

Asses the Use of Calcium Carbide for Increasing Mechanical and Thermal Properties of Alkali Activated GGBFS-MK Precursor

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ABSTRACT

The purpose of this paper is to investigate whether calcium carbide can effectively develop the strength of a slag-metakaolin geopolymer matrix. For all mixes, a solution of sodium hydroxide and sodium silicate was used as a liquid alkali activator. CaC_2 was added in ratios of 0, 0.5, 1.0, 1.5, 2.0 and 2.5% by weight. Compressive strength shrinkage and thermal resistance of alkali-activated composites were examined. The GGBFS-MK geopolymer pastes with/without calcium carbide were subjected to FTIR, XRD, and SEM investigations for inspecting the effect of the CaC_2 addition. The findings demonstrated that calcium carbide enhances compressive strength compared to the control mix, by producing more reaction products and providing a greater level of geopolymerization.

Keywords: Calcium Carbide, Compressive Strength, Geopolymer, Shrinkage.

Published Online: January 18, 2023

ISSN: 2684-4478

DOI: 10.24018/ejchem.2023.4.1.125

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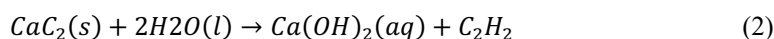
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I. INTRODUCTION

Recent years have seen a significant increase in enthusiasm for the advancement of environmentally friendly and low energy-consuming sustainable repair materials. The cement manufacturing process used in concrete production release a great amount of CO_2 , which is environmentally destructive. Hence, finding alternatives to regular Portland cement is crucial, the goal is to develop a substance that can replace cement while maintaining all of its desirable features. This product is made from alkali-activated materials which are named scientifically (Geopolymer) [1]-[4]. Geopolymers are formed by using industrial wastes or by-products to form a binder that resembles OPC and providing a similar function with environmental and technical benefits, reduction in CO_2 emissions by 80-90% and resistance to fire and chemicals [5], [6]. OPC and geopolymer differ primarily in their manufacturing processes, OPC relies on a high-energy manufacturing method whereas Geopolymer uses materials with very low energy like fly ash, slag, and a small quantity of chemicals (alkali medium). Despite geopolymer advantages like low-energy manufacturing process, waste utilization, and resistance to fire and aggressive chemicals, the use of geopolymer is limited to special performance applications these limitations are because there aren't enough long-term performance data and there aren't enough geopolymer feedstocks compared to OPC. [7]-[9]. Some additives can be used to address these negatives [9]-[11]. Calcium carbide (CaC_2) is one of these additives. CaC_2 is manufactured by chemical processing of limestone by heating lime and coke (carbon) to 2000-2100°C in an electric arc furnace, according to (1):



Since 1892 this method has not changed [12]-[13]. Acetylene gas is produced when (CaC_2) interacts with H_2O in geopolymer paste.



Horpibulsuk *et al.* [14] made non-bearing masonry units using fly ash (FA) and calcium carbide residue (CCR) as a binder instead of Portland cement (PC). According to the cost analysis, the (CCR)&(FA) masonry unit's material costs were about 40% inferior to those of the cement. In addition to being economical, a significant percentage of calcium carbide waste would be diverted from landfills, it would also lead to a substantial decrease in carbon emissions associated with cement production. Mohammadinia *et al.* [15] improved the mechanical capabilities of the construction and demolition (C&D) sector for use in pavement applications by using different mixtures of CCR. Phummiphan *et al.* [16] employed CCR and FA to improve geopolymer composites for preserving Lateritic soil in the margins as a long-lasting base of pavement, aiming to enable calcium carbide residue from waste to be utilized as a geopolymer binder promoter, this is crucial from environmental and commercial considerations. Raghunathan *et al.* [17] have tried calcium carbide powder (CaC_2) as aerator or as a gas-producing agent in different contents 1, 2, 3, 4 and 5%. In geopolymer paste CaC_2 reacts with water to produce acetylene gas [2]. Findings revealed that the maximum strength is given by 1% of CaC_2 . But mixes 2% and 3% of calcium carbide in paste show less compressive strength. In terms of low cost and environmental friendliness, a study by Phoo-ngernkham *et al.* [18] on alkali-activated high-calcium fly ash (FA) combined with (CCR) revealed that by combining (FA) and (CCR), the mortar's setting time dropped while its compressive strength and bond strength increased as CCR grew. Cong *et al.* [19] study silica fume addition to activated CCR and indicated that the consumption of excessive CCR facilitates the hydration product formation and modifies pore structure. Li [20] investigated that CCR could clearly enhance the compressive strength of geopolymer matrix leading to more practical applications for geopolymer in soil stabilization. Du and Yang [21] investigated the effectiveness of using CCR as an alkali activator for clay/plant ash geopolymers. Findings proved that the (CCR) clearly has a positive impact on compressive strength by increasing CaC_2 contents. Fly ash (FA)-based geopolymers were prepared by using CCR as an alkali activator without supplementation of any alkali and results showed that after geopolymerization, amorphous calcium (aluminate) silicate hydrate (C-(A)-S-H) gels formed in greater amounts, resulting in better mechanical properties, a study by Wang *et al.* [22]. Sun *et al.* [23] replaced the NaOH and KOH by (CCR-carbonates) and $(\text{Ca}(\text{OH})_2\text{-carbonates})$ system to activate GGBFS developing cemented paste backfill, results demonstrated compressive strength higher than the control but lower fluidity, which promised well for lowering production costs while ensuring production safety. The core objective of the presented work is to study the impact of CaC_2 used in different ratios on thermo-mechanical properties as well as study its effect on shrinkage. Mineralogical investigation and microscopic imagery of alkali activated GGBFS-MK composite were studied.

