Alkali activated ceramic waste with or without two different calcium sources

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Abstract. The aim of this investigation is to prepare geopolymer resin by alkali activation of ceramic waste (AACW) with different sodium hydroxide (NaOH) and liquid sodium silicate (LSS) concentrations. In order to prepare geopolymer cement, AACW was replaced by 10 and 30 % by weight (wt.,) of concrete waste (CoW) as well as 10 and 30 wt., % ground granulated blast-furnace slag (GGBFS). The results showed that, the compressive strength of AACW increases with the increase of activator content up to 15:15 wt., % NaOH: LSS. All AACW hardened specimens activated by 3:3 (MC6), 6:6 (MC12), 12:12 (MC24) and 15:15 wt., % (MC30) NaOH: LSS destroyed when cured in water for 24h. The MC18 mix showed higher resistivity to water curing. The results also showed that, the replacement of AACW containing 9:9 wt., % NaOH: LSS (MC18) by 10 (MCCo10) and 30 (MCCo30) wt., % CoW decreased the compressive strength at all ages of curing. In contrast, the MCCo10 mix showed the lower chemically combined water content compared to MC18 mix. The MCCo30 mix showed the higher chemically combined water content compared to MC18 and MCCo10 mixes. The compressive strength and chemically combined water of all AACW mixes containing GGBFS (MCS10 and MCS30) were higher than those of AACW with no GGBFS (MC18). As the amount of GGBFS content increases the chemically combined water increases. The x-ray diffraction (XRD) proved that as the amount of CoW content increases, the degree of crystallinity increases. Conversely, the replacement of AACW by GGBFS leads to increase the amorphiticity character. The infrared spectroscopy (FTIR) confirms the higher reactivity of GGBFS compared to CoW as a result of successive hydration products formation, enhancing the compaction of microstructure as observed in scanning electron microscopy (SEM).

Keywords: alkali activated ceramic waste; calcium sources; geopolymer cement; geopolymer resin

1. Introduction

Ordinary Portland cement (OPC) results from calcination of calcite and aluminosilicate materials up to 1450°C. The production of 1 tonne cement directly generates 0.55 tones of chemical CO_2 and 0.4 tonnes of CO_2 resulted from carbon fuel combustion, to become 1 tonne of

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cement generates 0.9 tones of CO_2 (Davidovits 2011, Puertas *et al.* 2008). Therefore, cement industry is considered one of the most industries contributes environmental pollution. The cement industry, together with the scientific community, is actively seeking alternatives to reduce the energy involved in cement production, to produce cement with low CO_2 emission and to reuse waste materials from other industries. Geopolymer cement is considered one of these alternatives, which requires low energy in its manufacture without minimum level of CO_2 emission. Where, one tone of geopolymer cement (prepared from alkali activation of geological rock, metakaolin (MK) and ground granulated blast-furnace slag (GGBFS) generates 0.184 tone of CO_2.

Later in 1972, Joseph Davidovits coined the name ''geopolymers'' (Davidovits 1999) to describe the zeolite like polymers. Geopolymers are the alumino-silicates polymers which consist of amorphous and three dimensional structures formed from the geopolymerization of alumino-silicates monomers in alkaline solution (Rowles and O'Connor 2003). Several investigations have been carried out on calcined clays (e.g., metakaolin (Zhang *et al.* 2010, Murat 1983, Cioffi *et al.* 2003, Wang *et al.* 2005, Duxson *et al.* 2007, Rashad 2013)) or industrial wastes (e.g., fly ash (Diaz *et al.* 2010, Kong and Sanjayan 2010, Guo *et al.* 2010, Temuujin and van Riessen 2009, Temuujin *et al.* 2010, Wang *et al.* 2015, Marjanović *et al.* 2015)) or metallurgical slag (Zhang et *al.* 2007, Chang 2003, El-Didamony 2013, Abdel Gawwadand Abd El-Aleem 2015, Abdel Gawwad *et al.* 2015, Christina *et al.* 2008).

Davidovits (2011) stated that, there are two types of geopolymer: geopolymer resin and geopolymer cement.From terminological point of view, geopolymer cement is a calcium based geopolymer binding system that hardens at room temperature. Meanwhile, resin is a binder that can not be set at room temperature but requires heat due to it dose not contain calcium ion. The effect of calcium ion source on the physico-chemical and mechanical properties of geopolymer resin was investigated by fewer research workers (Kumar *et al.* 2010, Phoo-ngernkham *et al.* 2015, Kabir *et al.* 2015, Bignozzi *et al.* 2014).

