

Impact of Ethylene Glycol Addition on the Physico-Chemical and Mechanical Properties of Alkali Activated GGBFS Pastes

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Abstract: This paper aims at study the effect of ethylene glycol (EG) addition on the physico-chemical and mechanical properties of alkali activated slag (AAS) pastes. Ground granulated blast-furnace slag (GGBFS) was activated by 6 wt., % sodium hydroxide (SH). 0, 2.5, 5, 7.5, and 10 wt., % of EG were added to AAS paste. The results show that, the setting times of AAS pastes elongate with the addition of EG. The drying shrinkage of AAS was reduced by ~ 7, 28, 53 and 68% in case of AAS containing 2.5, 5, 7.5 and 10 wt., % compared to control sample at 90 days. The chemically combined water contents increased with EG mass as, % at early ages of curing up to 7 days. The wet compressive strength decreased with the addition of EG. In contrast, the compressive strength of AAS pastes dried at 80°C for 24 h increased with EG percentage up to 7.5 wt., % . A significant reduction in compressive strength was observed in AAS containing 10 wt., % EG at all ages of curing. Dry AAS pastes containing 0, 2.5 and 5 wt., % showed a compressive strength regression at 90 days. The TG, DTG, FTIR and SEM proved that, the rate of polymerization of EG increases with time.

Keywords: AAS, EG, compressive strength, polymerization, GGBFS

1. Introduction

Alkali activated material has attracted great attention in recent years due to its excellent performances such as mechanical properties [1]–[3], durability [4]–[7], thermal stability [8],[9] and low environmental impacts [10]. It is usually produced by mixing alkaline activator solutions with solid raw materials. Alkali activated systems can be classified into two types: calcium and silica enriched (Ca + Si) system and aluminosilicates dominated (Si + Al) system [11]. The represented precursor of calcium enriched system is ground granulated blast furnace slag (GGBS), and the reaction product is a C-S-H (I) gel with a lower Ca/Si ratio and a high Al incorporation [12]. While the typical starting materials of Si + Al system is class F fly ash or metakaolin, having N-A-S-H type gels with three-dimensional network as the major final product [13]. Recently, there is a growing interest of mixing two different alkali activation systems together to form a blended Na₂O–CaO–Al₂O₃–SiO₂ system as several improved properties can result, including modified setting times [14], improved workability [15], reduced shrinkage [16], enhanced mechanical properties and durability [17]. In order to improve several properties of geopolymer materials, such as their brittle behavior and low flexural strength, which usually limit their extensive applications as a structural material, geopolymer composites have been introduced in the last years. Geopolymer based composites are usually obtained by incorporation of organic polymers [18]–[23], such as polyvinyl acetate, polypropylene, polyvinyl alcohol, or water-soluble organic polymers [24]–[28]. Recently [29], the authors have prepared novel organic-inorganic hybrid composites, adding epoxy resins to geopolymer, through an innovative synthetic approach based on a co-reticulation in mild conditions of organic and inorganic components, producing an intimate as well as homogeneous dispersion of the organic phase into the inorganic one. Through this novel synthetic approach, polymeric composites are not obtained by simply adding the organic polymer as fibers or emulsion as usually reported in

the literature, but the resin is allowed to crosslink in situ during the geopolymerization reaction. In this way, the use of compatibilizers is fully avoided [30]–[33]. These new materials, highly homogeneous up to micrometric level even at appreciable concentration of resin (up to 20% w/w), show good technological properties: in particular, in respect to the neat geopolymer, they present significantly enhanced compressive strength and toughness, suggesting their possible practical applications in the manufacture of high durable composites (e.g., thermo-resistant), in the field of restoration and repairing of damaged concrete and masonry. Another common problem of geopolymer based pastes representing an important barrier to their structural application is the high shrinkage during the curing process and the cracking of specimens caused by water loss under normal environmental conditions [34],[35]. These phenomena may be strongly reduced by adding superplasticizer [36],[37]. The effect of ethylene glycol addition on the physico-chemical and mechanical properties of AAS pastes was not studied until this time.

2. Materials and Methods of Investigation

2.1 Materials

The material used in this investigation are ground granulated blast furnace slag (GGBFS), sodium hydroxide (SH) and ethylene glycol (EG). GGBFS was provided by Iron and Steel Company, Helwan governorate, Egypt. SH was brought from SHIDO Company with purity 99%. EG with assay 99 % and density 1.11 g / cm³ was purchased from El Gomhouria Chemical Company, Egypt. The chemical composition of GGBFS is seen in Table (1). The XRD pattern of GGBFS is shown in Fig. (1). The XRD confirms the amorphous character of GGBFS. Fig. (2) shows the particle size distribution of GGBFS. It can be found from particle size distribution that GGBFS has about 90 % <50µm and 10% < 7µm.

2.1.1. Preparation of alkali activated slag-EG pastes

For the preparation of the paste samples, SH with 6 wt., % was dissolved in mixing water at water to binder (W/b) ratio of 0.24. After one day of SH solution preparation, 2.5, 5, 7.5 and 10 wt., % EG, respectively was added to solution of SH then added to GGBFS, and slow mixed for 5 minutes in the mixer. The mixer was stopped to collect any unmixed solids scraped from the sides of the mixing bowl and the paddle into the bowl. Then 2 more minutes of slow mixing and 5 minutes of fast mixing were applied to ensure homogeneity. For each mix, the freshly prepared cement paste was placed into one inch cubic mould and 25×25×285 prisms, then vibrated using vibration machine for one min. The surface was smoothed by a thin edged trowel. After moulding, the specimens were cured in 100 % relative humidity (R.H.) at room temperature, then demoulded and cured under tapwater for 90 days. The details of mixes are given in Table (2).

