



Synergetic Effect of Ethylene Glycol for Production of Hybrid Sustainable Geopolymer Concrete

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Abstract

The world is facing the challenges of global warming and climate changes. Cement industry is responsible for some of the major CO₂ emissions, so it is vital to discover an alternatives to ordinary Portland cement (OPC). Geopolymer technology can offer a viable solution towards this target where 80% to 90% reduction in CO₂ emission can be achieved by the substituting of (OPC) with geopolymer. The aim of this study is the development of ground granulated blast furnace slag with metakaolin (GGBFS-MK) based geopolymer concrete and examination of the possibility of its usage in the building sector as long-term structural materials. Ethylene glycol (EG) was used to create hybrid organic and inorganic geopolymer composites. The results of the research revealed that GGBFS-MK based geopolymer can be developed, and its physical/mechanical properties were shown to have a very good performance after adding (EG) in different ratios 0.5, 1, 1.5, 2, 2.5 % from the total binding weight. According to varied experimental tests FTIR (Fourier transformer infra-red), XRD (X-ray diffraction), and SEM (scanning electron microscope), compressive strength and thermal resistance, the best ratio was found to be 0.5% (EG) which submits a good workability, was not easily affected by temperature changes and gives strength higher than control which gave (66.73, 69.11, 81.93) MPa while using 0.5% (EG) gave (81.12, 85.52, 116.75) MPa at 7, 28 and 90 days of curing respectively.

Keywords: Geopolymer, Ethylene glycol, Composites, Strength, Hybrid

1. Introduction

A large amount of carbon dioxide (CO₂) is generated during the calcinations process in cement production [1]. Its consumption has been estimated to cross 2.5 billion tons yearly and thus demand for raw materials needed for the production is also huge. It is also associated with emission of large quantity of carbon dioxide air pollutant and caused damages to environment [2, 3]. Hence, a small minimization of Portland cement production could minimize the carbon dioxide emission to a great extent and contribute to significant environmental benefits. In addition it helps in saving the raw materials resources from depletion [4]. There is a need for alternative binders to reduce carbon dioxide. In recent years, alkali activated materials have attracted much interest due to their environmental merits[5]. Geopolymers are alkali activated semi-crystalline aluminosilicates formed due to the reaction of aluminosilicates reach materials as kaolinite and industrial wastes as fly ash, slag in alkali media [6]. Geopolymer development is rapidly growing because of their advantages in recycling raw materials they are considered as "green materials",

they have high early strength [7], high resistance to fire as well as chemicals [8, 9] and UV radiation [10], they have low porosity, and hence low water permeability [11]. Despite these benefits, geopolymers have disadvantages that limit their use as structural materials. Their brittle mechanical nature, which results in low ductility and fracture toughness, is one of these disadvantages [12]. In order to improve these shortcomings of geopolymer materials, some additives are used such as organic polymers which are often incorporated in their structure [13]. Polyethylene glycol (PEG) is a non-toxic polyether molecule that can be employed in a variety of manufacturing processes. PEG is a plasticizer that increases lubricity while also acting as a water retention agent in ceramic mass, binders, adhesives and soldering fluxes with well spreading ability[13].

The influence of PEG on the mechanical strength of geopolymer composites was examined by **Catauroa et al.** [14], Because of the chemical composition of the samples, total strength increased with ageing time, and PEG-free samples reached final

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mechanical strength faster than hybrid samples. **Colangelo et al.** [15] Polymer-modified mortars have higher compressive strength than unmodified mortars, and the polymer aids to prevent microcrack spreading. They also concluded that when organic polymer is added, total porosity reduces, which may contribute to enhanced durability. **El Didamony et al.** [16] studied the effect of adding ethylene glycol (EG) to alkali activated slag (AAS) pastes on the physico-chemical and mechanical properties. According to the findings, the setting times of pastes elongate with the addition of EG. The drying shrinkage was minimized compared to control sample. With the addition of EG, the wet compressive strength dropped, on the other hand, the compressive strength of dried pastes at 80°C for 24 h increased with EG. **Amer et al.** [17] used ethylene glycol with alkali-activated slag (AAS) pastes and proved that the setting times of paste elongate with EG addition. The addition of EG to activated GBFS has a good effect on the compressive strength, while EG minimize the drying shrinkage for GBFS geopolymer. **Bilek Jr et al.** [18] added polyethylene glycol (PEG) and ethylene glycol (EG) as shrinkage-reducing admixtures (SRAs), and investigated the effects of using various contents of SRAs. According to their findings, adding SRAs to composites reduced drying shrinkage but hardly effected on compressive strength. This was related to a refinement of the pore size distribution and a reduction in surface tension [19, 20].