II. EXPERIMENTAL

A. Materials

The starting materials used in this study are Ground Granulated Blast Furnace Slag (GGBS) obtained from Iron and Steel factory- Helwan, Egypt for (GGBS) and metakaolin obtained from Aluminum Sulfate Co. of Egypt (ASCE). Calcium carbide (CaC_2) were used at different dosage from 0.5 to 2.5% by mass of powder, CaC_2 was obtained from Fisher scientific Company, UK. Table I is summarized The Chemical composition of GGBS and MK used in this study. The XRD pattern of MK and GGBS is shown in Fig. 1. Liquid Sodium Silicate LSS (Na_2SiO_3) used in the present investigation obtained from Fisher company composed of 17% Na_2O and 32% SiO_2 with silica modulus $\text{SiO}_2/\text{Na}_2\text{O}$ equal 1.88, and its density is 1.46 g/cm^3 . Mixed proportionally at equal volume of NaOH solution as alkali activator. superplasticizer was added for better dispersion of the added compositions.

TABLE I: CHEMICAL COMPOSITIONS OF STARTING MATERIALS, WT %

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	TiO_2	L.O.I
GGBS	36.59	10.1	1.84	33.07	6.43	1.39	0.74	3.52	0.52	-
MK	61.1	31.4	1.51	0.32	0.11	0.12	0.14	0.13	2.44	2.33
CaC_2	1.47	1.13	0.16	68.34	0.08	0.15	-	0.88	-	27.7

B. Mortars Preparation

Equal volumes of liquid Sodium Silicate LSS (Na_2SiO_3) and sodium hydroxide solution (SH) were used as activators to produce GGBFS-MK geopolymer composite. (SH) was obtained from 280 g of sodium hydroxide pellets in 1 L of distilled water (7M) and then allowed to cool down for 24 h before use. CaC_2 was added at dosages of 0.5, 1, 1.5, 2, and 2.5 % by mass of powder. The activator and CaC_2 were added. Then the mix is casted in cubic molds of 2.5 cm length and smoothing of the surface takes place by a thin-edged trowel after molding specimens were cured under ambient temperature for 24, de-molded specimens cured at 40°C with a 100% relative humidity (R.H.). Specimens were removed from their curing condition, dried at 80°C for 24 h and then subjected to compressive strength, whereas the resulted crushed specimens

were dried at 105 °C to prevent further hydration, until the examination time specimens should preserve in a well tight container. They are measured at 7, 28, 60 and 90 days. Compressive strength performed according to ASTM C109 M [24]. Geopolymer Mix design of the mortar are presented in Table II.