Ceramic and red clay brick wastes (CW and RCBW) are considered good alumino silicate materials, which can be used as alternative to MK and FA. Alkali activated CW and RCBW for the production of geopolymer resin was investigated by few researchers (Reig *et al.* 2013, El-Fadaly*et al.* 2014). Allahverdi and Kani (2009) examined the effect of concrete waste on the properties of activated RCBW. According 28 days compressive strength, they concluded that the RCBW is more suitable than CoW for geopolymerization process.

In this study, CW was activated by different sodium hydroxide and liquid sodium silicate contents to evaluate the optimum percentage of activator which gives geopolymer resin with higher mechanical properties and resistivity to water curing. In order to prepare geopolymer cement, the CW mix with optimum activator content was replaced by 10 and 30 wt., % concrete waste (CoW) as well as 10 and 30 wt., % GGBFS as two different calcium sources.

2. Materials and experimental methods

2.1 Materials

The materials used in this investigation are ceramic waste (CW), ground granulated blast-furnace slag (GGBFS), and concrete waste (CoW). GGBFS was provided by Helwan Steel Company, Helwan, Egypt. The activators used in this study were sodium hydroxide and liquid sodium silicate (NaOH and Na2SiO3). NaOH with purity 99.99 % was purchased from fisher

Table 1 Chemical compositions CW, GGBFS and CoW, wt.,%

Oxides	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$_5$	MnO	BaO	L.O.I	Total
CW	68.30	19.50	5.29	1.57	0.26	1.08	1.88	0.06	0.90	0.29	-	-	0.72	99.99
GGBFS	38.70	11.90	0.51	32.90	4.08	0.68	0.78	2.42	0.49	-	3.92	3.29	-	99.97
CoW	30.63	1.79	1.59	37.45	3.87	0.02	0.24	1.07	0.15	0.06	-	-	23.10	99.98

scientific chemical company, United Kingdom (UK). Liquid sodium silicate (LSS) with 17 % Na2O, 32 % SiO2 and density of 1.46 was obtained from El Gomhourya Chemical Company, Egypt. The chemical compositions of CW, CoW and GGBFS as determined by X-ray fluorescence (XRF) were listed in Table 1. The mix composition of CW, GGBFS and CoW, were given in Table 2. The mineralogical compositions of these materials are shown in XRD patterns (Fig. 1). The mineralogical analysis shows that, CW exhibits different crystalline peaks related to quartz (Q) and anorthite (A) minerals. GGBFS is completely amorphous with a hump in the range of 20-40 2θ. The XRD of The crystalline peaks observed in XRD pattern of CoW are characteristic for quartz, calcite and dolomite.

Table 2 Mix compositions of AACW, AACW / CoW and AACW / GGBFS blends, wt., %

Mix No	Mix Name	GGBFS	CW	CoW	NaOH:Na$_2$SiO$_3$
1	MC6	-	100	-	3:3
2	MC12	-	100	-	6:6
3	MC18	-	100	-	9:9
4	MC24	-	100	-	12:12
5	MC30	-	100	-	15:15
6	MCCo10	-	90	10	9:9
7	MCCo30	-	70	30	9:9
8	MCS10	10	90	-	9:9
9	MCS30	30	70	-	9:9