Khater & El Naggar [21] found that the addition of (EG) led to an enhancement in mechanical characteristics associated with the gain in compressive strength, flexural and tensile strength as comparison to control mix, better modification in microstructural properties has been proven. Nevertheless **Poornima Net al.** [22] set a Comparison between air and water cured geopolymer derived from GGBS-fly ash and Air curing of Ordinary Portland cement assisted by PEG mixed water. The experimental results showed an enhancement of Calcium silicate hydrate (CSH) gel with good permeability and improvement of strength for alkali mediated Ground granulated blast furnace slag (GGBFS) that partially blended with fly ash (FA) geopolymer mortars prepared under air, water curing.

Table 1: Chemical composition of raw materials

Materials	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	TiO ₂	L.O.I	Total
MK	61.1	31.4	1.51	0.32	0.11	0.12	0.14	0.13	2.44	2.33	99.6
GGBS	36.59	10.1	1.84	33.07	6.43	1.39	0.74	3.52	0.52	--	99.96

The target of the present paper is to study the effect of EG on the physico-mechanical properties of hybrid geopolymer pastes as well as study its effect on shrinkage. The paper also deals with the mineralogical investigation as well as microscopic imagery of the hardened pastes.

2. Experimental

2.1. Materials

The starting materials used in this study are metakaolin (MK) and Ground Granulated Blast Furnace Slag (GGBS) which are obtained from "Aluminum Sulfate Co. of Egypt (ASCE)" and "Iron and Steel factory, Helwan, Egypt" respectively. Ethylene glycol (EG) used at different dosage from 0.5 to 2.5% by mass of powder, EG with density 1.11 g / cm³ and purity 99 % was obtained from El Gomhouria Chemical Company, Egypt. Table 1 shows the chemical composition of MK and GGBFS used in this work. Figure (1) shows the XRD patterns of MK and GGBS. Liquid Sodium Silicate LSS (Na₂SiO₃) used in the present work received from Fisher company and has a density of 1.46 g/cm³, it was mixed proportionally at equal volume of (NaOH) solution as alkali activator. To prevent coagulation, superplasticizer was obtained from Sika Company and added in various ratios from the total weight of binder for better dispersion of the added compositions.

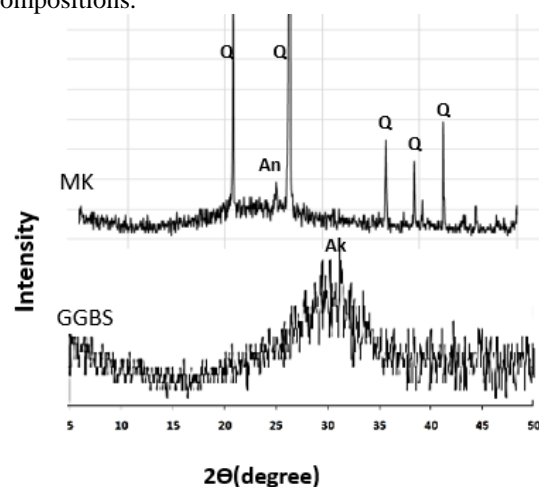


Fig.1: X-ray diffraction pattern of the starting raw materials (Q= Quartz, An= Anatase, AK= Akermanite)

Geopolymer mortars consisting of organic and inorganic mix, where the organic was ethylene glycol (EG), MK and GGBS are the inorganic compounds activated by using an equal volume (75ml) of sodium hydroxide (NaOH, 7M) and liquid sodium silicate LSS (Na_2SiO_3). EG was added at dosages of 0.5, 1, 1.5, 2, and 2.5 % by mass of powder. The related ratio based on the fact that viscosity will increase by EG increasing, so we should use small ratios, also related to findings of the previous work. Mixes were poured into 2.5 cm length cubic moulds, smoothed with a thin edged trowel, and cured at room temperature for 24 hours, the demoulded specimens cured at 40 °C with a 100% relative humidity (R.H.).

Specimens were taken out from their curing condition, dried at 80°C for 24 hours, and then tested for compressive strength, the crushed specimens being dried at 105°C to prevent further hydration, until the testing time, it kept in an airtight container. Compressive strength was carried out according to ASTM C109 M [23] at 7, 21, 28, 60 and 90 days. Table 2 shows the geopolymer mix design for the mortar.