C. Investigation Methods

XRF-Axios (PW4400) WD-XRF Sequential Spectrometer was used to investigate the chemical composition of starting materials. Mechanical testing was carried out using 5 tons German Bruf digital compression testing machine with a loading rate of 100 kg/min. The FTIR testing used for clarification of the amorphous constituents of geopolymer composites using Jasco-6100, samples were mixed with KBr before testing, FTIR conducted in the wave number region between 400 and 4000 cm^{-1} [25], [26] whereas the XRD analysis was performed for 2 theta from 10° to 50°. The morphology of the hydration products was examined by scanning electron microscope (SEM) Inspect S (FEI Company, the Netherlands) equipped with an energy-dispersive X-ray analyzer (EDX). The thermo-mechanical properties tested for the composites by exposing them to elevated temperature in the furnace. Each sample after cured at 28 d subjected to thermal treatment by raising the temperature at 10 °C/min from room temperature (R.T.) to elevated temperature at the interval of 300 °C, 500 °C, 800 °C at a sustained period of 2 hr. The compressive strength of each sample tested after air annealing to R.T. and % retention of the strength calculated. The morphology examined by scanning electron microscopy (SEM).

TABLE II: MIX COMPOSITION OF ALKALI ACTIVATED PASTES

Mix no.	(MK) (%)	GBS (%)	CaC ₂ (%)	LSS (%)	SH (%)	W/C (%)
C0	50	50	0.0	0.125	0.125	0.25
C1	50	50	0.5	0.125	0.125	0.25
C2	50	50	1	0.125	0.125	0.25
C3	50	50	1.5	0.125	0.125	0.25
C4	50	50	2	0.125	0.125	0.25
C5	50	50	2.5	0.125	0.125	0.25

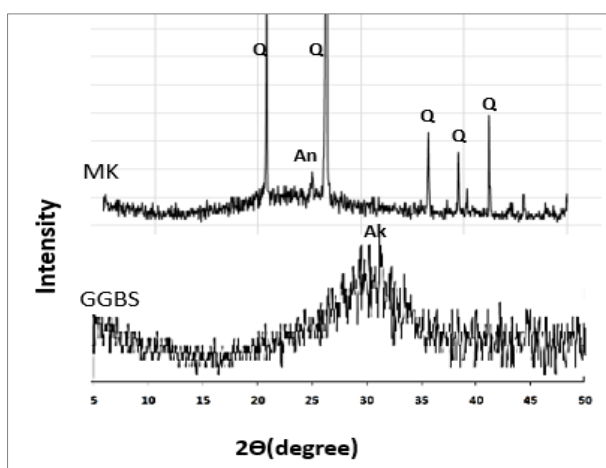


Fig. 1. X-ray diffraction pattern of the starting raw material (Q= Quartz, An= Anatase, AK= Akermanite).

III. RESULTS AND DISCUSSION

A. FTIR

Fig. 2 illustrates the use of FTIR spectra to examine the geopolymerization rate of GGBFS-MK geopolymer paste with/without calcium carbide at 90 days of curing. All specimens are positioned in the range between 400 and 4000 cm^{-1} . Significant broad bands at approximately 1655–1600 cm^{-1} and 3450 cm^{-1} related to O-H bending and O-H stretching. These are corresponding to water molecules in the polymeric skeleton voids [27]-[28] in addition to hydration binder formed by interaction of slag with MK forming CSH and CASH, the broad peak located at 970-1050 cm^{-1} was affiliated to (T-O-Si) asymmetric stretching vibration, while the peak at about 1100 for (Si-O-Si) asymmetric stretching vibration for silica non-solubilized. This wave number reflects the geopolymerization degree within the matrix which is more notable than that of (O-Si-O) (454 cm^{-1}) bending mode [29]. This connected to the O-Si-O bending mode represents the unreacted quartz remaining in the mixture. Two peaks, at 781 cm^{-1} and 691 cm^{-1} , were assigned to quartz vibration (Si-O-Si), whereas that at 575 cm^{-1} most likely resulted from the symmetric stretching vibration of (Si-O-Si) [30].

The band of carbonate (CO_3) can be observed between 1400–1450 cm^{-1} . In addition to out-of-plane bending of CO_2 at around 870 cm^{-1} . From the pattern, we can notice the shifting of the asymmetric

stretching vibration of (T-O-Si) to lower wave number up on using 0.5% CaC₂ as a result of intensifying geopolymerization reaction. In addition to the decreasing of the intensity of asymmetric stretching (Si-O-Si) for non-solubilized silica confirms the increased dissolution of silica with CaC₂ addition. Increasing CaC₂ ratio to high ratio leads to the intensity decrease of asymmetric stretching vibration (T-O-Si), which leads to the increase of the porosity leading to low cohesion between the reacting particles.

