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Fibre reinforced alkali activated composites exposed to elevated temperature

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Abstract

This work aims to reveal the effect of fibres on the strength and durability of alkali activated composites (AAC). AAC specimens were prepared by alkali activation of Ground granulated blast furnace slag (GGBS). Two types of fibres i.e. micro steel (~237.8µm) and polypropylene (~32.06µm) were used as reinforcement. The properties of fibre reinforced AAC specimens were compared with those of fibre reinforced ordinary portland cement (OPC) counterparts. AAC specimens yielded higher compressive and tensile strength than OPC specimens. Fibre reinforced specimens exhibited improved strength over those without fibres. Specimens reinforced with polypropylene fibre showed better performance in terms of water absorption, and apparent porosity. This may be attributed to the finer size of polypropylene fibres. The specimens were then exposed to elevated temperatures up to 900ºC. Compressive strength was observed to decrease after exposure to elevated temperatures in both AAC and OPC specimens. OPC specimens completely crumbled upon reaching 900ºC while AAC specimens remained intact with appearance of surface cracks. From the present study, it is concluded that fibre reinforcement leads to significant increase in strength and size of fibres controls the durability of the specimens.

Keywords: Alkali activation, Compressive strength, Micro steel, Polypropylene, Tensile strength

1. Introduction

The environmental effect of manufacturing ordinary Portland cement (OPC) has been a serious concern for over three decades. The production of one ton of cement liberates about one ton of CO_2 to the atmosphere as a result of decarbonation of limestone in the kiln and the combustion of fossil fuels [1]. Many researchers have pointed out the need for a better sustainable material to substitute conventional concrete. Geopolymers are binders so produced using source materials of alumino silicate origin activated using alkaline solutions [2]. The geopolymers produced using source materials with high percentage of calcium oxide such as Ground Granulated Blast Furnace Slag (GGBS) are also termed as Alkali Activated Composites (AAC) [3]. GGBS is a byproduct produced in iron industries and is a non-toxic material, and could prove to be a good raw material for making high value AAC which can be utilized in fire resistant applications. Alkali activated materials (AAMs) are alternative green materials produced by activating a high calcium aluminosilicate precursor with an alkali source [4-9]. They can be activated at ambient temperature with the main product formed as calcium-aluminosilicate hydrate C-A-S-H [10-12]. However, the main reaction product formed in geopolymer materials is sodium-aluminosilicate hydrate N-A-S-H [13]. The geopolymerisation behavior, physical, mechanical properties and fire resistant characteristics of GGBS based AAC are strongly dependent upon the chemical composition in the reaction system [14].

Alkali activated slag(AAS) is considered the most popular among alkali activated material (AAM) for the fact that it consume relatively less energy when used to replace OPC as binder [15]. Moreover, AAS composites have been reported to provide superior mechanical and durability properties [16]. Disadvantages in the use of alkali activated slag are its high shrinkage [17], and comparatively quick setting [18].

The addition of short, discontinuous fibres plays an important role in improving the mechanical strength of concrete [19]. Addition of fibres to concrete makes it more homogeneous and isotropic and transforms it from a brittle to a ductile material [20]. Fibre reinforced showed significantly better mechanical strength than portland cement based mortar [21]. The superior performance of alkali activated slag mortars is due to higher bond properties between fibres and AAC matrix [22]. Thus, the tensile properties of binders can further be improved with the incorporation of fibres.

Literature on fibre reinforced AAC is available in plenty. However, it is hard to find literature on fibre reinforced AAC on elevated temperature. The present work is focused on the study of properties of fibre reinforced AAC in elevated temperature up to 900ºC and comparison of results with those of OPC specimens.

2. Experimental program

2.1 Materials

The materials used for the present experimental investigation are Ordinary Portland Cement of 43 grade and GGBS (SiO_2=35.01% Al_2O_3=17.13% Ca0=36.58%). For activating solution, laboratory grade sodium hydroxide pellets (RANKEM)