2.3. Investigation Methods

Table 2: Mix Composition of Alkali Activated Pastes

Mix no.	MK (%)	GGBS (%)	EG (%)	LSS (%)	NaOH (7M) (%)	SP (%)	T.W/b (%)
E0	50	50	0.0	0.125	0.125	0.8	0.25
E1	50	50	0.5	0.125	0.125	1	0.25
E2	50	50	1	0.125	0.125	1.2	0.25
E3	50	50	1.5	0.12	0.12	1.4	0.24
E4	50	50	2	0.12	0.12	1.6	0.24
E5	50	50	2.5	0.12	0.12	1.8	0.24

3. Results and discussion

3.1 FTIR and XRD analysis

Figure (2) shows the FTIR spectra of geopolymer specimens with various EG ratios after 90 days. The figure depicts the progressive increase in hydration bands with ethylene glycol addition up to 0.5% (EG) as demonstrated by the stretching vibrations of O–H band at about 3455–3700 cm^{-1} as well as bending vibration of H–O–H bond at about 1630 cm^{-1} both bands usually correspond to present bond in the geopolymer hydration, i.e., corresponding to water molecules present in the material as well as CSH and NASH gel, This increase due to activation of geopolymer reaction by EG through increasing the polymerization rate leading to formation of amorphous NASH gels, where EG bind the microcrystals of inorganic materials and arrange their orientation to form natural composites with good properties [21]. With an increase in EG of up to 2%, the main asymmetric band decreases significantly, as a result of viscosity increasing by the added glycol monomer which hinders the

Starting materials investigated by using XRF-Axios (PW4400) WD-XRF Sequential Spectrometer-Netherlands, while mineralogical characterization was done by Philips PW 3050/60 diffractometer using a Cu-K α source. The infrared spectral analysis used for elucidation of the amorphous constituents of geopolymer composites using Jasco-6100–America, with the aid of KBr binder in the range from 400 to 4000 cm^{-1} . Mechanical testing was carried out using 5 tons German Brüf digital compression testing machine with a loading rate of 100 kg/min [24, 25]. The hydration products morphology were examined by scanning electron microscope (SEM) Inspect S (FEI Company, the Netherlands) equipped with an energy-dispersive X-ray analyzer (EDX). Drying shrinkage percent was calculated at ages of 28 and 90 days of curing. The thermo-mechanical properties tested for the composites by exposing them to high temperatures in the furnace. Each samples after cured at 28 d subjected to thermal treatment at 300°C, 500°C, 800°C at a sustained period of 2 h. firing shrinkage percent was calculated also the compressive strength tested after air annealing to RT and residual percent of the strength calculated.

geopolymerization reaction as reflected on the lower intensity of asymmetric band [16], the figure reflects the growth of the hydration bands as well as asymmetric vibration band with EG up to 0.5%, confirming the activation done by EG for geopolymerization reaction.

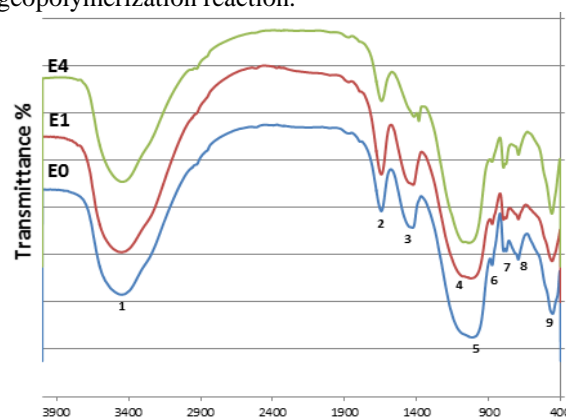


Fig. 2: FTIR spectra of alkali-activated composite with various ratios of EG at 90 days of curing.

(1:Stretching vibration of O–H bond, 2:bending vibration of (HOH), 3: stretching vibration of CO₂, 4: asymmetric stretching vibration (Si–O–Si), 5: asymmetric stretching vibration (T–O–Si), 6: symmetric stretching vibration of CO₂, 7: symmetric stretching vibration (Al–O–Si), 8: symmetric stretching vibration (Si–O–Si), 9: bending vibration (Si–O–Si and O–Si–O))

Figure (3) illustrates the XRD pattern of 90 days of cured alkali-activated GGBFS-MK composites treated with different ratios of EG. The pattern shows a rise between 20 and 35° in 2θ, which is a normal behavior in amorphous materials Related to geopolymer matrix [26]; this region increased with EG addition up to 2% relating to increase of geopolymerization reaction rate by the added glycol. XRD pattern reflects the growth of CSH with EG addition up to 0.5% while further increase in added monomers leads to an increased viscosity and so reflected negatively on the rate of geopolymerization and hydration reaction.