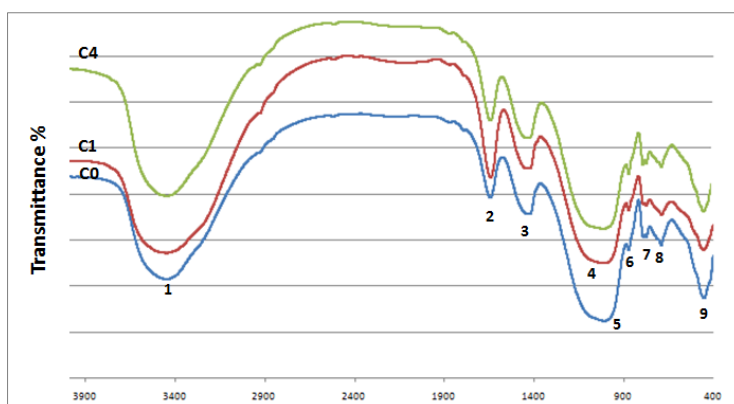


Fig. 2. FTIR spectra of alkali-activated composites at 90 days of curing with various ratios of CaC₂.

[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)].

B. XRD Analysis

The XRD patterns of 90 days curing GGBFS – MK mixtures with/ without CaC₂ are depicted in Fig. 3. The main reaction product of the samples is identified as amorphous geopolymer constituents (NASH) in the range of 17-35° 2θ [31], as this hump increase the intensity of the formed geopolymer increase leading to the formation of dense matrix. In addition to the amorphous geopolymer constituents there is a noticeable peak for CSH at about 29.2° 2θ, which results in extra strength of the matrix. Using CaC₂ at 0.5% leads to the formation of calcium-silicate-hydrate gel (C-S-H) at about 29.2° 30.62°, 32.25° 2θ, which subjected to decrease upon increasing CaC₂ content.

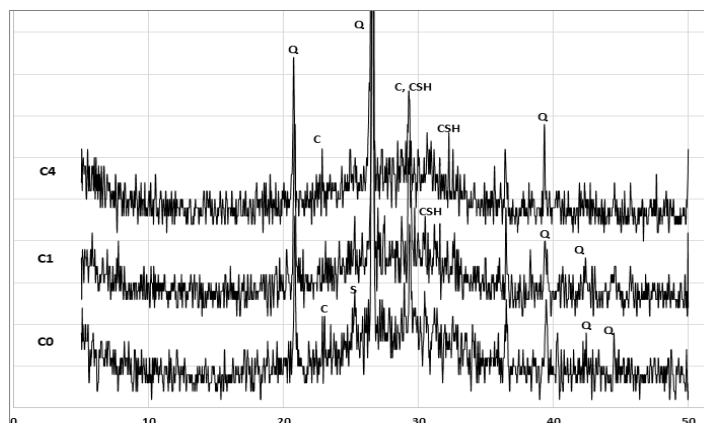


Fig. 3. XRD pattern of 90-day alkali- activated GGBFS-MK composite with various ratios of CaC₂ (Q= Quartz, C= Calcite, S= Sodalite, CSH= Calcium Silicate Hydrate).

C. Compressive Strength

Fig. 4 clearly shows the Compressive Strength of GGBFS- MK specimens treated with CaC₂ up to 90 days and dried at 80°C for 24 hrs, where the strength values upon using CaC₂ have greater values than control. High compressive strength behavior is due to the characteristic of CaC₂ as promoter. CaC₂ is mainly consisted of calcium hydroxide (Ca(OH)₂). Thus, the use of calcium promoters such as CaC₂ can enhance compressive strength of geopolymer mortars. Ca(OH)₂ from hydration products chemically react with Al₂O₃ and/or SiO₂ to form pozzolanic reaction speeding up the behaviour of hardening and enabled the pastes setting. The highest compressive strength of (1044.48) kg/cm² for 0.5% CaC₂. The effectiveness of CaC₂ comes from the release of Ca²⁺ and OH⁻ ions in the aqueous phase [32]. Either excessive dose of CaC₂ (i.e., the C2, C3, C4 and C5 samples) resulting in a generally low compressive strength. This may be due to the incorporation of higher ratio of CaC₂ leads to the inclusion of Ca(OH)₂ content that will undergoes precipitation by the effect of the used alkalis leading to the formation of precipitates rather than CSH binding material and so decreasing the alkali content which is driving force for geopolymer formation and

accumulation. This outcome proved that the geopolymerization went on and more geopolymeric gels formed, thus actually improving the compressive strength. The findings from FTIR spectroscopy compatible well with the compressive strength results and morphological features analysis.