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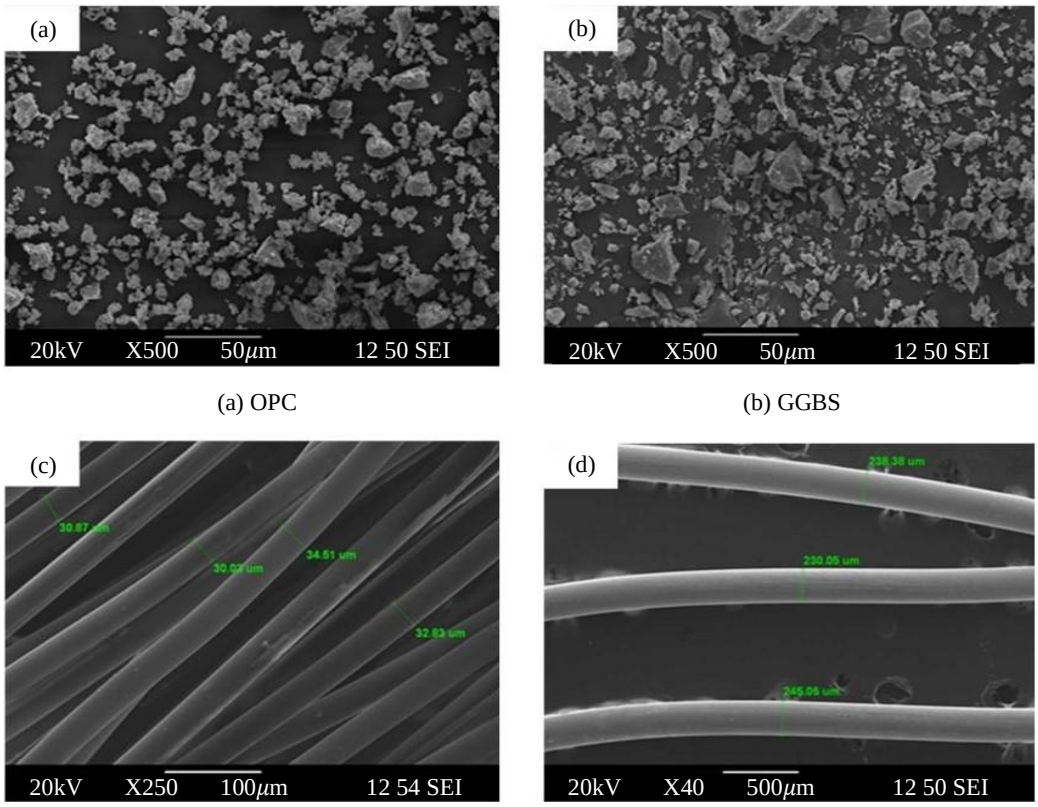
(Na_2O=77.5%, H_2O=22.5%) and sodium silicate (Stanbio Reagents Pvt. Ltd, Kolkata) (SiO_2=30.65%, Na_2O=14.15%, H_2O=55.2%) were used. Two types of fibres i.e. micro steel and polypropylene served as fibre reinforcement at different percentages and their properties are highlighted in Table 1. SEM micrographs of OPC, GGBS, micro steel fibre and polypropylene fibre are shown in Figure 1.

Table 1 Properties of the Fibres

Type of fibre	Diameter (μm)	Length (mm)	Aspect ratio	Tensile strength (MPa)	Modulus of elasticity (GPa)
Micro steel	278	21	88.31	~2300	200
Polypropylene	32	12	655.02	~350	~0.35



(a) OPC (b) GGBS

(c) Polypropylene fibre (d) Micro steel fibre

Figure 1 SEM of materials

2.2 Specimen preparation

The alkaline activating solution used in the manufacture of alkali activated composites (AAC) was a mixture of sodium hydroxide solids, sodium silicate solution and water. The activating solution had 8% Na_2O and 8% SiO_2 for all the activated GGBS specimens. Required extra quantity of water was added to sodium hydroxide pellets and subsequently sodium silicate solution was further introduced to obtain the activator. Upon cooling, the activator solution was kept agitated using a magnetic stirrer till its use in mixing. For all the specimens, the water-binder ratio was maintained at 0.33. The ingredients were mixed thoroughly in a non-absorbent container to obtain a homogenous mix and later transferred into cube moulds (50x50x50 mm) and cylindrical mould (50 mm ϕ x 100 mm). For reinforcement, two types of fibres namely micro steel and polypropylene were used at two different weight fractions i.e., 0.25% and 0.50%. Table 2 shows the details of specimens prepared in the experimental program. The OPC specimens were water cured for 28 days before testing. However, AAC specimens were cured dry at ambient temperature until tested. Images of some demoulded specimens are presented in Figure 2.

2.3 Tests methods

Three samples each were tested for determining the required properties and average values are reported with standard deviations indicated by error bars. The compressive strength were conducted after 28 days as per ASTM C109 [23]. Water absorption, apparent porosity of the specimens were performed as per ASTM C948-81 [24] and split tensile strength as per ASTM C496 [25].Scanning electron microscopy (SEM) on samples sputter coated with gold were conducted on Sigma 300, Carl Zeiss and scanned at magnification of 500x and 10000x. Quantification of elements by EDX were performed at an accelerating voltage of 20 kV. A muffle furnace was used to expose the specimens at elevated temperature. The specimens were exposed to elevated temperature of 300, 600 and 900ºC at a constant heating rate of 5°C / min and were retained at the designated temperature for 24 hours. The specimens were then allowed to cool in the muffle furnace. The effect of elevated temperature on surface and physical changes, reduction in weight and residual compressive strength were investigated and a comparison was made between the specimens.

Figure 2 Demoulded cylinder specimens

Table 2 Details of test specimens

Specimen ID	OPC	PP1	PP2	MS1	MS2	GGBS	GPP1	GPP2	GMS1	GMS2
Cement %	100	100	100	100	100	-	-	-	-	-
GGBS %	-	-	-	-	-	100	100	100	100	100
Water/binder ratio	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33
Polypropylene %	-	0.25	0.50	-	-	-	0.25	0.50	-	-
Micro steel %	-	-	-	0.25	0.50	-	-	-	0.25	0.50

3. Results and discussion

3.1 Scanning Electron Microscopy (SEM)

Properties such as water absorption, sorptivity and porosity depend on the microstructure. SEM images and corresponding EDS for OPC specimen and alkali activated GGBS specimen are shown in Figure 3. Images of both specimens depict good formation of gel. However, GGBS specimen present comparatively a better microstructure as compared to that of OPC specimen. This will result in lesser porosity and lower water absorption in alkali activated slag specimens. Additionally, this would impart higher mechanical strength and superior durability.