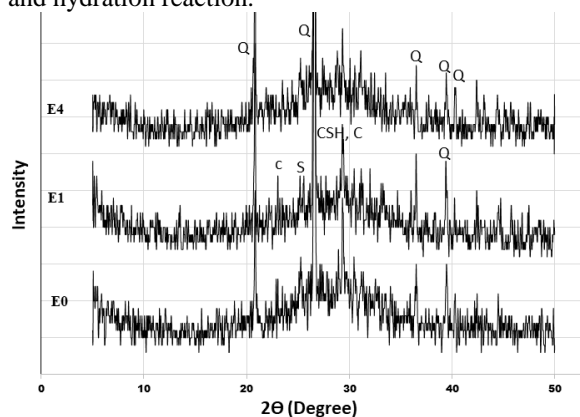


Fig. 3: XRD pattern of 90-day alkali- activated GGBFS-MK composite with various ratios of EG (Q= quartz, S= sodalite, CSH= Calcium silicate hydrate, C= calcite)

3.2 SEM microscopy

SEM analysis was performed on E0, E1 and E4 (0, 0.5, 2) % EG respectively after 90 days of curing figure (4). (Figure 4-A) shows that E0 has not compacted geopolymer composition and has some unreacted fragments, with EG addition continuous activation and formation of hydration products increased. While using 0.5 % EG ratio results in extra enhancement for geopolymerization reaction by forming additional organic polymer that densified the matrix (Figure 4-B). Further increase in the EG up to 2 % increased the viscosity, hence the porosity increased produces low compaction matrix leading to the weakening of formed composite (Figure 4-C).

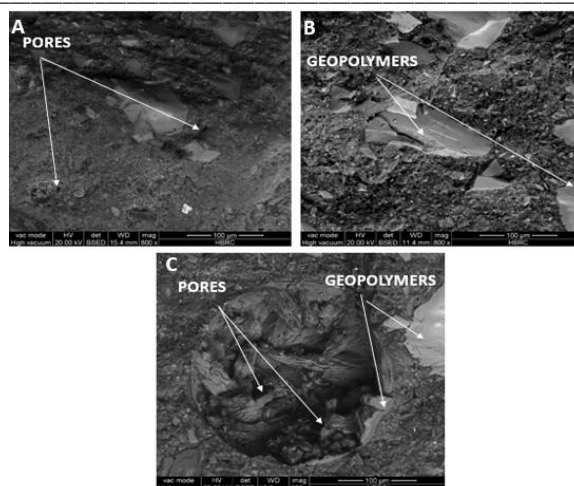


Fig. 4: SEM of 90 days alkali- activated GGBFS-MK composite containing A) 0 % B) 0.5% and C) 2 % EG.

3.3 Compressive strength

Figure (5) clearly indicates that the GGBFS- MK specimens treated with Ethylene Glycol and dried at 80°C for 24 hrs. have greater mechanical strength values than untreated ones, and there is a clear increase in mechanical strength values as curing time is increased. The dry compressive strength optimum value is for E1(0.5% EG)≈116.75 MPa at 90 days of curing, this connect to polymerization property of ethylene glycol (EG) which forming polyethylene glycol (PEG) filling the pores in the matrix as well as enforce formation of hydration products leading to compacted structure. Lower compressive strength values for more EG ratios up to 2.5% at all curing ages compared to E0 (0.0% EG) and E1 (0.5% EG) mixes. Increasing EG % means viscosity increase in the medium, which retards GGBFS-MK hydration, accordingly the dry compressive strength decreases. XRD and FTIR confirm the increased amorphous geopolymer as well as CSH with increasing EG to 0.5% then depressed to gradual decrease with further EG increase.

3.4 Drying Shrinkage

The drying shrinkage of alkali-activated composites is caused by the evaporated water lost to the environment [27]. The addition of shrinkage-reducing admixtures like EG effectively decreases the drying shrinkage of the formed composites and hardly influenced their compressive strength [18]. Figure (6) shows that the addition of ethylene glycol has a positive effect by reduction of drying shrinkage by approximately 40% and 30% for specimens cured at 28 and 90 days respectively. This shrinkage reduction after incorporating EG is associated with the refinement of the pore size distribution as well as

decrease of the surface tension leads to a built-in matrix. The best value is for incorporation of E1 (0.5% EG) this result compatible with the compressive strength results as well as XRD and FTIR investigations.