2.2. Methods of investigations

The initial and final setting times were both measured using the Vicat test according to ASTM C191[40]. The compressive strength of the specimens was measured after 1, 7, 28, 90 days according to ASTM C109M [38]. Shrinkage was determined according to ASTM C490 [39]. The length change of activated slag mortar with or without magnesium oxide at any age was calculated as follows:

$$L = (L_x - L_i) / G$$
 where: L = change in length at x age, %, L_x = comparator reading of specimen at x age minus comparator reading of reference bar at x age, L_i = initial comparator reading of specimen minus comparator reading of reference bar at the same time. G = nominal gauge length, 285 mm.

Thermogravimetric analysis (TGA) were carried out by heating the sample in nitrogen atmosphere up to 1000 °C with a heating rate of 20 °C/min using a DT-50 Thermal Analyzer (Schimadzu Co-Kyoto, Japan). Infrared spectral analysis was carried out on some selected samples of hydrated paste to provide additional information on the hydrated products. The infrared spectral analysis was recorded from KBr discussing Genesis FTIR spectrometer in the range of 400-4000 cm⁻¹. The scanning electron microphotographs were taken with Inspect S (FEI Company, Holland) equipped with an energy dispersive X-ray analyzer (EDX) to examine the microstructure of the fractured composites at the accelerating voltage of 200 V to 30 KV and Power zoom magnification up to 300000 xs.

3. Results and Discussion

3.1. Setting Times

Fig. (3) shows the initial and final setting times (IS and FS) of AAS pastes containing 0, 2.5, 5, 7.5 and 10 wt., % EG. It is clear that, the initial and final setting times were slightly elongated up to 5 wt., % then sharply elongated up to 10 wt., % EG. This may be attributed to that, the EG retards the activation of slag hydration, which caused by its reaction with sodium hydroxide.

3.2. Drying Shrinkage

Fig. (4) shows the drying shrinkage of AAS pastes with or without EG. It is important to note that, the addition of 2.5, 5, 7.5 and 10 wt., % EG reduces the drying shrinkage of AAS pastes by ~ 7, 28, 53 and 68% after 90 days of curing in 50% RH. This is attributed to that, EG reduces the evaporation of water from the surface of AAS paste and also helps in water retention [37]. In other word, this could probably be explained by the hydrophilic colloidal properties and the inhibited water evaporation due to the filling and sealing effects of impermeable polymer films [43].

3.3. Chemically Combined Water Contents

Fig. (5) illustrates the chemically combined water (Wn., %) contents of AAS with or without EG. The results show that, the Wn., % content of AAS with no EG (M0) increases with curing time, indicating that the continuous activation of GGBFS hydration and formation of hydration products with time. The results also show that, the Wn., % increases with the amount of EG at early ages of hydration up to 7 days. This fact may be explained by the presence of unpolymerized EG in the AAS system. On the other hand, the Wn., % content of AAS pastes containing 5, 7.5 and 10 wt., % EG (M5, M7.5 and M10) decreases with time up to 28 days followed by an increase at 90 days. These may be related to that, the continuous polymerization of EG monomer forming poly ethylene glycol (PEG) with low OH groups that leads to the decrease of Wn., %. After 28 days of hydration the amount of CSH increases, which responsible for the increase the Wn., %. The Wn., content of AAS containing 2.5 wt., % EG (M2.5) decreases with time up to 7 days then increases at later ages of hydration from 28 up to 90 days, demonstrating that the low amount of EG is completely polymerized with the first 7 days. The M5 mix shows highest Wn., % at later ages of hydration, suggesting the formation of high amount of CSH in conjunction with PEG. Conversely, M10 mix shows the lowest Wn., % at later ages of curing. This fact proves that, the high amount of EG has retardation effect for the activation of GGBFS hydration. Thereby, the amount of hydration products in GGBFS, which responsible for the increase in the Wn., content, decreases. At 90 days of curing, there is no a great difference in the Wn., % between all mixes.

3.4. Wet Compressive Strength

Fig. (6) represents the compressive strength of M0, M2.5, M5, M7.5 and M10 mixes. It is obvious that, the compressive strength of all hardened pastes increases with time up to 90 days. This is attributed to that, the continuous activation of GGBFS hydration and accumulation of more hydration products. Also, the compressive strength decreases with the amount of EG, especially at early ages of hydration. This fact is due to that, the viscosity of the medium increases with EG content, which in turn affects the activation of GGBFS hydration. At later ages of hydration up to 90 days the EG begins to polymerized in AAS matrix causing a significant increase in compressive strength, especially for M2.5 and M5 mixes. The M10 mix shows the lowest compressive strength at all ages of hydration, due to the high

retardation effect of EG. The addition of 2.5-5 wt., % EG gives the higher wet compressive strength at 90 days. Therefore these values are sufficient to improve the wet compressive strength of hardened AAS pastes.

