectrometer, (Panalytical, Netherland, 2009). The X- ray diffraction -XRD analysis was carried out using a Philips PW3050/60 Diffractometer. The data was identified according to the XRD software. Perkin Elmer FTIR Spectrum RX1 Spectrometer (Fourier Transformation Infra-Red) was used to evaluate the functional groups in the sample. Small amounts of potassium bromide (KBr) and Geopolymer powder were mixed together and placed in the sample holder then pressed at 295 MPa for 2 minutes to produce a specimen for examination. The FTIR wave number ranged from 400 to 4000 cm⁻¹ [13, 14].

Water absorption measurements of the Geopolymer bricks were carried out according to **ASTM C140** [15]. The percentage of absorption was calculated using the following equation:

Absorption (%) = $[(W2 - W1)/W1] \times 100$

Where W1 = weight of specimen after complete drying at 105 °C; W2 = final weight of surface dry sample after immersion in water for at least 24 hours.

Compressive strength tests were carried out using five tones German Brüf pressing machine with a loading rate of 100Mpa/s determined according to **ASTM-C109** [16]. The microstructure of the hardened specimens was studied using Scanning Electron Microscopy- SEM Inspect S (FEI Company, Netherland) equipped with an energy dispersive X-ray analyzer (EDX). Removing of the free water was accomplished by drying the crushed specimens for 24 hours at 105 °C [12].

3. Results and Discussion

3.1. X-Ray diffraction analysis

Geopolymers are often described as 'X-ray amorphous' [17, 18]. Fig.2 shows the XRD pattern of mixes A, B, C and E; after 28 days curing with a featureless hump centered at approximately $17 : 35^{\circ}$ (2 Θ), denoting amorphous phases (silica and alumina) that are actively involved in Geopolymerization reaction. The presence of sharp phases indicates crystalline phases; quartz and feldspar originated from the parent materials, sand and grog. The patterns also confirmed the presence of calcium silicate hydrate CSH) and calcite which is probably due to carbonation of the geopolymer specimen which may occur while curing or transporting, and faujusite (zeolite), which is formed due to the effect of hydrothermal alkaline reaction of aluminosilicate materials resulting in the development of zeolites [19]. Furthermore, it was observed that more faujusite crystals are formed in specimens C and E with high Si/Al ratio, while fewer faujusite crystals formed in specimens A and B with low Si/Al ratio. The formation of higher Si/Al ratio crystals, with its slower alumina release rate, can be attributed to the lower availability of aluminum, which provides a suitable environment for the formation of crystal phases with higher silicon content [20].

The patterns for the four Geopolymer mixes show that the main crystalline phases (Quartz and hematite) vary with the change in slag/grog ratio, indicating that these phases are not reactive or involved in the Geopolymerization process, but simply present as an inert fillers in the mix. Additionally, the high intensity and span of the CSH peak and the higher hump in specimen C (40% grog), compared to the other mixes, indicate a higher Geopolymer content.

The X-ray patterns of Geopolymer specimen C (40% grog) at different curing periods are shown in Fig. 3. The patterns illustrate an increase in the span of the CSH peak and higher amorphous hump with a curing time of up to 90 days. This can be explained by an increase in rate of dissolution of slag and grog, as well as an increase in rate of the release of calcium, silica and alumina into the system with curing time, thus positively affecting the polymerization process and increasing Geopolymer content. Also, the increase in interactions between the calcium supplied from slag along with the freely dissolved silica in the system resulted in the formation of a crystalline CSH phase at later curing stages.



Figure (2): XRD patterns of 28 days Geopolymer mixes having various grog ratios. [C: Calcite, CSH: Calcium silicate hydrate, Fj: Faujasite, H: Hematite, Q: Quartz].



Figure (3): XRD patterns of 28 days Geopolymer mix containing 40 % grog as a partial replacement of slag at various curing ages. [CSH: Calcium silicate hydrate, F: Feldspar, Q: Quartz].

3.2. Fourier Transform Infrared FTIR spectroscopy

FTIR spectra of 28 days cured at 40 °C and 100% RH, Geopolymer specimens with various percentages of grog substitution are shown in Fig. 4. The FTIR bands are as follows: stretching vibration of O-H bond at approximately 3440 cm⁻¹; stretching vibration of CO₂ is located 1415-1450 cm⁻¹; asymmetric stretching vibration (T–O–Si) at about 950 - 975 cm⁻¹ where T=Si or Al; symmetric stretching vibration of CO₂ at approximately 870 cm⁻¹; symmetric stretching vibration (Al–O–Si) approximately 760 cm⁻¹; symmetric stretching vibration (Si–O–Si) at approximately 676 cm⁻¹ and bending vibration (Si–O–Si and O–Si–O) in the region of 440-450 cm⁻¹.