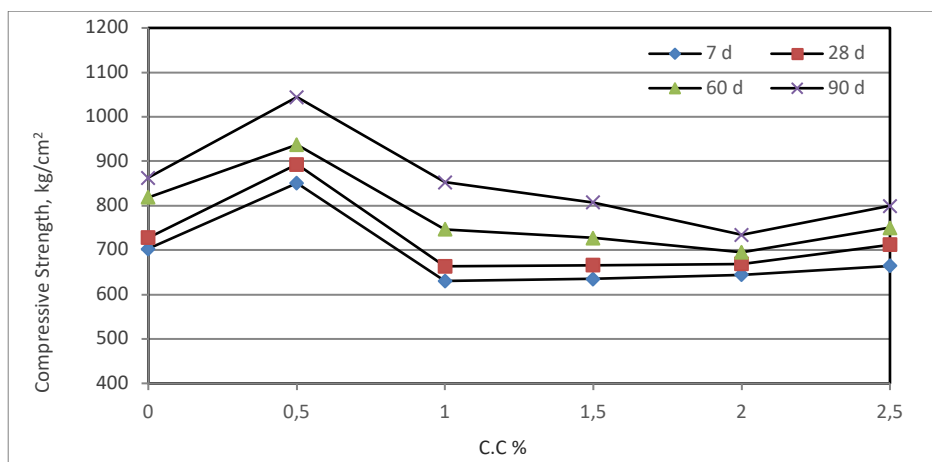


Fig. 4. Compressive strength of GGBFS-MK composite with different CaC_2 ratios.

D. Scanning Electron Microscopy Analysis

The SEM micrograph of geopolymer paste with/without calcium carbide cured at 90 days is illustrated in Fig. 5. The micrograph of Fig. 5-B (0.5% CaC_2) shows a dense matrix with the presence of dense laminars of geopolymer that fill most of the voids within the matrix leading to the formation of a dense matrix, additionally Ismail *et al.* [33] reported that CSH spreading within the matrix which is formed interaction of $(\text{Ca}(\text{OH})_2)$ with SiO_2 and Al_2O_3 through pozzolanic reaction creating C-S-H and C-A-S-H gel coexisting with N-A-S-H, impacting the development of compressive strength as a result of microstructure homogeneity.

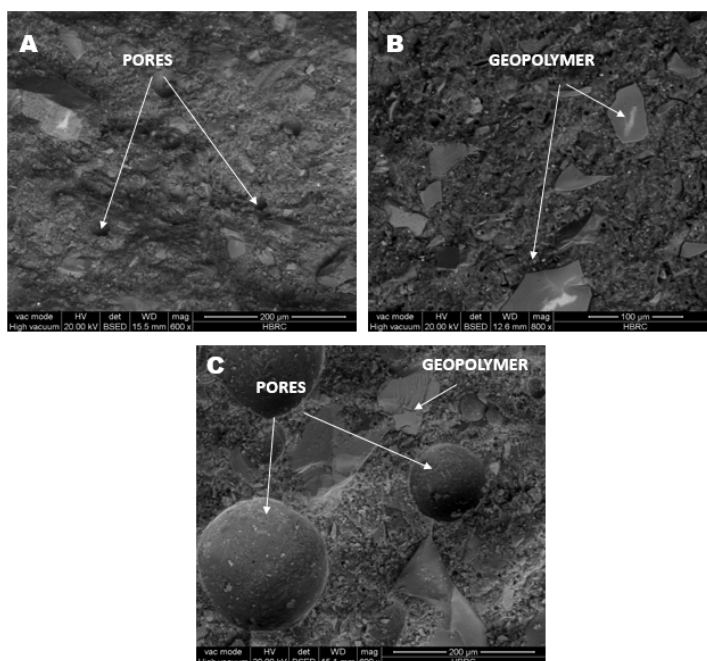


Fig. 5. SEM of 90 days alkali- activated GGBFS-MK composite containing: a) 0%; b) 0.5% and; c) 2% CaC_2 .