3.5. Dry Compressive Strength

Fig. (7) shows the compressive strength values of hardened pastes made of GGBFS-EG dried at 80°C for 24 h. It is shown that, the dry compressive strength M2.5 mix is higher than that of all mixes at 1 day of curing. This is related to that, the EG is polymerized to form polyethylene glycol (PEG) in open pores, which affecting positively in the compressive strength. The M5, M7.5 and M10 mixes show the lower compressive strength compared to M0 and M2.5 mixes. A significant increase in compressive strength was observed in case of M2.5, M5 and M7.5 mixes, especially at 7 and 28 days. This fact may be due to that, the drying accelerates the polymerization rate of EG and activation of GGBFS hydration forming more PEG and hydration products leading to increase the compressive strength. AAS containing 10 wt., % of EG shows the lower compressive strength at all ages of curing. This reverse to that, the addition of 10 wt. % of EG leads to increase the viscosity of the medium, which retards GGBFS hydration, and therefore the dry compressive strength decreases. A significant regression in the compressive strength was observed in M0, M2.5 and M5 mixes at 90 days of curing. This may be due to that, centering of hydration products caused by the increase in the degree of crystallinity. This regression in compressive strength cannot be observed in case of M7.5 and M10 mixes. This may be attributed to that, the addition of 7.5 and 10 wt. % of EG increases the viscosity of the medium, and therefore, prevents the crystallization of hydration products.

3.6. FTIR spectroscopy

Fig. (8) represents the FTIR- spectra of AAS (M0) at 1, 28 and 90 days of curing. The absorption bands at 453-458 cm⁻¹ are related to O-Si-O bonds bending vibration, absorption bands from 669-694 cm⁻¹ are due to symmetric stretching vibrations of the Si-O-Al bridges. The more intense absorption bands at 959-967 cm⁻¹ are due to asymmetric stretching vibration of Si-O-T (where T is Si or Al). Absorption bands from 1464-1490 cm⁻¹ are due to calcium carbonate or hydrotalcite (Ht)-like phases. Absorption bands from 1642-1649 cm⁻¹ are related to bending H-O-H vibration, absorption bands from 3446-3474 cm⁻¹ are assigned as stretching vibration of O-H groups. Absorption bands of water are indication to crystalline H₂O of the hydrated products such as C-S-H and C-A-H [41]. It is clear that, the intensity of absorption band related to asymmetric stretching vibration of Si-O-T increases with curing time from 1 up to 90 days. This should be related to the continuous activation and formation of hydration products. Also, the high full width at half maximum (FWHM) of this band decreases with curing time, which is due to the increase of the crystallinity of the samples caused by ordering of the structure or increase the substitution of Si by Al in silicate networks [42]. The band intensity of bending vibration H-O-H and OH group stretching vibration

increases with curing time. This may be related to that the amount of hydration products increases with curing time.

Fig. (9) shows the FTIR- spectra of AAS pastes containing 5 wt., % of EG (M5) at 1, 28 and 90 days of curing. Obviously, the FWHM of a symmetric stretching vibration of absorption band corresponds to Si-O-T at 958-968 cm⁻¹ decreases with curing time. This proves that the crystallinity degree, which caused by ordering of the structure of CSH or CASH, increases with curing time. The intensity of absorption bands, related to bending H-O-H vibration at 1649-1655 cm⁻¹ and stretching vibration of OH group at 3436-3449 cm⁻¹, of M5 mix at 1 day of curing is higher than those at 28 and 90 days. This may be related to unpolymerized EG that presents in GBFS matrix at 1 days of curing. As the curing time increases the polymerization rate of EG enhances causing the decrease in OH groups and, therefore, the intensity of these bands decreases. The intensity of these bands at 90 days is higher than those at 28 days. This may be due to the increase of hydration products with time.

3.7. Thermogravimetric analysis (TGA/DTG)

Fig. 10 (a, b) illustrates TG/DTG of M0 mix at 1, 28 and 90 days. The weight loss corresponding to the decomposition of hydration products was determined from the TG plots. The weight loss at 1000 °C was found to be 7.45 %, 9.17 and 10.23 % for 1, 28 and 90 days of hydration. The peak of each hydrated product was determined by DTG. Endothermic peak at 50-200 °C is due to dehydration of free water and C-S-H. The endothermic peak at 200-400 °C is characteristic for Ht. Other peak observed at 400-600 °C is related to the decomposition of calcium hydroxide (CH). The decomposition of carbonate groups was observed in the range of 600-800°C. Generally, the weight loss activated GGBFS sample increases with time up to 90 days as shown in TG curves (Fig. 10a), suggesting the continuous activation of GBFS hydration and accumulation of hydration products as seen in (Fig. 10b). The peak of CH observed in activated samples cured at 1 day is disappeared at later ages of curing. This may be related to that the CH is mainly consumed as a result of the reaction with aluminate and silicate species in the matrix forming CSH and CASH.

Fig. 11 (a, b) shows TG/DTG of M5 mix after 1, 28 and 90 days. The weight loss of M5 mix (Fig. 11a) at 1 day is greater than that of at 28 and 90 days of curing. This fact must be explained by the presence of unpolymerized EG that can be observed from the increase in peak intensity around 50-200 °C. The weight loss of this sample cured at 90 days is greater than that of at 28 days of hydration, due to the increase of hydration products with curing time.