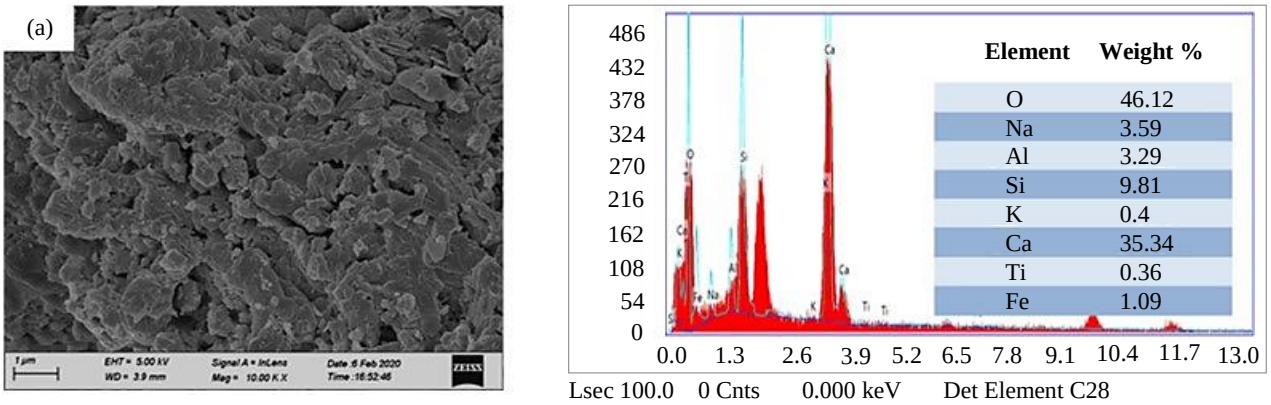
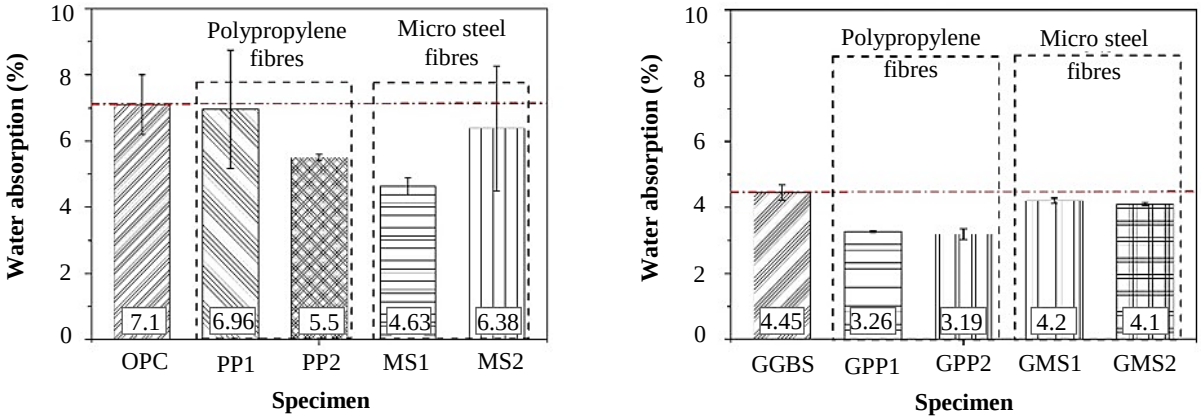


Figure 3 SEM Image of specimen before exposure

Elemental weight percent as determined by EDS shows significantly higher Ca and lower Si content in OPC sample. However, weight percentage of Ca and Si is approximately equal in GGBS specimen. The microstructural changes are attributed to the formation of different hydration products for GGBS and OPC specimens i.e. C-A-S-H and C-S-H gels respectively. The C-A-S-H gel has a lower Ca/Si ratio of 1.22 as compared to that OPC (Ca/Si ratio of 3.60). Previous researchers have also reported similar lower Ca/Si for C-A-S-H gel in the range of 0.9-1.22 [12, 17, 26].

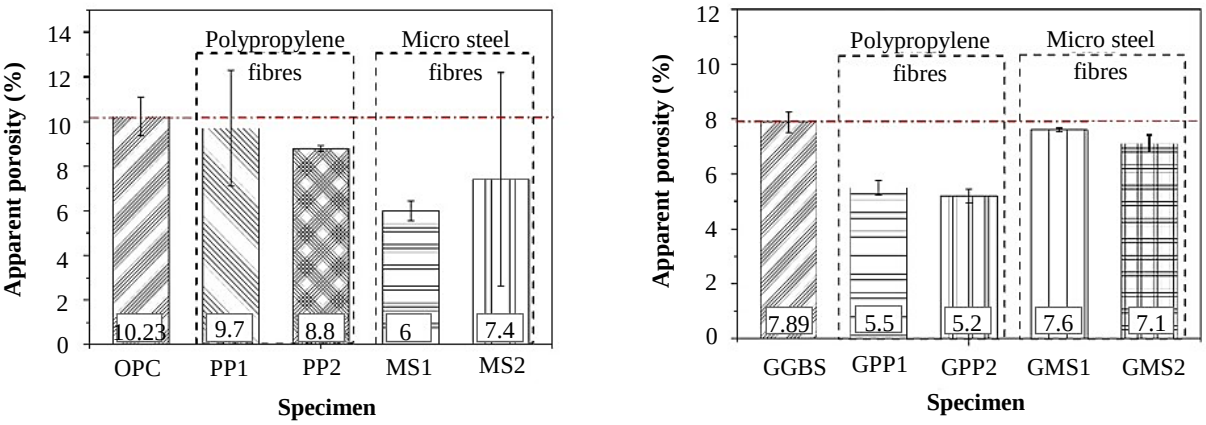
3.2 Water absorption and apparent porosity