From the pattern it is evident that there is an increase in the intensity of N-A-S-H (sodium alumino silicate hydrates) gel and a shift to a lower wave number with an increased percent of substitution of slag by 40% grog. Also, an increase in intensity of broad bands appeared in all IR spectra of the mixes, in the region of 3500 and 1600 cm⁻¹ assigned to stretching (-OH) and bending (H-O-H) vibrations of bound water molecules, which are surface absorbed or entrapped in the large voids of the polymeric network [**21**, **22**]. This implies that Geopolymers ability to adsorb water in their three dimensional framework increases with a higher rate of substitution. The increased substitution percentage, up to 40%, increased the intensity of the Si–O–(Si, Al) asymmetric band, resulting in a shifting to lower frequencies. This referred to the development of amorphous aluminosilicate structure. Also, there is a clear shoulder in specimen E (80% grog) at about 1100 cm⁻¹ for this vibration band, which is believed to be formed due to a presence of unreacted silica in the Geopolymer matrix as ratio Si/Al pass the desirable limit 3: 3.90.

The appearance of bands in the regions of 1450 cm⁻¹(v C–O), and 870 cm⁻¹(δ C–O) are typical of CO₃²⁻ vibrational groups which are present in inorganic carbonates [23]. Moreover, there is an increase in intensity of formed inorganic carbonates with grog substitution of up to 20%, due to carbonation. This could lower the pH of the mix, together with the effect of excess free silica species in the Geopolymer mixes with Si/A ratio over 3.9. Thus, lowering the final compressive strength of these specimens.

It can be seen from Fig.5 for FTIR spectra of Geopolymer specimen C (40 % grog), at different curing ages, that the asymmetric stretching vibration (T-O-Si) increases in intensity, implying more Geopolymer content. Moreover, the symmetric stretching vibration band (Si-O-Si) shifts to a lower wave number with increased curing time. This can be attributed to the release of aluminum in the reaction system over time and possibly replacing some silicon ions which results in the formation of AlO⁴ tetraheda and Al-O-Si bonds. This is due to the Al-O bond vibrating at a lower wave number than the Si-O-Si bond, so it would be expected to shift stretching vibration band (Si-O-Si) to lower wave number as well [24].



Figure (4): FTIR spectra of 28 days cured (40 $^{\circ}$ C and 100% R.H.) Geopolymer specimens having various 40% slag as a partial replacement of grog. [1: Stretching vibration of O-H bond, 2: Bending vibrations of (HOH), 3: Stretching vibration of CO₂, 4: Asymmetric stretching vibration (T–O–Si), 5: Symmetric stretching vibration of CO₂, 6: Symmetric stretching vibration (Al–O–Si), 7: Symmetric stretching vibration (Si–O–Si), 8: Bending vibration (Si–O–Si and O–Si–O)].



Figure (5): FTIR spectra of Geopolymer specimen C cured at different ages (40 $^{\circ}$ C and 100% R.H.). [1: Stretching vibration of O-H bond, 2: Bending vibrations of (HOH), 3: Stretching vibration of CO₂, 4: Asymmetric stretching vibration (T–O–Si), 5: Symmetric stretching vibration of CO₂, 6: Symmetric stretching vibration (Al–O–Si), 7: Symmetric stretching vibration (Si–O–Si), 8: Bending vibration (Si–O–Si and O–Si–O)].

3.3. Compressive Strength

The compressive strength values of Geopolymer specimens containing different grog content from 0 up to 100 wt, %, as partial substitution of blast furnace slag at different curing times of 1, 7, 28, and 90 days at 40 °C with 100 % relative humidity are illustrated in Fig.6. It is obvious that the compressive strength of the slag Geopolymer specimen, without replacement, increases with curing time but decreases slightly by 4% on partial substitution with 20% grog but increases again by 7% on partial substitution of 40% grog.