3.8. SEM microscopy

Fig.(12). represents the SEM of AAS containing 0 (M0) and 5 wt., % EG (M5) at 1 and 90 days of curing. Generally, the SEM micrographs of each mix at 90 days are denser and more compact microstructure than those of at 1 day. This confirms the continuous activation and formation of hydration products with time. The SEM of M5 mix at 1 day of curing has phases with fibrous morphological shapes

present in pores and on the surface of slag grains; this is related to low polymerized EG. These phases cannot be observed at later ages of curing, due to the formation of high amount of hydration products, which cover PEG.

4. Conclusion

Several findings can be concluded as follows:

- 1) The addition of EG leads to elongate setting times of AAS pastes as well as reduce the drying shrinkage.
- 2) The chemically combined water content increases with EG wt., % at early ages of hydration. AAS containing 5 wt., % EG shows the highest chemically combined water content after 90 days of hydration. Conversely, the addition of 10 wt., % leads to decrease the amount of chemically combined water at 90 days as compared to the control sample.
- 3) The wet compressive strength decrease with EG content from 1 up to 28 days. The compressive strength of M2.5 and M5 mixes was higher than that of the M0 at 90 days of curing. The dry compressive strength increase with EG content up to 7.5 wt., % then decreases with addition of up to 10 wt., %.
- 4) The results of combined water is in a good harmony with TG/DTG and FTIR.
- 5) The SEM proved that the fibrous morphological shape of PEG appeared in open pores and on the surface of slag grains at 1 day of hydration. While at later ages of hydration the PEG cannot be observed.

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Table 1: Chemical compositions of GGBFS

Oxide, %	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	TiO ₂	P ₂ O ₅	L.O.I	Total
GGBFS	37.81	13.14	0.23	38.70	7.11	1.03	0.19	1.19	0.40	0.17	-	99.97

Table 2: Mix Composition of Alkali Activated Slag Cement Pastes

Mix. symbol.	GGBFS, wt. %	SH, wt. %	W/C ratio
M0	100	6	0.24
M2.5	100	6	0.24
M5	100	6	0.24
M7.5	100	6	0.24
M10	100	6	0.24