Water absorption and apparent porosity are simple and effective method to study the durability properties of binders. The effect of polypropylene and micro steel fibre content on water absorption is shown in Figure 4. It is evident that incorporation of fibres decreases the physical properties considerably. Compared with control OPC specimen, the water absorption of PP1, PP2, MS1 and MS2 decreased by 1.97, 22.53, 34.78, and 10.14% respectively. For AAC specimens, a decreasing trend of 26.74, 28.31, 5.62, and 7.86 % for GPP1, GPP2, and GMS1and GMS2 respectively was observed with respect to control GGBS specimen. A similar decreasing trend is also observed in respect of apparent porosity for OPC as well as AAC specimens as shown in Figure 5. The lower water absorption can be attributed to the decrease in porosity. However, water absorption tends to increase with fibre content. Significant increase in porosity and water content may be expected at higher dosage of fibres.



(a) OPC (b) AAC

Figure 4 Water absorption of OPC and AAC specimens



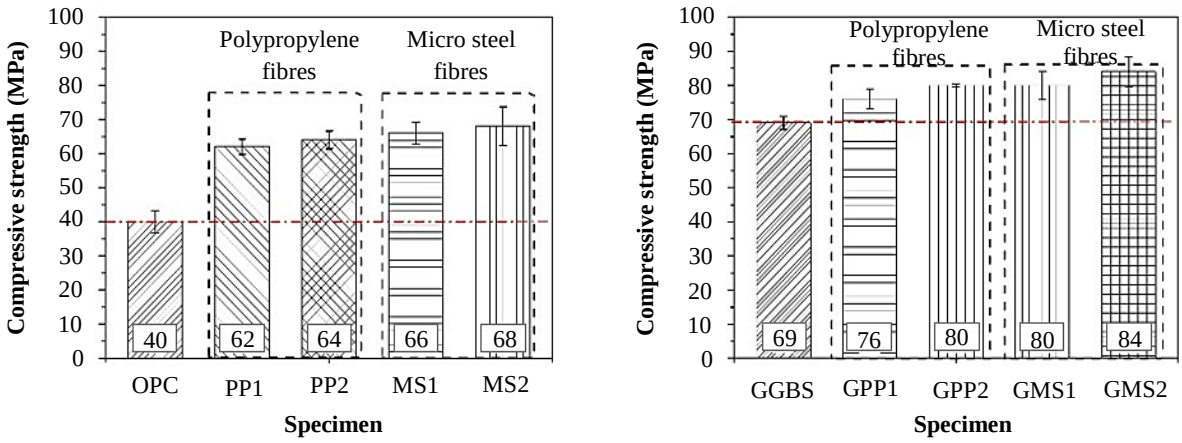
(a) OPC (b) AAC

Figure 5 Apparent Porosity of OPC and AAC specimens

It is also clearly observed that water absorption of AAC specimens is lower when compared to their OPC counterparts. Comparatively control specimen of AAC specimen (GGBS) has water porosity and apparent porosity values lower by 37.3 and 22.87% respectively. This may be attributed to better microstructure in AAC specimen as indicated by SEM in Figure 3. In fibre reinforced OPC specimens, increasing micro steel fibres resulted in increased water absorption and apparent porosity. It is possible that fibres tend to clump together during mixing, entrapping water-filled pores that subsequently turn into voids [27]. However, a reverse trend is noticed in the AAC specimens for both water absorption and apparent porosity. This may be explained by the fact that the larger micro steel fiber leads to entrapping of more air voids [28].

3.3 Compressive strength and tensile strength

Results of compressive strength of OPC and AAC specimens after 28 days from casting are shown in Figure 6. AAC specimens yielded higher compressive strength than their OPC counterparts. An increase of about 72% in compressive strength of control AAC (GGBS) specimen with respect to that of OPC specimen was observed.