Further CaC_2 addition as shown in Fig. 5c affects negatively on microstructure as well as the compressive strength development of the binders. Moreover, the calcium hydroxide in the excess of calcium carbide is able to react with CO_2 in the atmosphere producing CaCO_3 , which formed by the increased porosity at higher ratio of CaC_2 which leads to an increase in carbonation where the calcite (CaCO_3) formation mostly restricted to the large clusters rather than being uniformly distributed throughout the matrix [34]. This may be explaining the negative effects of more additions of CaC_2 on the microstructure dense and compressive strength improvement. The pattern reflects also the increased calcite contact with further CaC_2 increases, as a result of increased porosity C_2H_2 release leading to carbonation increase.

E. Drying Shrinkage

Generally, concrete shrinkage mostly arises from the shrinkage of the paste matrix due to the high dimensional stability of the aggregates [35]. The increment of drying shrinkage with respect to time is plotted in Fig. 6. Drying shrinkage is a consequence of loss of moisture from the hardened matrix to the environment. Due to the emptying of the fine pores in the concrete, negative capillary pressure develops, which causes a volume reduction of the concrete [36]-[38]. The incorporation of CaC_2 can diminish the drying shrinkage slightly in geopolymer mixtures. The lowest drying shrinkage percent was with 0.5 % CaC_2 which can reduce shrinkage by approximately 14 % and 15 % for specimens cured at 28 and 90 days respectively. This is attributed to the tightly binding of the water molecules within the three-dimensional geopolymer network and which prevents the evaporation of these molecules and leads to the decrease of the drying shrinkage values with time.

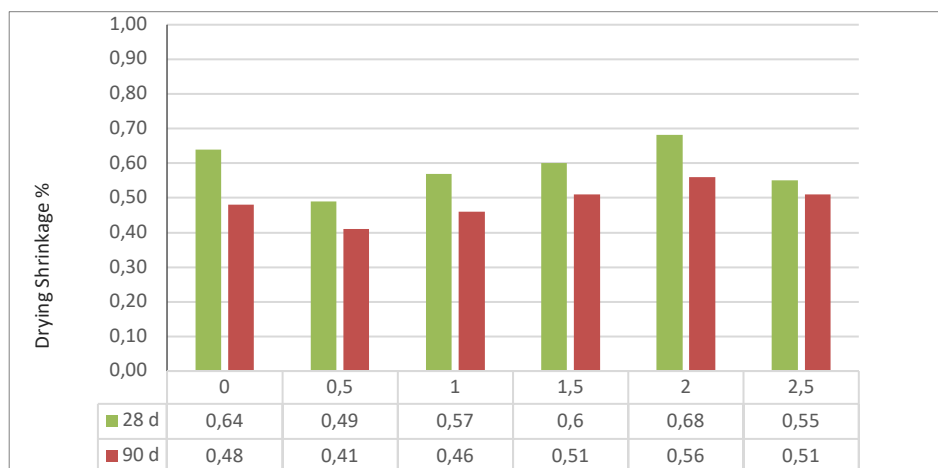


Fig. 6. Drying Shrinkage of different mixes at 28 and 90 days of curing.

F. Thermal Resistance

Geopolymers provide better thermal stability than OPC at high temperatures [39]. For Alkali activated GGBFS-MK composite to examine the effects of thermal treatment on the weight loss, mechanical characteristics, and shrinkage of both CaC_2 containing and CaC_2 free geopolymer after 28 days of curing in 100% RH, certain number of cubes of some selected pastes were dried at 80°C for 24 hr. then fired at 300°C, 500°C and 800°C. The weight and shrinkage before and after firing were taken in addition to compressive strength as compared to that dried at 80°C at 28 days.

1) Mass loss of geopolymers

Starting materials and composition of the geopolymer have a direct impact on the weight loss at high temperatures. The weight loss through evaporation of free water and a portion of chemically bonded water from geopolymers occurs between R.T. and 300°C, which is probably connected to the degradation of hydration products. Si-OH, Al-OH and Ca-OH groups dehydroxylate at 300–550°C. Over 750°C the loss of weight is due to the decomposition of carbonate species [40], [41]. Fig. 7 shows that the addition of CaC_2 at all temperature ranges resulted in a quite lower weight loss percentage than the control. Additionally, as the temperature rose, the weight loss percentage increased. The largest mass loss was noticed at 800°C, due to the rise in carbonate dissociation.