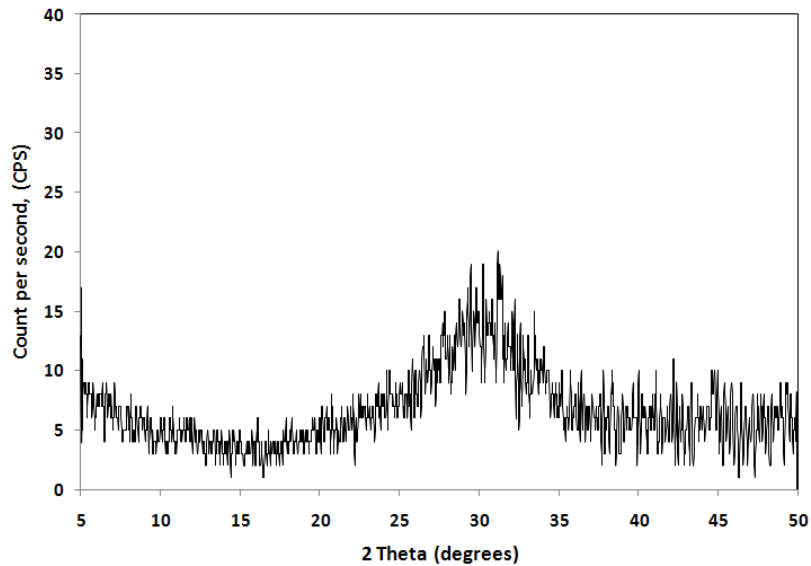


Figure 1: XRD pattern of GGBFS

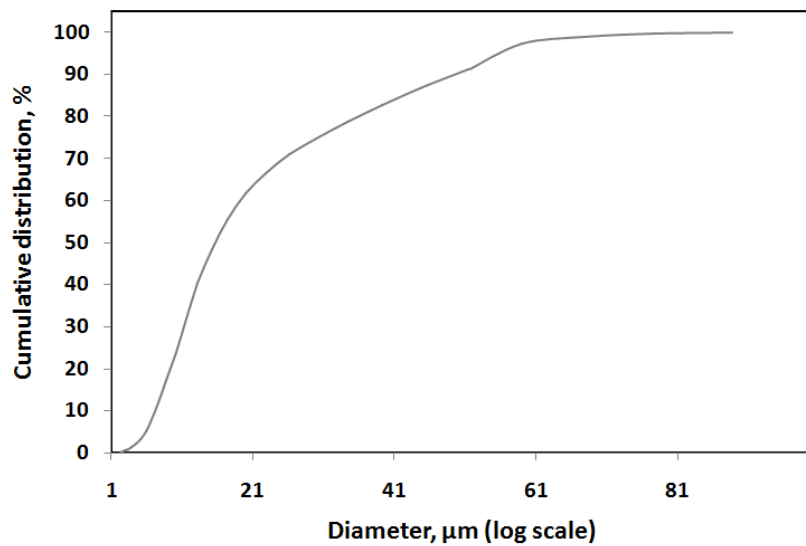


Figure 2: The particle size distribution of GGBFS

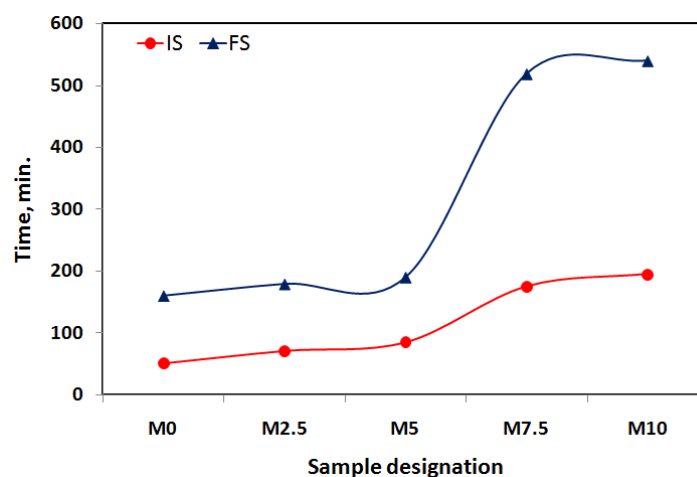


Figure 3: Setting times of AAS pastes containing 0, 2.5, 5, 7.5 and 10 wt., % EG

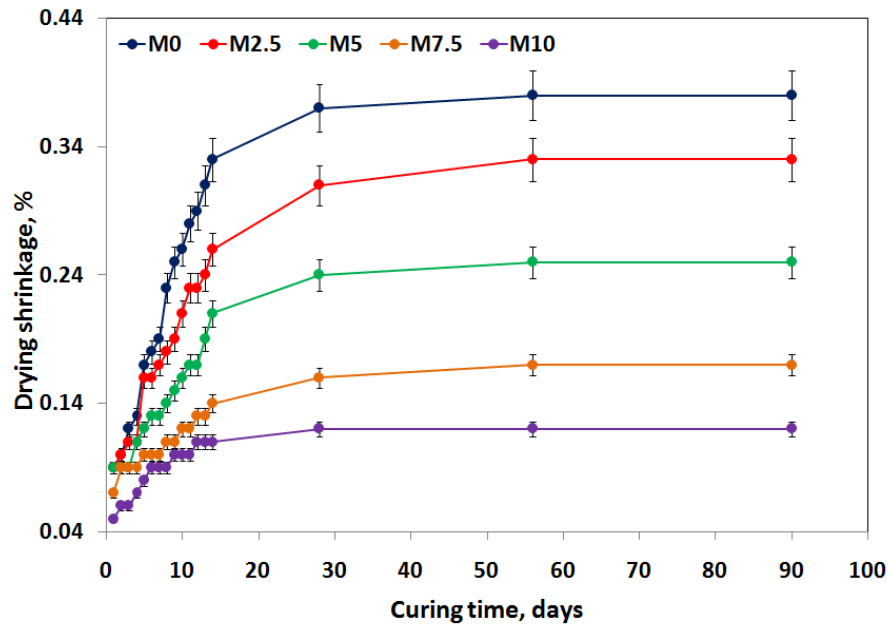


Figure 4: Drying shrinkage of alkali activated GGBFS with or without EG

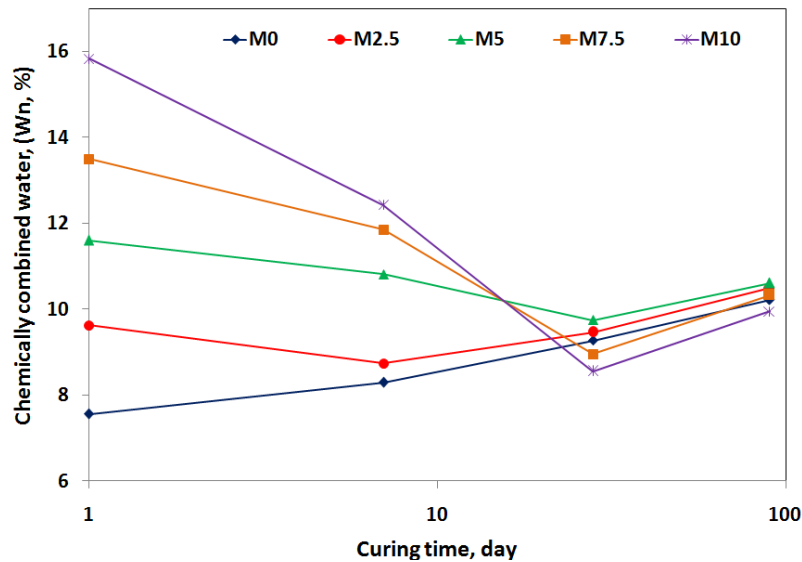


Figure 5: Chemically combined water (Wn, %) contents of AAS with or without EG