2.2 Preparation of alkali activated CW, CW/CoW or CW/GGBFS blends

Firstly, CW and GGBFS were crushed in automatic grinding machine to reach a fixed particle size passes from 90 µm sieve. In order to prepare geopolymer resin, CW was activated by 3:3, 6:6, 9:9, 12:12 and 15:15 NaOH:Na2SiO3 weight ratio. Geopolymer cement was prepared by the replacement of CW, activated with 9:9 NaOH: LSS, by 10 and 30 % CoW as well as 10 and 30 wt., % GGBFS. CW, CW/CoW or CW/GGBFS dry mixes were placed on smooth, non absorbent surface. The mixing water containing activator for all mixes was adjusted at 0.31 of water to solid ratio (W/S). The mixing operation was then completed by slowly mixing for about 2 min then continuous and vigorous mixing by means of ordinary gauging trowel for about three minutes, then cast into the cubic (25×25×25 mm) moulds in two layers and manually-vibrated to eliminate air voids. The samples were then covered to avoid moisture loss. Geopolymer resin samples were kept in an oven at
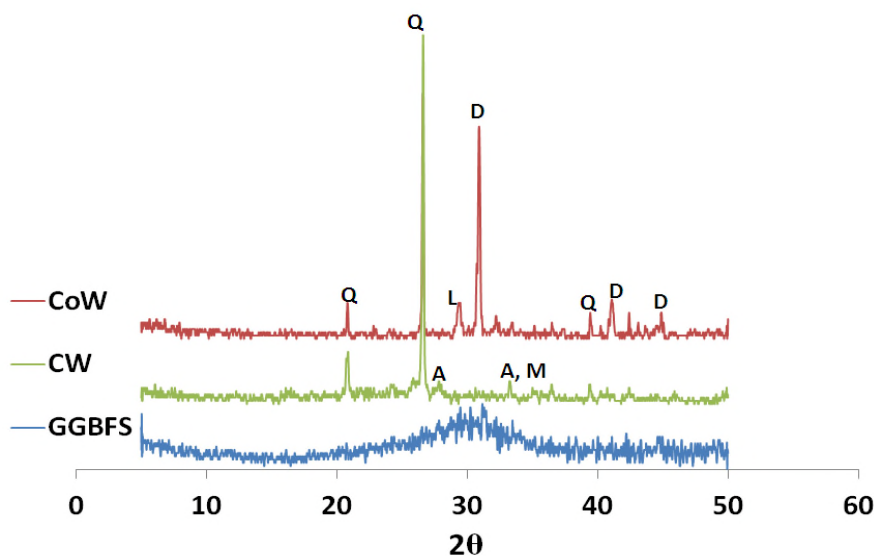
Fig. 1 XRD patterns of GGBFS, CW and CoW

80°C for 24h then cured in tap water to examine its resistivity to water curing. Geopolymer cement samples were cured in humidity chamber at 100% RH and 37±2°C for 24h. All samples were demoulded then cured in tap water at 23±2°C until reaching the time of testing.

2.3 Methods of investigation

The combined water content was determined by igniting a certain weight of dried crushed specimens at 1000°C for 60 minutes soaking time. The chemically combined water content was regarded as the ignition loss of dried crushed specimen. Duplicate experiments were carried out in order to ascertain good reproducibility of the results. Chemically combined water content is calculated on the ignited weight basis according to the following equation: Combined water (Wn), %=[(W$_0$-W$_i$)/W$_i$]*100 Where, W$_0$=dried sample weight and W$_i$=ignited sample weight. The compressive strength was recorded after 1, 3, 7, 14, 28, 56 days according to ASTM C109M (2012). XRD analysis was carried out on a Philips PW3050/60 X-ray diffractometer using a scanning range from 5 to 50 2θ degrees with a scanning speed of 1second/step and resolution of 0.05°/step. Infrared spectral analysis (FTIR) was carried out for some selected hydrated samples to provide additional information on the phase transition. The FTIR was recorded from KBr discussing Genesis-IIFT-IR spectrometer in the range of 400-4000cm^{-1}.The scanning electron microphotographs were obtained with Inspect S (FEI Company, Holland) equipped with an energy dispersive X-ray analyzer (EDXA).