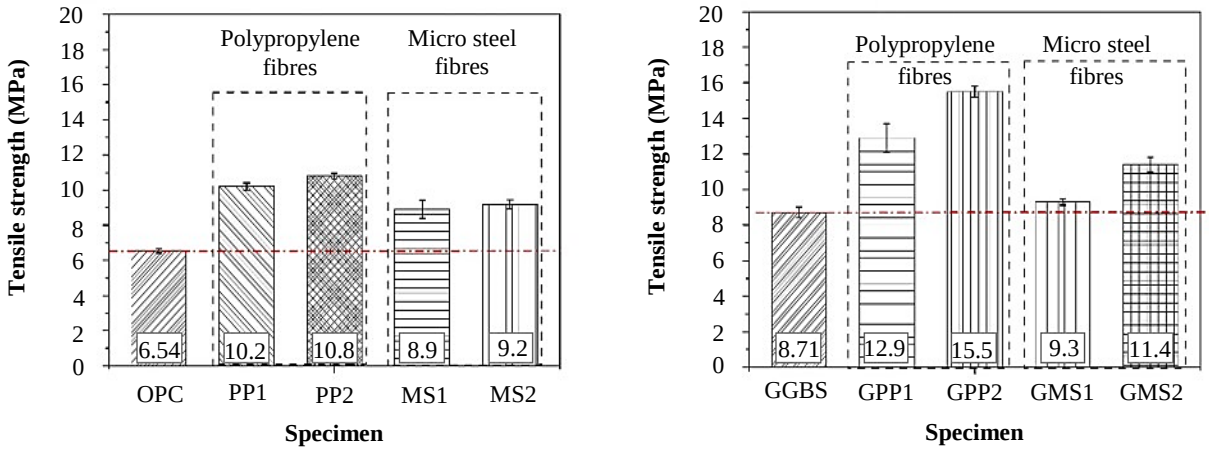
(a) OPC (b) AAC

Figure 6 Compressive strength of unexposed samples

The high compressive strength of alkali activated slag is attributed to the calcium silicate hydrate gel (C-S-H) formed with composition and structural differences from those of cement specimen [29]. Reinforcement with fibre leads to increase in compressive strength for both OPC and AAC specimens. Moreover, fibre percent in the mix directly influenced the compressive strength of the specimens. For OPC specimens, improvement percentage of 55, 60, 65 and 70% were observed for PP1, PP2, MS1 and MS2 as compared to control OPC specimen respectively. Also, for AAC specimen, the compressive strength increases by 10.14, 15.94, 15.94 and 21.73% respectively for GPP1, GPP2, GMS1 and GMS2 respectively.

Maximum compressive strength of 84 MPa was obtained for GMS2 (AAC specimen with 0.5% microsteel fibre). It amounted to 21.7% increase from control AAC specimen (GBS). Compared to MS2 specimen (OPC with 5% micro steel fibre), GMS2 specimen showed exceptionally higher compressive strength. This could be due to better microstructure as noticed in SEM micrographs. However, the increase in strength in fibre reinforced OPC specimens over plain OPC specimen was much higher than those of AAC specimens and it reached as high as 70% for MS2 specimen. It clearly shows the effectiveness of incorporating fibre into OPC specimens. Increase of compressive strength by fibre inclusion may be attributed to the relatively small dimensions of these fibers, which gave these fibers the ability to delay the micro-crack formation and to arrest and prevent their propagation afterwards up to a certain extent [30-32].

Split tensile strength of cylindrical specimens were performed after 28 days and the results are presented in Figure 7. The tensile strength of control OPC and GGBS specimens were 6.54 and 8.71 MPa respectively.



(a) OPC (b) AAC

Figure 7 Tensile strength of unexposed samples

The addition of 0.25 and 0.50% polypropylene and micro steel has increased the split tensile strength value of control OPC specimens by 55.9, 65.1, 36.1 and 40.67% respectively. Similarly, for AAC specimens, percentage increase (as compared with control GGBS specimen) were noted as 48.1 and 77.91% for the addition of 0.25 and 0.50% polypropylene with 6.71 and 30.88% for corresponding addition of micro steel. Addition of polypropylene were found to be more beneficial in split tensile strength as compare to the addition of micro steel fibre for both OPC and AAC specimens. Comparative lower value of split tensile strength for micro steel fibre reinforced specimens is attributed to the fact the steel fibres create internal voids which reduces the density of the specimens [33].

Further, the compressive strength and split tensile strength for OPC and AAC specimens are correlated and has been compared with recommendations of ACI 318 [34] as shown in Figure 8. Both the curves corresponding to fibre reinforced OPC and AAC specimens are nearly parallel to the reference curve as per ACI. However, with the introduction of fibre reinforcement, the tensile strength tends to improve as observed in the figure.