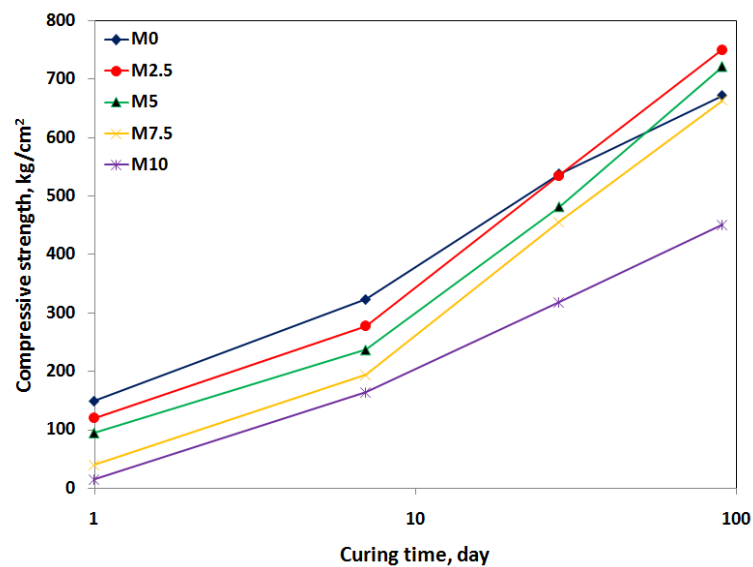


Figure 6: Wet compressive strength of M0, M2.5, M5, M7.5 and M10 mixes

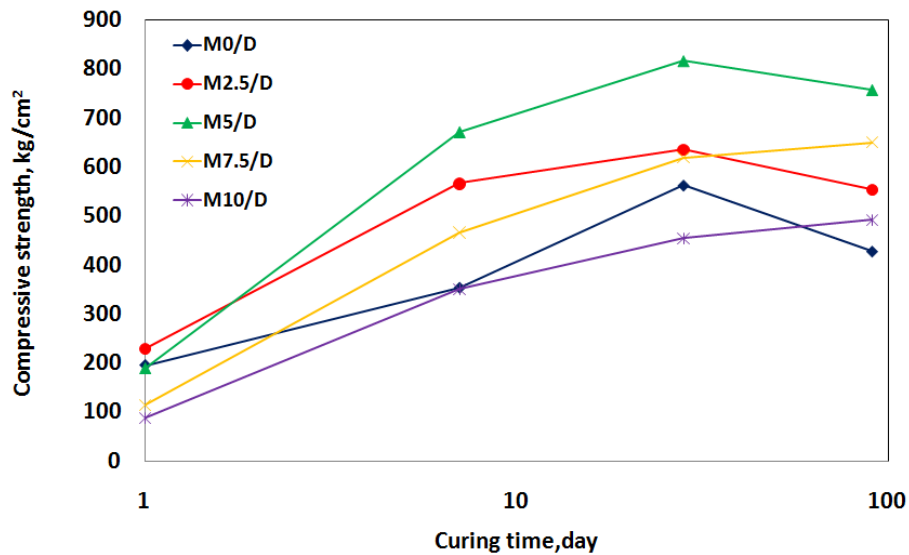


Figure 7: Dry compressive strength of M0, M2.5, M5, M7.5 and M10 mixes

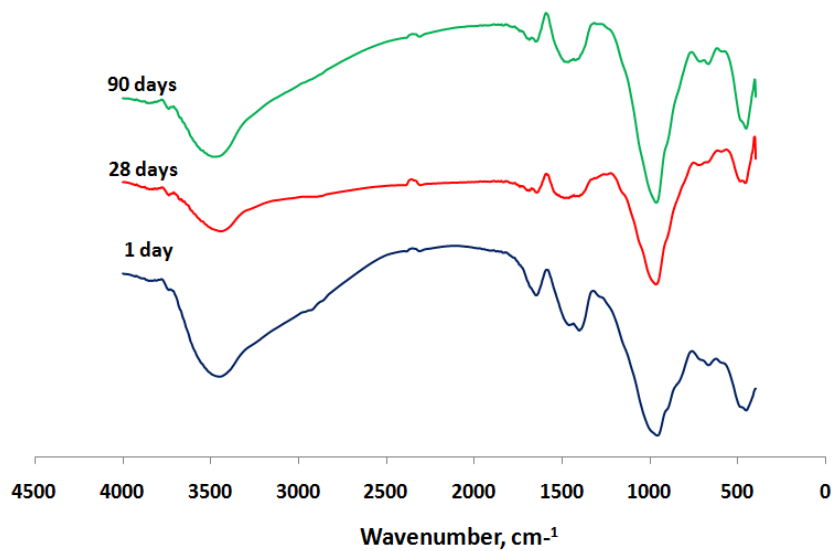


Figure 8: FTIR- spectra of AAS (M0) at 1, 28 and 90 days of curing

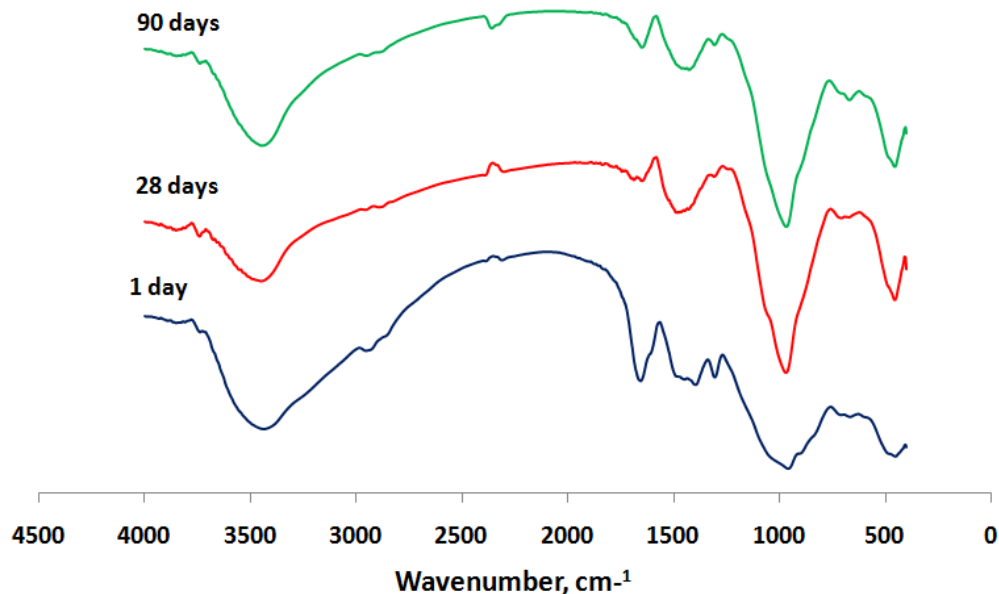


Figure 9: FTIR- spectra of AAS pastes containing 5 wt. % EG (M5) at 1, 28 and 90 days of curing