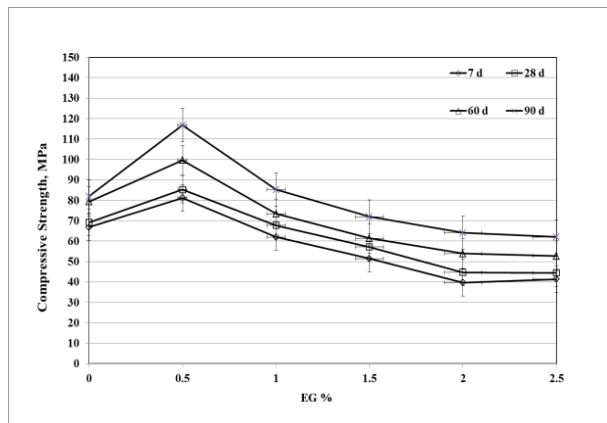


Fig.5: Compressive strength of GGBFS-MK composite with different EG ratios.

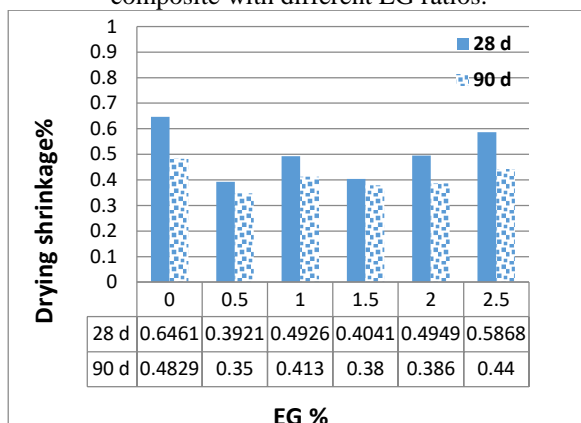


Fig.6: Drying Shrinkage of E0, E1, E2, E3, E4 and E5 mixes at 28 and 90 days of curing.

3.5 Thermal fire resistance

Thermal properties of geopolymers are superior to cement concrete [28] For Alkali activated GGBFS-MK geopolymer after 28 days of curing in 100% RH, certain number of cubes of some selected pastes were dried at 80°C for 24 hrs., and then fired at 300°C, 500°C and 800°C. The weight and shrinkage before and after firing was taken in addition to compressive strength as compared with that dried at 80°C at 28 days

3.5.1 Weight loss

The most significant geopolymers parameter weight loss for fire resistance evaluation is related to the moisture loss of free water and chemically bonded water after heating at temperature $\leq 300^\circ\text{C}$. From 300~500°C the observed weight loss corresponds to the dehydroxylation of Si-OH, Al-OH, and hydrating binding materials. The weight loss over 750°C is due to the decomposition of carbonate species [29, 30].

Figure (7) indicated that the weight loss % decreased by addition of ethylene glycol compared with control composite, this means that the EG addition leads to an enhancement of thermal stability of geopolymer composite. Which may be due to the formation of well-organized and condensed composites that mostly free of pores especially at lower dose of ethylene glycol. It can be also noticed the decreased weight loss ratio by increasing temperature up to 800°C as compared with low temperature.

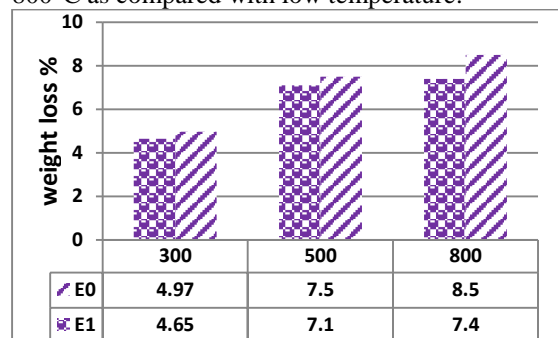


Fig. 7: Weight loss% for mixes E0 and E1 versus treatment temperatures

3.5.2 Firing shrinkage

Behaviour of Cracking and smashing up the geopolymer could be noticed on account of contracted pores, free water evaporated from the pores leading to cracks propagation resulting in shrinkage correspondingly. The appearance of a considerable shrinkage leading to cracks. In the temperature range of 100–300°C, early shrinkage occurs because of dewatering. Up to 500°C, the growth occurred continually, but at 800°C, there was a significant shrinkage. EG as additive can be incorporated with the geopolymer mix to enhance the resistance to thermal effect through minimizing shrinkage when specimens exposed to heating process. Average of firing shrinkage % can be calculated by the equation:

$$F.S \% = (DL-FL)/DL * 100$$

Where: DL for dried length and FL for fired length. Figure (8) showed less shrinkage value with EG treated specimen (E1) than control one (E0). This is clearly observed at all temperatures; by EG addition shrinkage decreased approximately by more than 70%, 50%, 30% at 300°C, 500°C and 800°C respectively. The decreased firing shrinkage of EG – geopolymer up to 500°C as compared with control mix favours high stability of this composite where temperatures between 300-500°C leads to activation of unreacted materials leading to incorporation in geopolymer formation and so forming dense homogenous structure. Increasing temperature to 800°C leads to the transformation into crystalline feldspars which have low contribution in strength as well as firing stability.

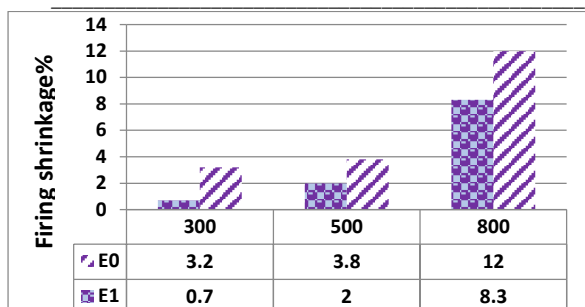


Fig. 8: Firing shrinkage of mixes E0 and E1 versus treatment temperatures

3.5.3 Strength Change Factor (SCF)

Geopolymers formed from rich aluminosilicate materials offer an enhancement of the geopolymers strength at room temperature, but sometimes not when subjected to high temperatures. The strength of geopolymer paste depends on two contradictory processes, the damage process because of the thermal effect and the process of further geopolymerization [31]. When the temperature is raised to 800°C, both mixes have low compressive strength. Loss of strength when temperature increased is because of chemically bound water in calcium-rich gels disappears, which ends to cavity structure and phase separation [33]. Average of SCF % can be calculated by the equation:

$$\text{SCF \%} = (S_0 - S_f) / S_0 * 100$$

Where: S_0 for strength after drying at 80°C and S_f strength after firing. From figure (9) we can notice that there is a strength gain at 300°C, 500°C, where the geopolymerization enhanced by the transformation of crystalline materials into amorphous one. These materials positively enhance geopolymer reaction. Also, the figure reflects the increased strength gain at 300 as compared with 500°C giving more vitreous component as well as dissolution of non-solubilized particles. At 800°C the composites tend to be crystalline most of their components transfer into feldspars which have low contribution to strength gain as well as geopolymer formation.

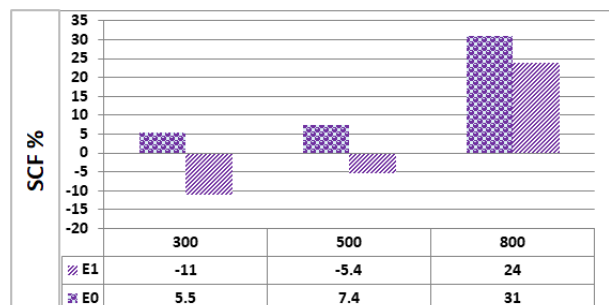


Fig. 9: Strength Change Factor (SCF) % for mixes E0 and E1 versus treatment temperature.

4. Conclusions

The key findings of the current study summarized in the following:

1. Addition of ethylene glycol monomer to GGBFS-MK geopolymer composites enhanced mechanical as well as morphological properties. The maximum compressive strengths were achieved by adding 0.5% of EG.
2. The FTIR, XRD revealed that amorphous structure was increased with EG up to 0.5%, hence highest strength with compacted and dense structure.
3. With regard to study of weight loss, firing shrinkage and compressive strength after exposing to high temperature 300,500 and 800°C, it was conducted that:
 - Weight loss % was slightly decreased by addition of ethylene glycol compared with control composite.
 - The firing shrinkage clearly increased after exposed to temperatures higher than 300°C and significant shrinkage observed at 800°C, addition of EG enhanced the thermal resistance through decreasing the firing shrinkage.
 - Compressive strength of geopolymer specimens increased with increasing firing temperature up to 500°C. but at 800°C there was a strength loss.
4. The great strength and thermal stability of geopolymer composites makes them suitable for use in a wide range of construction applications.

* Conflicts of interest

There are no conflicts to declare

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