3. Results and discussion
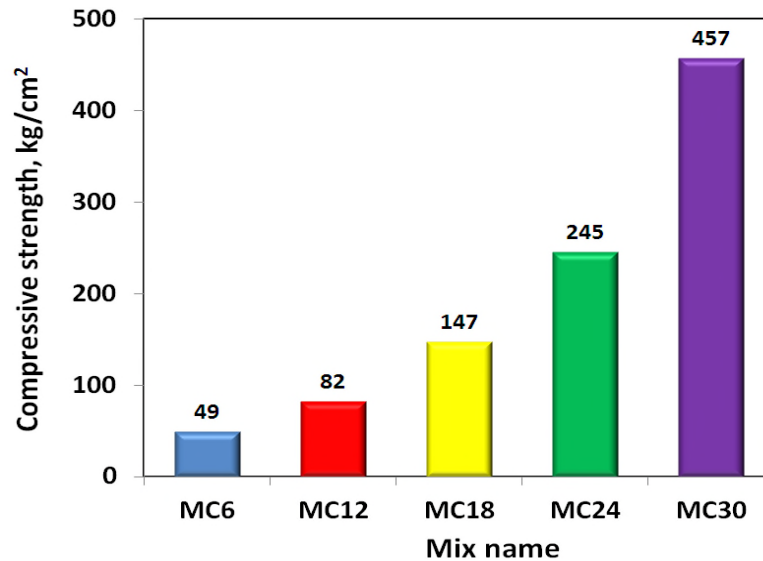
3.1 Mechanical properties of geopolymer resin

Fig. 2 Compressive strength of AACW mixes cured at 80 °C for 24h

Fig. 3 Visual observation of CW activated by different NaOH& Na2SiO3content after 24h of water curing

3.1.1 Compressive strength

Fig. 2 shows the compressive strength of geopolymer resin made of CW activated by 3:3 (MC6), 6:6 (MC12), 9:9 (MC18), 12:12 (MC24) and 15:15 (MC30) NaOH: Na2SiO3 after one days of curing in oven at 80°C. The compressive strength of alkali activated ceramic waste (AACW) increases with the increase of NaOH: Na_2SiO_3 percentage up to 15:15wt., %. This is mainly due to that, the alkali activator was found to have profound effect on both the length of the induction period and the rate of network formation. In addition, as the amount of alkali activator increases, the dissolution rate increases releasing higher amount silicate, aluminate and/or aluminosilicate in the matrix. This may appear to imply faster geopolymer network formation.

3.1.2 Resistivity of AACW to water curing

After drying of AACW, the samples are transferred to water tank to study its behavior in water curing. Fig. 3 shows that, all CW samples activated by 3:3, 6:6, 12:12 and 15:15 NaOH: Na_2SiO_3 are completely destroyed after 24h of curing in tap water. It can be say that, the activation of CW with

high alkali content leads to produce hardened geopolymer resin with high mechanical properties and no resistivity to water curing. This may be explained by the expansion occurred as a result of absorbing a lot amount of water by free sodium silicate present in geopolymer matrix. Also, the activation of CW by low amount of NaOH: Na_2SiO_3 (3:3 and 6:6) has the same behavior in water curing. This is due to that the amount of activators used do not sufficient for dissolution of aluminosilicate in CW, yielding porous geopolymer with low binding capacity. After immersing in water, the water penetrates through the pores leading to destruction of the cubic sample. The optimum percentage of activator which gives relatively higher mechanical properties and resistivity to water curing is 9:9 NaOH: Na_2SiO_3.

3.2 Physico-chemical and mechanical properties of geopolymer cement

3.2.1 Chemically combined water

Fig. 4 represents the chemically combined water content of AACW replaced by 10 and 30 % CoW as well as 10 and 30 % GGBFS. It is evident that, the replacement the AACW by 10 wt., % CoW leads to the decreaseof chemically combined water content, suggesting the lower activity of CoW compared to CW. Further addition of CoW causes the increase in combined water content.

This may be explained by the presence of carbonate containing phases (calcite and dolomite) contribute in the increase of the chemically combined water content. On the other hand, as the amount GGBFS increases, the chemically combined water content also increase, proving the higher GGBFS reactivity compared to CW. Furthermore, the increase in GGBFS content leads to increase the CaO/SiO2 ratio, forming hydration product with high combined water content.
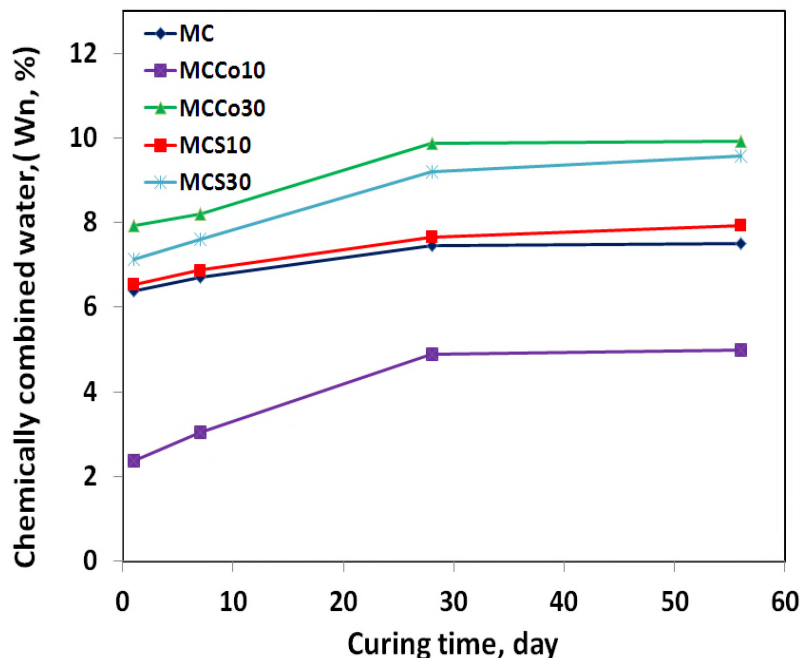