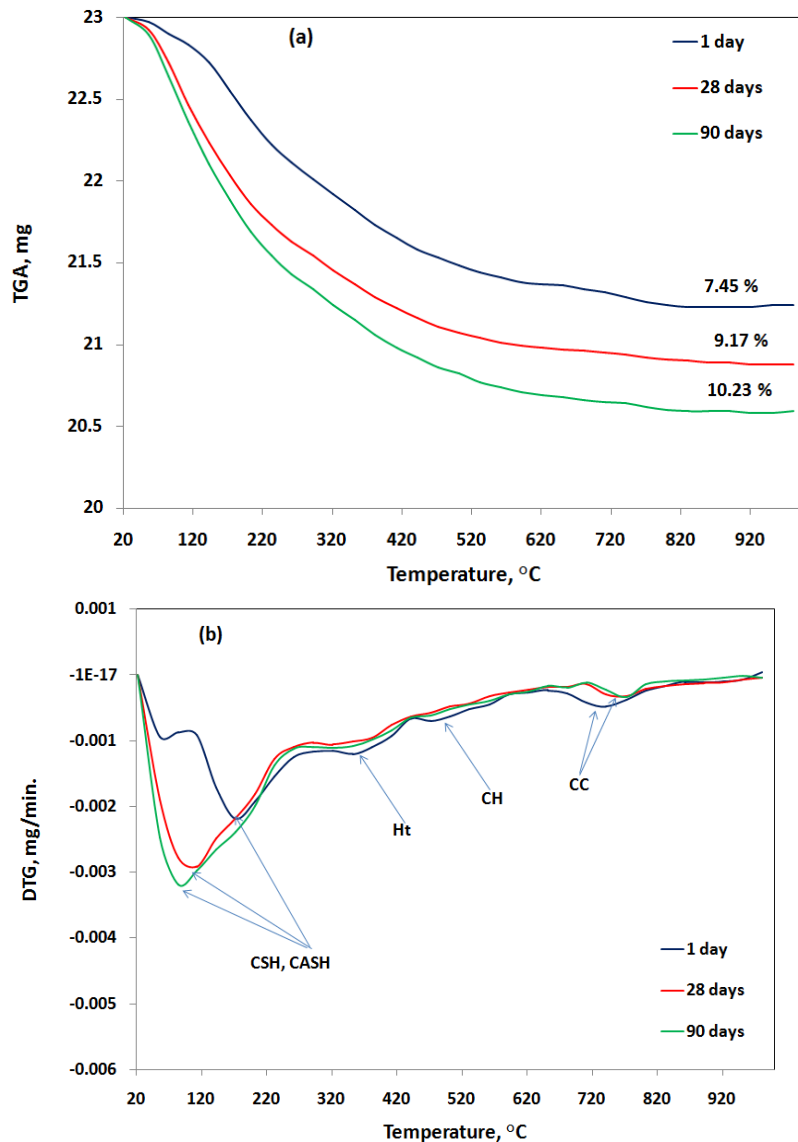
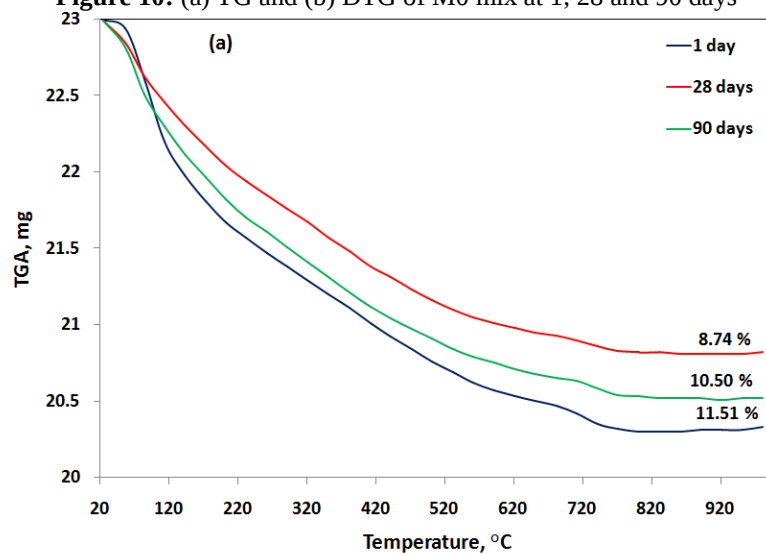