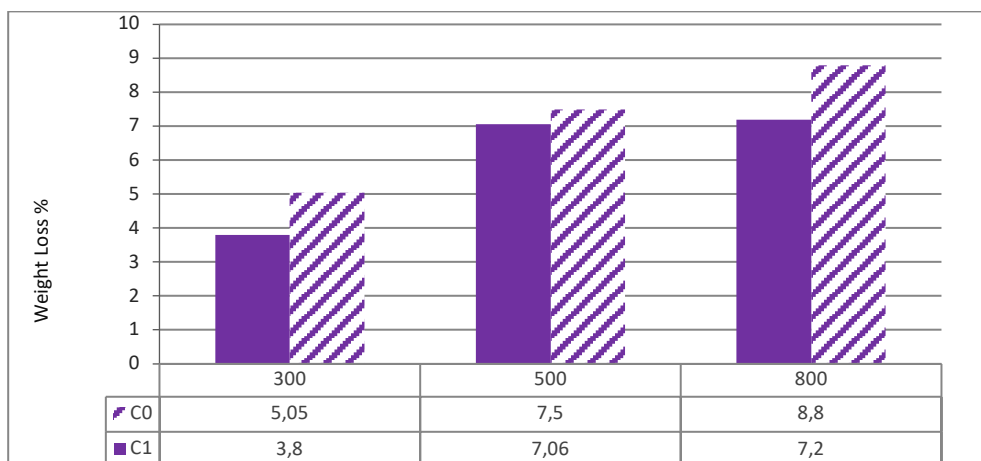


Fig. 7. Weight loss% for mixes C0 and C1 versus treatment temperatures.

2) Strength Change Factor (SCF)

After being heated to a high temperature, the tested geopolymer pastes had their properties compared to a reference substance that had been dried at 80°C. Equation (3) can be used to determine the average of Strength Change Factor.

$$SCF \% = \frac{s_0 - s_f}{s_0} * 100 \quad (3)$$

where,

s_0 for strength after drying at 80°C and s_f for strength after firing.

The strength change factor versus temperature is represented in Fig. 8, for geopolymer with CaC_2 exposure to 300°C caused the material to gain strength. Such an effect may be caused by the release of adsorbed moisture causing stiffening of the gel and increasing surface tensions between gel particles or by encouraging polycondensation amongst chain-like geopolymer gels [42]. At higher temperatures, when exposed to temperature over 400°C, it has been found that the compressive strength of ordinary cement concrete drops significantly [43], compressive strength of geopolymers also decreased, loss of strength at high temperature is associated with the disappearance of chemically bound water in calcium-rich gels, This causes phase separation and cavity structure [44], [45]. When the temperature is raised to 800°C, a clearly detectable decrease in compressive strength. Fig. 8 shows that the addition of CaC_2 can enhance the thermal resistance compared to the untreated one.

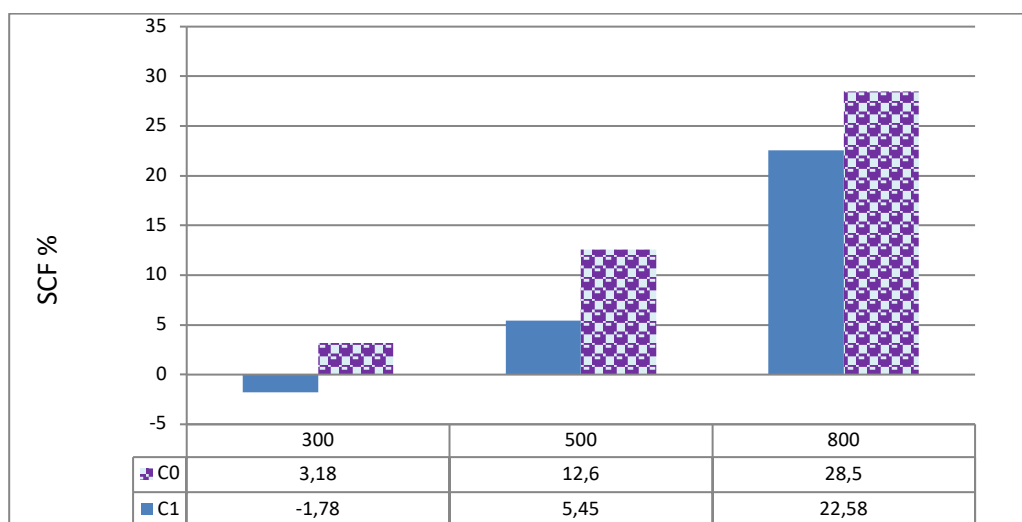


Fig. 8. Strength Change Factor (SCF) % for both mixes C0 and C1 versus treatment temperature.