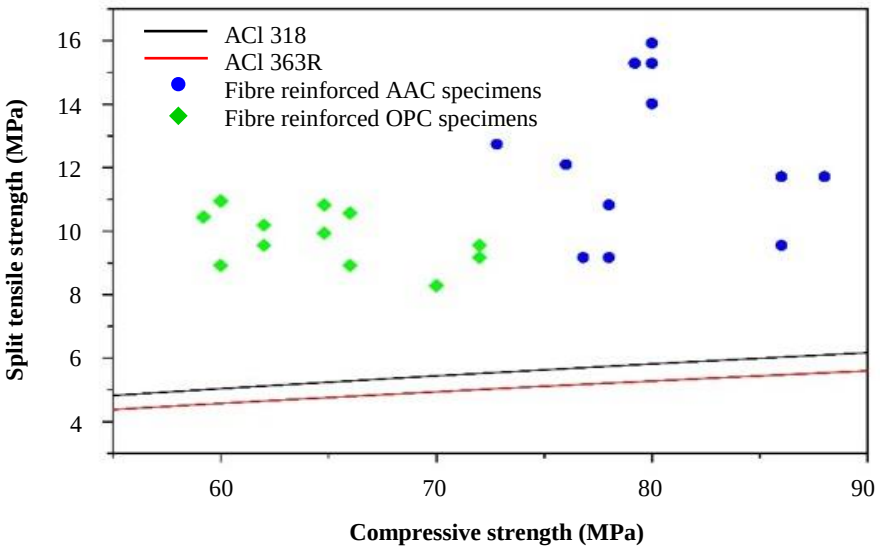


Figure 8 Relation of split tensile strength and compressive strength

3.4 Surface appearance at elevated temperature

The surface texture of the fibre reinforced OPC and AAC samples after exposure to various elevated temperatures are shown in Figure 9 and Figure 10 respectively. The specimens exhibited tremendous changes in physical appearance with increasing exposure temperature. Large cracks were visible in specimens PP1 and MS1 as early as 600ºC. However, AAC specimens remained intact even at 900ºC. As the fire resistance capacity of AAC is higher than OPC, the benefits of fibre reinforced were more prominent in AAC specimens as compared to OPC [35]. The AAC samples retained their original color at 300ºC and the color started to lighten as the temperature increases.

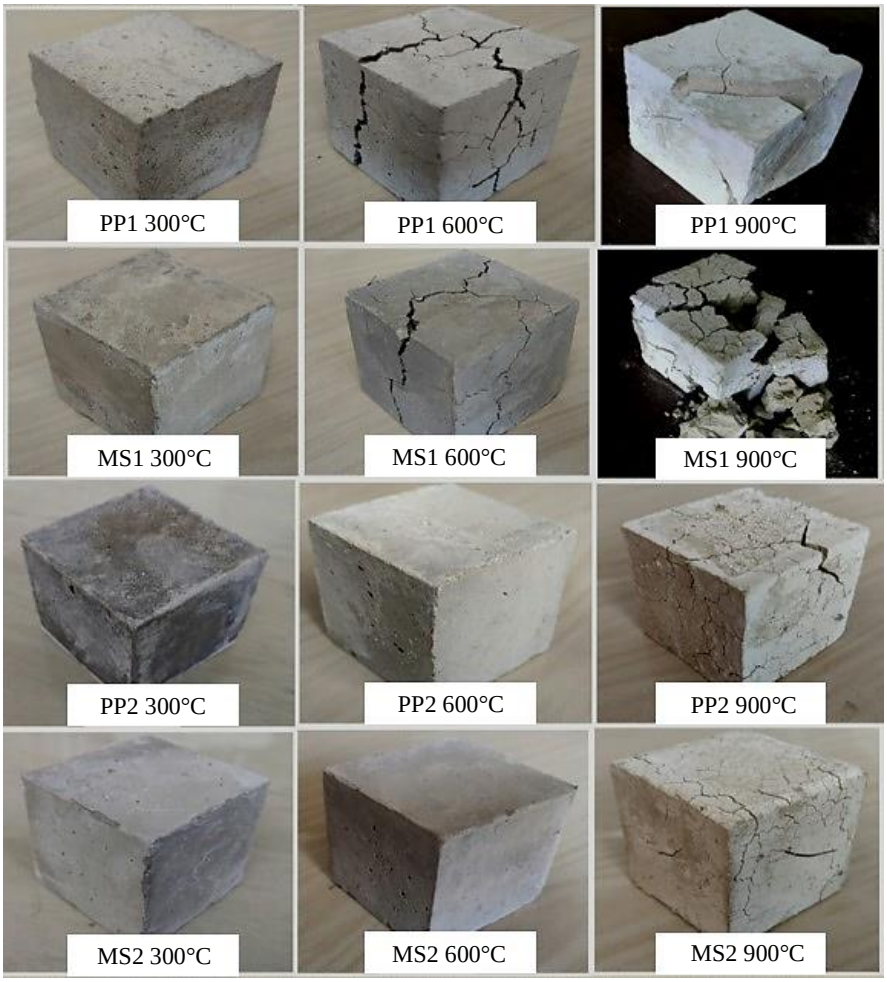


Figure 9 Surface image of fibre reinforced cement paste samples

Figure 10 Surface image of fiber reinforced AAC paste samples

3.5 Weight loss at elevated temperature

The weight evolution of specimens during the entire duration of elevated temperature exposure are shown in Figure 11 and Figure 12 for fibre contents of 0.25% and 0.5% respectively. The weight loss can be categories into three temperature ranges such as ambient to 300ºC, 300-600 and 600-900ºC. Below 300ºC, the percentage weight loss for the specimens were almost similar till 300ºC for both the fibre contents which also involves melting of polypropylene fibres [36]. However, upon reaching 600ºC, fiber reinforced OPC specimens (PP1, MS1, PP2 and MS2) display significant weight loss as compared to its AAC counterparts. This becomes more substantial at elevated temperature of 900ºC. The maximum weight loss recorded was around 30% for fibre reinforced OPC specimens. This is due to reason that conventional concrete degrades due to the destruction and decomposition of moisture from the crystalline hydrates of C-S-H [20]. Whereas for AAC specimens, the movement of bonded water in the matrix is possible due to the presence of nano pores in its microstructures without causing any damage to the aluminosilicate network [37]. Fibre reinforced alkali activated specimens showed lesser weight loss, irrespective of the difference in type of fibre used in both 0.25% and 0.5% fibre content. In both OPC and AAC specimens, specimens reinforced with polypropylene fibre exhibited the least weight loss with a value slightly above 20%.