Moreover, the effect of curing Geopolymer specimens for up to 90 days, with replacement of slag by grog up to 40%, was found to enhance the mechanical properties by about 30% more than if cured for 28 days. This can be explained by the increase in molar ratio of Si/Al up to 3.90, which generally increased the compressive strength of the Geopolymer mix. This implies a higher alumina and silica species in the Geopolymer system that are ready for Geopolymerization reaction. A similar result was encountered by **Steveson & Sagoe-Crentsil** [25]. They found that the Geopolymer specimens with Si/Al = 3.5 and 3.9 showed trends of increased strength with a higher silica concentration. This can be attributed to the filling effect, due to the dissolution of slag, grog and the formation of hydration products (CSH) as well as the Geopolymer gel filling the empty cavities which form compacted Geopolymer structures. This eventually leads to a higher compressive strength. Furthermore, the pore volume distribution of sodium activated geopolymer shift into smaller pores as Si/Al ratio increases [26].

However, with a higher replacement of slag, 60%, 80% and 100% grog with a Si/Al ratio of 4-4.29, the strength significantly decreased by 70% with 100% grog replacement. This can be explained due to the extent of raw material reaction decreased, as soluble silica content increased (higher Si/Al ratio), in the activating solution at a constant Na_2O/H_2O ratio, resulting from decrease in pH and an increase in solution viscosity [27].



Figure (6): Compressive strength of alkali activated Geopolymer brick specimens having various grog ratios as a partial replacement of slag.

As shown in Fig.7, the water absorption values of Geopolymer specimens A, B, and C, with 40% substitution of slag by grog, is nearly the same. This may be attributed to an increase in the degree of Geopolymerization and the formation of a new amorphous aluminosilicate gel phase resulting in a more dense structure. However, when slag is substituted by grog in the Geopolymer specimen's by more than 40% in D, E, and F mixes, the water absorption values start to increase. This can be related an increase in the Si/Al ratio and a decrease in the rate of Geopolymerization process, this in turn leads to less dense structure.



Figure(7): Water absorption of alkali activated Geopolymer brick specimens having various grog ratios as a partial replacement of slag.

3.4. Scanning electron microscopy (SEM)

The SEM micrographs of A, B, C, and E of the Geopolymer specimens are shown in Fig.8A, B, C and D respectively. The microstructure of specimens A & B (Fig. 8A&B) revealed the formation of aluminosilicate Geopolymer beside a small amount of dispersed CSH crystals. Furthermore, an increased rate of substitution, specimen C (Fig. 8C) the morphology of the formed Geopolymer paste is affected leading to the formation of condensed Geopolymer paste with fewer pores and increased amounts of small dispersed CSH crystals. Consequently, this will enhance the binding characteristics of Geopolymer specimen C. Additional substitutions, up to 80% grog, specimen E (Fig. 8D) clearly shows that there is an apparent negative impact on the amount of formed aluminosilicate geopolymeric material and CSH. Thus, it is notably reflected on the mechanical properties of the resulting Geopolymer product.



Figure (8): SEM micrographs of 28 days alkali activated slag Geopolymer specimens having various ratios of grog. A) 0% grog, B) 20% grog, C) 40 % grog, D) 60% grog.

4. Conclusion

This research has been performed with the general goal of synthesizing slag/grog Geopolymer brick as well as investigating its physical, mechanical and microstructural characteristics. The major findings of this work are summarized as follows:

- 1. It was found that partial replacement of slag by grog, up to 40%, in Geopolymer brick specimens with molar Si/Al ratio 3: 3.9 effectively increased compressive strength, enhanced microstructural properties and formed compact geopolymeric structures, as it increased the rate of Geopolymerization reaction.
- 2. Geopolymer brick specimens formed by replacement of slag by grog with a ratio of 40% to 100% and with molar Si/Al ratio > 3.9, resulted in a decline in the binding characteristic of Geopolymer and formed a less dense structure.
- 3. It was concluded that the Geopolymer brick specimens produced from the substitution of slag with grog up to 40% can compete with the fired clay brick both mechanically and physically, in yielding compressive strength values that exceeds 40 MPa, heavy duty bricks [28] which can be used in severe weather conditions ASTM- C62 [29].
- 4. The produced Geopolymer bricks possess more than 90% reduction in carbon dioxide as compared with other traditional clay bricks.

- 5. The effect of curing Geopolymer up to 90 days for specimens with replacement of slag by up to 40% grog was found to enhance the mechanical properties by approximately 30% than those cured for 28 days.
- The water absorption of all the produced Geopolymer bricks ranged between 11 to 16% lower than required for the severe weather clay bricks, according to ASTM-C62 [29].

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The authors have declared no conflict of interest and confirm that all experimental works done in this article under our responsibility and our own work that funded by the previously mentioned project and there is no conflict of interest within this work.

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