Fig. 4 Chemically combined water content of AACW replaced by 10 and 30 % CoW as well as 10 and 30 % GGBFS
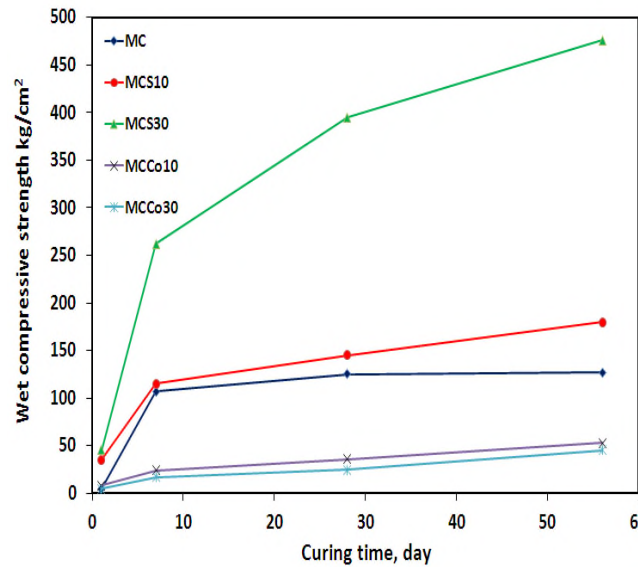
Fig. 5 Compressive strength of AACW replaced by 10 and 30 % CoW as well as 10 and 30 % GGBFS
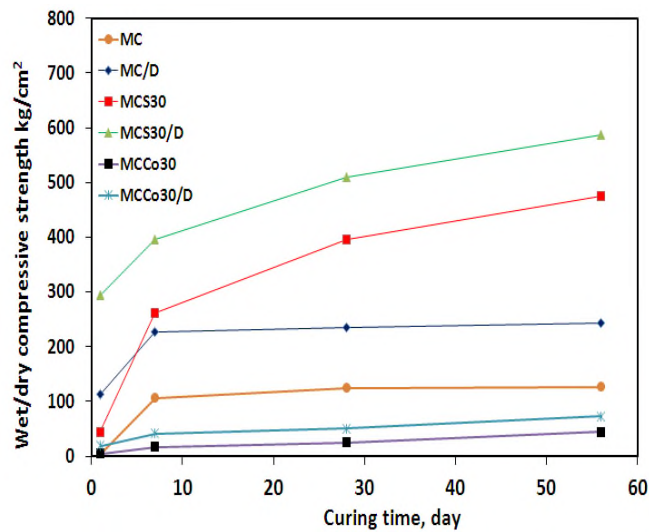

Fig. 6 Wet/dry compressive strength of AACW replaced by 30 % CoW and GGBFS

3.2.2 Wet compressive strength

Fig. 5 displays the wet compressive strength of AACW and AACW replaced by 10 and 30 wt., % CoW as well as 10 and 30 wt., % GGBFS. The increase of AACW replacement level by concrete wastes leads to decrease the compressive strength value at all ages of hydration. This indicates the lower CoW reactivity compared to CW. In contrast, with the increase of slag content the compressive strength increases. This is attributed to that the GGBFS provides the system by very active calcium aluminosilicate species which in turn increase the probability of hydration products formation, leading to increase the compressive strength. It is important to observe that,

the compressive strength values of AACW-GGBFS are higher than those of AACW-CoW, confirming the higher activity and amorphiticity of GGBFS compared to CoW.