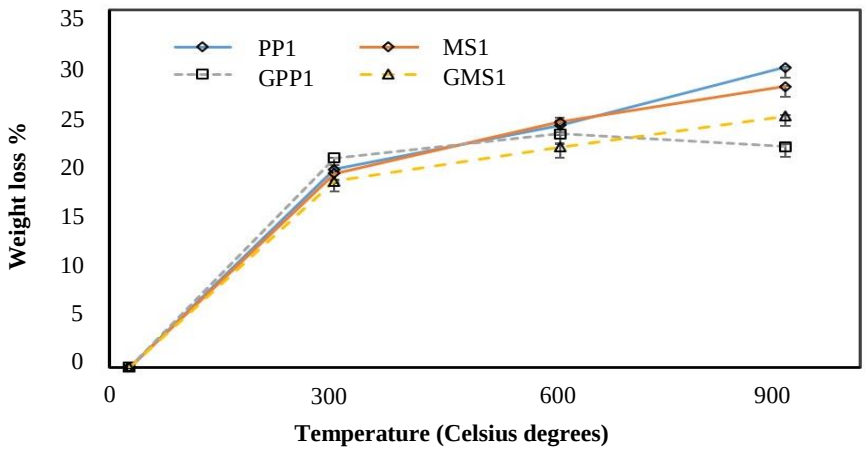


Figure 11 Weight evolution for fibre reinforcement of 0.25%

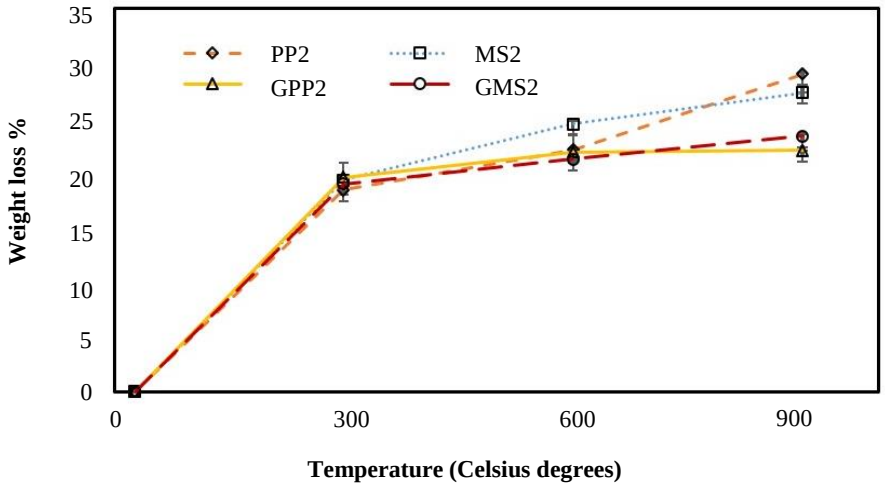


Figure 12 Weight evolution for fibre reinforcement of 0.50%

3.6 Residual strength of specimen exposed to elevated temperature

The residual strength of the specimens was found as ratio of strength of exposed specimen to their corresponding initial strength before exposure, expressed as percentage. Figure 13 and 14 shows the residual strength against increasing exposure temperature for fibre contents of 0.25% and 0.5% respectively. At both fibre contents, the specimens exhibited decrease in compressive strength after exposure to elevated temperature. For 0.25% fibre dosage, steel fibre reinforced specimen (MS1) retained higher residual strength than polypropylene fibre reinforced counterpart (PP1) at lower elevated temperature up to 600ºC. MS1 specimen retained a residual strength as high as 83.33% against 69.78% for PP1 specimen at 300ºC. The residual strength reduced to 33.83% for MS1 and to17.74% for PP1 at temperature of 600ºC. However, at higher temperature of 900ºC, PP1 showed greater residual strength than that of MS1. When the temperature increased from 600ºC to 900ºC, the residual strength of PP1 reduced to 5.26% while steel fibre reinforced specimen MS1 failed to record any strength. Similar trend was also observed for 0.5% fibre reinforced OPC specimens. Steel fibres restrict the initiation and expansion of cracking due to their high tensile resistance which maintain higher residual strength at lower temperature [38]. With increase in the exposure temperature beyond 600ºC, more damage and cracking occur as the vapour pressure due to inner moisture of concrete cannot be released resulting in larger strength loss [38]. Pliya et al. [39] stated the loss of adherence between paste and steel fibre as the reason for complete loss of strength at high temperature. On the other hand, polypropylene fibre melts and vaporize due to its low melting point which results in microchannels in the concrete [32, 38, 40, 41]. This alleviates and releases greater vapour pressure at high temperature exposure resulting in better performance. Larger cracks and spalling of concrete were also observed in case of steel fibre reinforced OPC specimens.