Figure 10: (a) TG and (b) DTG of M0 mix at 1, 28 and 90 days



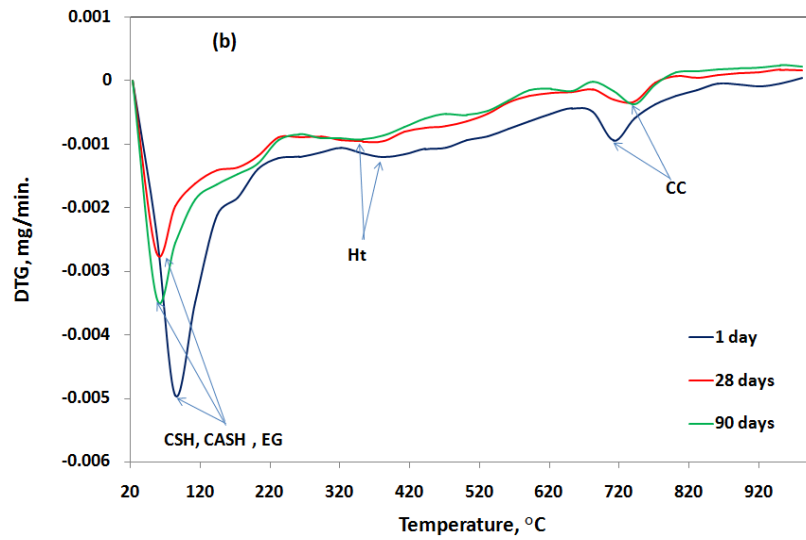


Figure 11: (a) TG and (b) DTG of M5 mix after 1, 28 and 90 days

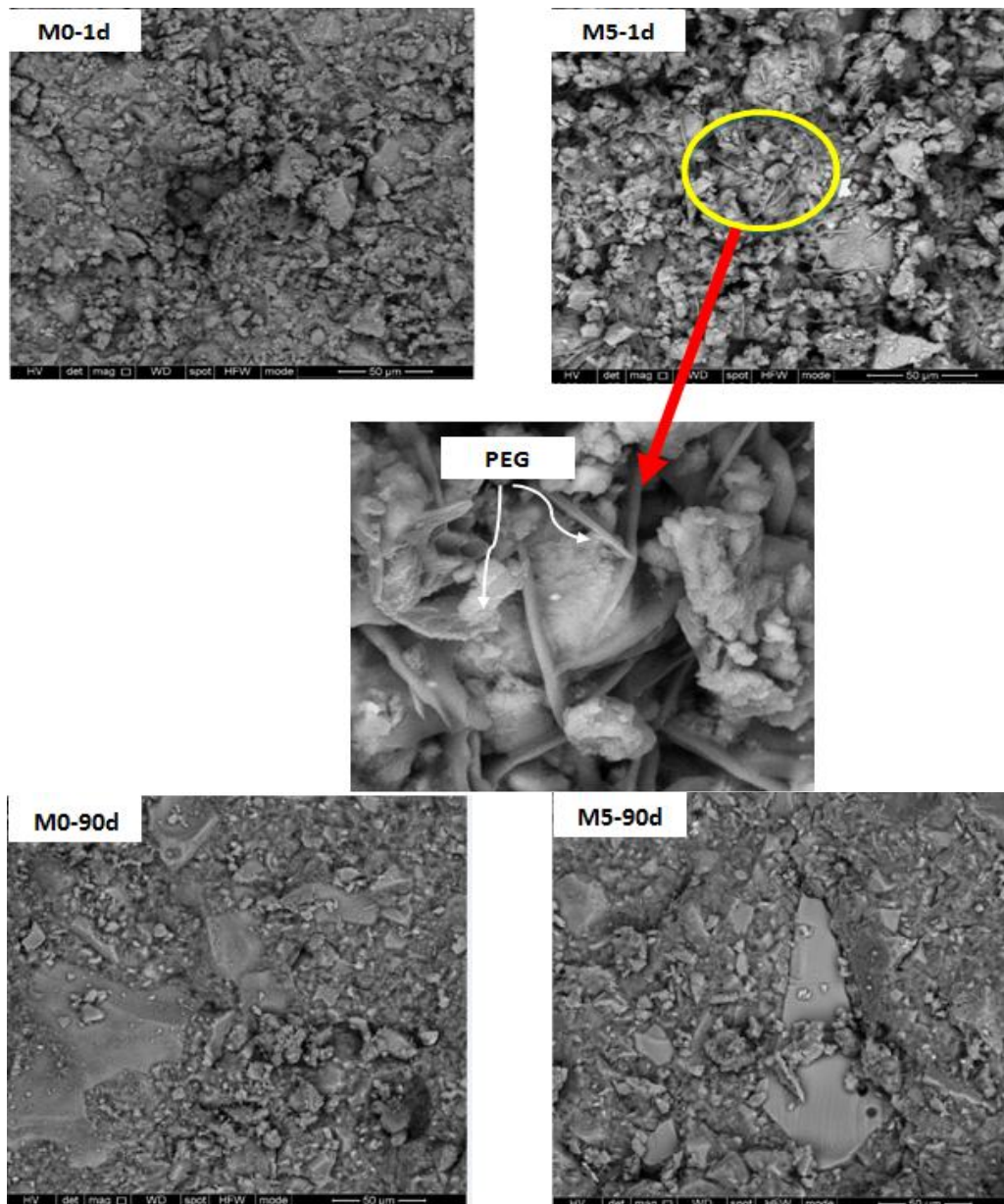


Figure 12: SEM of AAS containing 0 (M0) and 5 wt., % EG (M5) at 1 and 90 days of curing