3.2.3 Wet/dry compressive strength

Fig. 6 shows the compressive strength, (wet and dried samples at 80°C for 24h) values of AACW and AACW replaced by 30 wt., % CoW or GGBFS. It is shown that, the dry compressive strength values are higher than those of wet compressive strength for all mixes at all ages of curing. The drying process marginally affects the compressive strength of AACW-CoW mixes. A significant increase in compressive strength was observed when the drying process applied on AACW-GGBFS mixes. These facts proved the higher GGBFS reactivity compared to CoW.

3.2.4 XRD Analysis

Fig. 7 represents the XRD patterns of AACW (MC18) and AACW replaced by 30 % CoW (MCCo30) and 30 % GGBFS (MCS30) after 28 days of curing. It is clear that, the XRD pattern of MCS30 mix seems to be less crystalline comparedto MC18 and MCCo30 mixes. This confirms amorphous nature of GGBFS compared to CoW and CW. This can be observed from the decrease of peak intensity related to quartz when AACW replaced by 30 % GGBFS. The CSH peak is difficult to detect by XRD, especially in the presence of high crystalline quartz phase due to it is amorphous nature.

3.2.5 FTIR spectroscopy

Fig. 8 shows the FTIR spectra of AACW (MC18) and AACW replaced by 30% CoW (MCCo30) and 30% GGBFS (MCS30) after 28 days of curing. It is obvious that, the FWHM of absorption band related to asymmetric stretching vibration of Si-O-T (T=Al or Si) in case of MCS30 is smaller than that of MC mix. Also, the replacement of CW by 30% GGBFS leads to shifts this band to lower wavenumber. Both shift to lower wavenumber and the decrease in FWHM are attributed to the ordering of silicate network caused by enhancement crystallinity degree of CSH. c, The replacement of AACW by 30% CoW leads to the increase FWHM, suggesting the
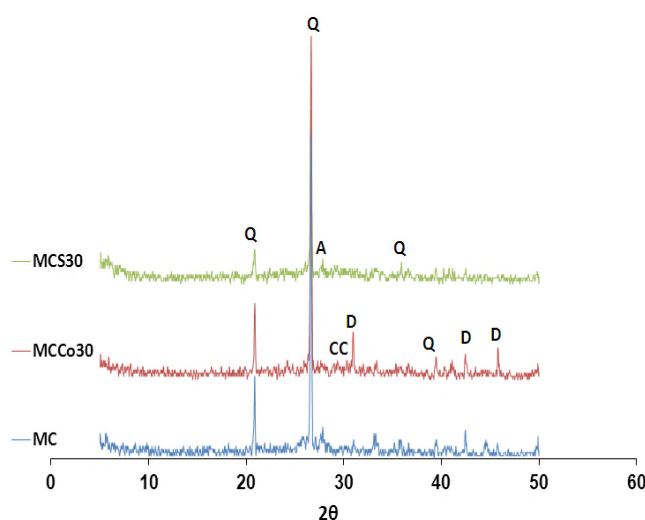

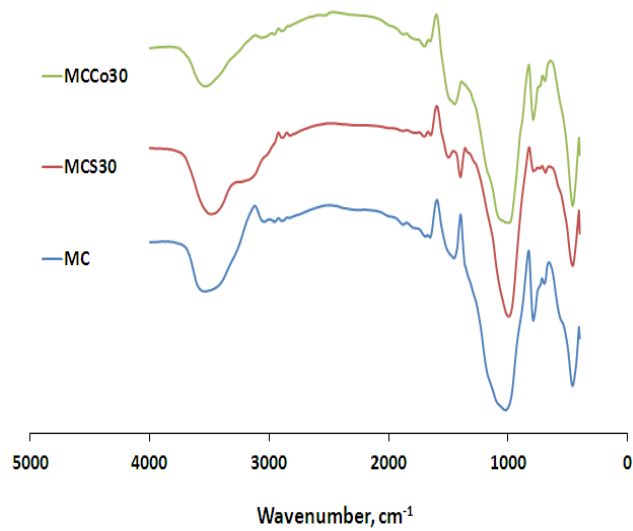Fig. 7 XRD patterns of AACW (MC) and AACW replaced by 30% CoW and GGBFS