Among alkali activated specimens, polypropylene fibre reinforced specimens performed better throughout the exposure temperature when compared to the steel fibre reinforced specimens at both fibre contents of 0.25% and 0.5%. However, unlike steel fibre reinforced OPC specimens, alkali activated steel fibre reinforced GMS1 and GMS2 specimens still managed to retain residual strengths of 10% and 10.8% respectively even at highest elevated temperature of 900ºC. This could be attributed to the fact the AAC specimens leads to formation of lesser amount of $Ca(OH)_2$ than those of OPC specimens [42, 43]. With regard to residual strength, fibre reinforced OPC specimens exhibited higher values at lower exposure temperatures up to 300ºC than those of AAC specimens for both fibre dosages. AAC specimens began to yield higher residual strength as the temperature increased beyond 300ºC. The decomposition of $Ca(OH)_2$ after 350ºC in OPC specimens may be the reason for such observation. Irrespective of type of fibre, 0.5% fibre dosage is found to give comparatively higher residual strength in both OPC and AAC. Moreover, polypropylene fibres could be a better option for use in higher temperature in both OPC as well as AAC specimens.

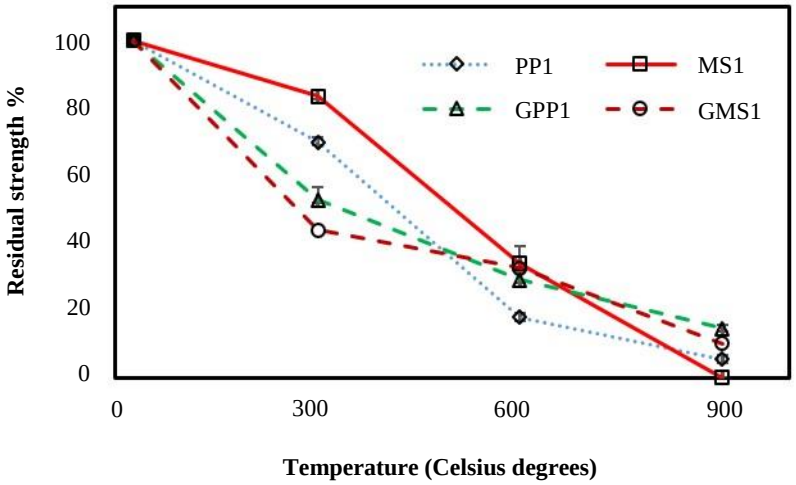


Figure 13 Relative compressive strength at elevated temperature for 0.25% fibre content

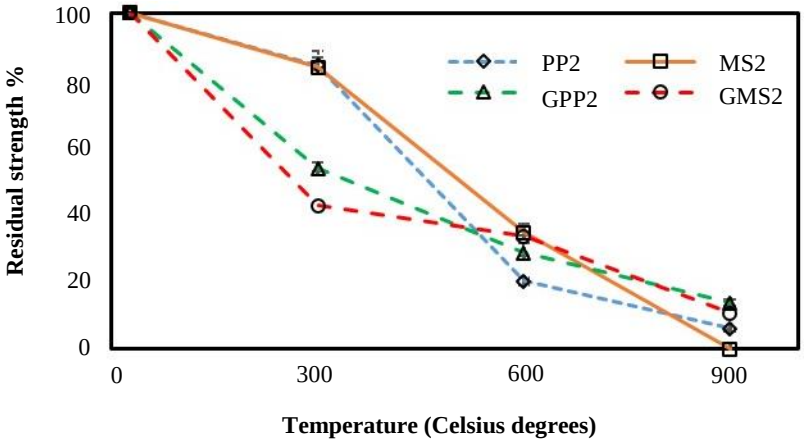


Figure 14 Relative compressive at elevated temperature for 0.50% fibre content

4. Conclusion

The binders produced by alkali activation of GGBS were found to be more resistant against water absorption than those binders produced using ordinary Portland cement. 0.5% polypropylene fibre reinforced AAC shows least water absorption indicating improved porosity with increasing fibre content. Mechanical strength increases with introduction of fibres in both OPC as well as AAC specimens. However, gain in compressive strength was highly significant for AAC with microsteel fibres while tensile strength was more prominent with polypropylene fibres.

On exposure to lower elevated temperature up to 300ºC, fibre reinforced OPC specimens retained higher residual strength than those of AAC. This can be attributed to non-decomposition of $Ca(OH)_2$ in OPC which begin at approximately 350ºC. OPC reinforced with steel fibre completely lost its strength at 900ºC due to cracking and spalling after loss of adherence between steel fibre and paste. Polypropylene fibre reinforced OPC specimens, on the other hand presented residual strength of about 5-6% at the same temperature which is due to release of vapour pressure through the microchannels formed after melting of fibres. The best performance in terms of residual strength occurred in 0.5% polypropylene fibre reinforced AAC. Up to 300ºC, both fibre reinforced OPC and AAC specimens showed rapid weight loss. Beyond 600ºC, loss in weight for fibre reinforced AAC was insignificant as compared to those of OPC counterparts. Complete spalling occurred in plain OPC specimen at 900ºC. OPC incorporated with microsteel prevent spalling though accompanied with formation of large cracks. AAC with polypropylene fibre resulted in least cracks showing good compatibility between the PP fibre and AAC paste. Reinforcement by fibre was found to be beneficial in improving its properties at elevated temperature though polypropylene fibres imparted better performance than microsteel fibres.

5. Acknowledgments

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