3) Firing shrinkage

The thermal shrinkage of geopolymers at age of 28 days increased with temperature increase as presented in Fig. 9. For the geopolymer dried at 80°C and fired specimens. Average of firing shrinkage % can be calculated using (4).

$$F.S \% = \frac{DL - FL}{DL} * 100 \quad (4)$$

where DL for dried length and FL for fired length.

Fig. 9 showed less shrinkage value with CaC_2 treated specimen (C1) than control one (C0). The removal of water during the polycondensation stage caused the thermal shrinkage. This process also resulted in the formation of capillary tensions, which drew the particles together and produced water, the production of which increased as the temperature goes up [46]-[47].

A new crystalline phase formation is most likely the cause of the considerable increase in thermal shrinkage that occurred when the geopolymer was heated to 800°C. At temperature 300°C and 500°C there was a slightly increased shrinkage, but at 800°C, there was a significant shrinkage increase. CaC_2 as additive can enhance the geopolymer resistance to heating process.

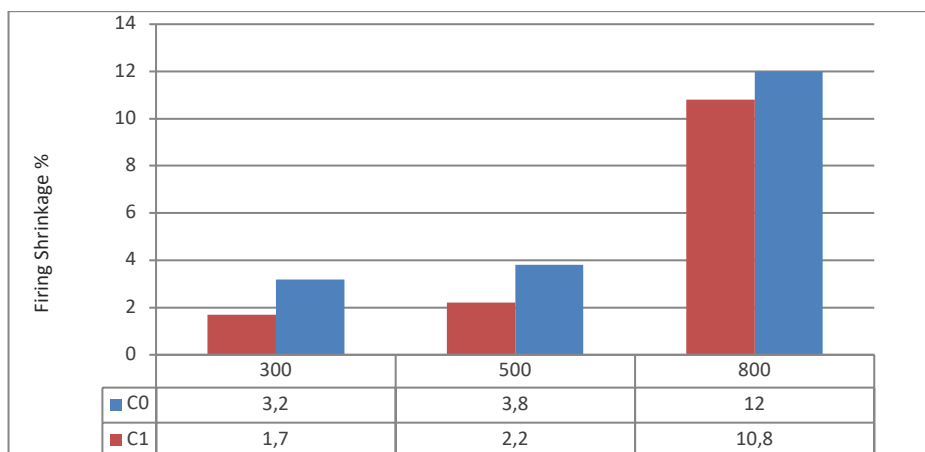


Fig. 9. Firing shrinkage of mixes C0 and C1 versus treatment temperatures.

IV. CONCLUSION

In this study, the impact of calcium carbide on the thermo-mechanical properties of alkali-activated GGBFS-MK pastes was demonstrated using Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and Scanning electron microscopy (SEM).

- In a word, the mechanical properties of GGBFS-MK geopolymer composites coupled with CaC_2 are greatly enhanced. The FTIR, XRD revealed that the maximum compressive strength value was achieved by adding 0.5% of CaC_2 . Also morphological examination showed a dense impacted matrix with the addition of 0.5% of CaC_2 .
- Drying shrinkage also was elucidated, CaC_2 addition slightly enhancing the shrinkage reduction. The lowest drying shrinkage percent was with 0.5% CaC_2 .
- It can be concluded from the above analysis that the GGBFS-MK geopolymer composites coupled with CaC_2 possess good thermal stability even at high temperatures.
 - i. Weight loss percentage slightly decreased with the addition of CaC_2 .
 - ii. Shrinkage increased as temperature increased, a sharp increase happened at 800°C, when the addition of CaC_2 can minimize shrinkage to some extent.
 - iii. For mechanical properties after firing there was a compressive strength gain of geopolymer specimens with increasing firing temperature up to 300°C. At 500°C and 800°C the Compressive strength decreased.

CONFLICT OF INTEREST

Authors declare that they do not have any conflict of interest.

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