Fig. 8 FTIR spectra of AACW (MC) and AACW replaced by 30% CoW (MCCo30) and GGBFS (MCS30) after 28 days of curing
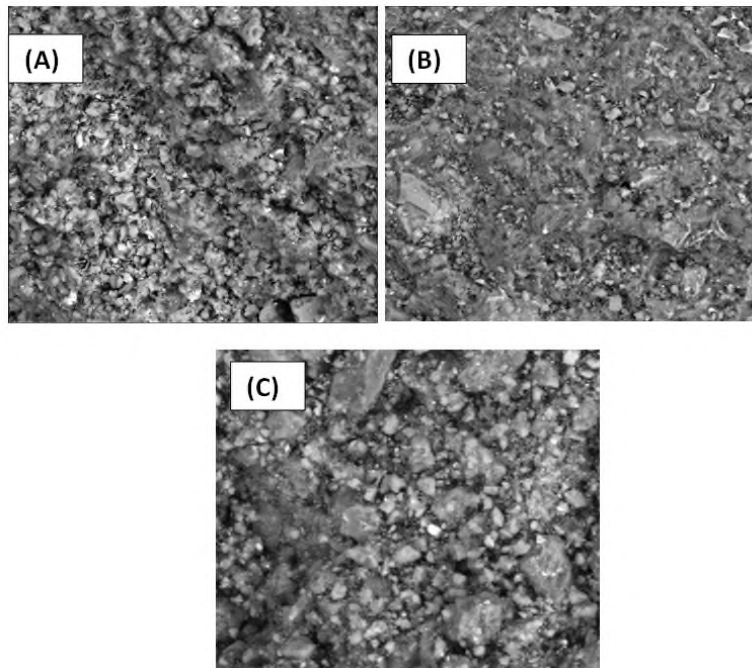

Fig. 9 SEM microstructures of AACW (A) and AACW replaced by (B) 30% GGBFS and (C) CoW after 28 days of curing

low reactivity of CoW compared to CW and GGBFS as a result of leading to form disordered microstructure formation. These facts are in a good harmony with the compressive strength results. The intensity of absorption bands related to H-O-H bending vibration and OH group stretching

vibration in case of MCS30 mix is higher than those of MC18 and MCCo30 mixes, reflecting the combined water content results.

3.2.6 SEM microscopy

Fig. 9 represents the SEM microstructure of AACW (MC18) and AACW replaced by 30% CoW (MCCo30) and 30% GGBFS (MCS30) after 28 days of curing. The SEM observation shows that, the microstructure of MCCo30 mix seems to be inhomogeneous and less compact containing large and small aggregate distributes along the geopolymer matrix, confirming the lower activity of CoW compared to CW, which negatively affect the compressive strength values. The replacement of AACW by 30% GGBFS leads to form more compact, dense and homogeneous microstructure. This indicates that the reactivity of GGBFS is higher than that of CW and CoW. In other words, the addition of GGBFS leads to increase the activated species which condensed to form geopolymer, filling open pores and therefore, the densification and ordering of microstructure enhances.

4. Conclusions

In this paper, the geopolymer resin was prepared from alkali activation of low cost aluminosilicate materials (ceramic waste) with different alkali contents. In order to prepare geopolymer cement, two calcium sources have been used (CoW and GGBFS).

Several findings were observes from this work as follows:
- The compressive strength of AACW increases with the increase of alkali activator content.
- All mixes of ceramic waste activated by 3:3, 6:6, 12:12 and 15:15 wt., % NaOH: LSS destroyed as a result of water curing. Meanwhile, AACW activated by 9:9 wt., % NaOH: LSS gave the relatively higher compressive strength and resistivity to water curing.
- The replacement of AACW containing 9:9 wt., % NaOH: LSS by 10 and 30 wt., % CoW caused a significant decrease in the compressive strength. In contrast, the chemically combined water of AACW containing 30 wt., % CoW showed the highest chemically combined water content compared to AACW with no and with 10 wt., % CoW. The chemically combined water content of AACW containing 10 wt., % CoW was lower than that of control sample.
- The compressive strength and chemically combined water content increases with the increase replacement level of AACW by GGBFS at all curing times.
- The XRD proved that the higher crystallinity of mix containing CoW compared to that containing GGBFS. The FTIR confirms the higher reactivity of GGBFS compared to CoW as a result of successive hydration products formation, enhancing the compaction of microstructure as observed from SEM.

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