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RESEARCH ARTICLE - CIVIL ENGINEERING

Effects of Elevated Temperatures on Residual Properties of Concrete Reinforced with Waste Polypropylene Carpet Fibres

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Abstract In this study, the effect of waste polypropylene carpet fibres and palm oil fuel ash (POFA) on the mechanical and microstructural properties of concrete exposed to elevated temperatures was investigated. Concrete samples were exposed to high temperatures up to 800 °C then cooled to ambient temperature before tests. Four mixes containing carpet fibres (0 and 0.5%) and POFA (0 and 20%) were prepared. Mass loss, residual ultrasonic pulse velocity, compressive strength, scanning electron microscopy, X-ray diffraction and differential thermal analysis were performed to investigate the effects of carpet fibres and POFA on the performance of the concrete at elevated temperatures. The results showed that both carpet fibres and POFA were associated with a significant enhancement in the fire resistance and residual compressive strength and also eliminating the explosive spalling behaviour of the concrete at elevated temperatures. Furthermore, the role of carpet fibres and POFA is discussed through the microstructural analysis and fibre–matrix interactions as function of heat treatment.

Keywords Elevated temperatures · Concrete · Waste carpet fibres · Palm oil fuel ash · Residual performance · Microstructure

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1 Introduction

Sustainability considerations in concrete industry have led to the development of new green concrete by utilisation of waste fibrous materials. Fibres have been widely used to enhance the ductility of concrete [1]. Recently, the detection and recognition of fibres for the reinforcement and improvement of concrete have rapidly increased the need for practice in research, development and concrete industries. Several types of fibres have been adapted efficaciously in the various applications of concrete. In addition, technical developments brought forward the advancement of fibres with different materials, geometric forms and properties to increase the advantages in concrete constructions. Modern manufacturing methods and demands on fibres, which are to be used in concrete, have since been developed. Therefore, different features of fibre-reinforced concrete have been introduced to the market globally [2,3].

Fire represents one of the most severe potential risks to which structures may be subjected. The behaviour of structures exposed to elevated temperatures is mostly associated with stress distribution, cracking, spalling and surface microcracking. In some circumstances, the concrete structure is exposed to elevated temperatures and pressures throughout its service for a substantial period, for example, concrete in a reactor vessel, coal gasification, nuclear plant and other applications. The significant impacts of high temperature on concrete structure are the dehydration of cement paste, variation in water content, increase in porosity, thermal expansion and cracking, modification of pore pressure and decrease in strength and thermal spalling owing to extreme pore pressure [4–6].

A great deal of attempt has been made, and various practices have been used to manage high temperatures as well as to evaluate the residual performance of concrete struc-

tures. To develop concrete properties, fibrous materials can be added into the concrete. The purpose of such addition is to enhance its toughness, tensile and flexural strengths, resistance against impact loads and other mechanical properties [7]. Indeed, fibrous materials have exhibited good performance in developing the fire resistance capacity of concrete components [8,9]. Various kinds of fibres, either polymeric or metallic, are generally utilised in concrete mixture for their benefits [10–12]. The most common fibres used in concrete are steel, glass and synthetic fibres such as nylon and polypropylene (PP) as well as natural fibres and fibres from pre- and post-consumer wastes.

In waste streams, carpets are classified as textiles and are generated from either pre- or post-consumer products. According to Carpet Recycling UK, cited in [13], 400,000 t of carpet are sent to landfills annually. In the USA alone, approximately 1.9 million tonnes of textile waste were generated in 2007, accounting for 4.7% of the total municipal solid waste. Of this, 15.9% of the textile waste was recovered. Waste carpet fibres can be potentially used in the manufacturing of concrete as doing so is a hypothetically effective method to reduce the disposal of waste materials and at the same time decrease the amount of raw materials used in concrete industries. Moreover, the concrete manufactured would be lightweight and possess good acid and alkali resistance [14,15].

The utilisation of pozzolanic ashes as supplementary cementing materials in concrete is an effective way to develop the properties of concrete composites [16–18]. In recent periods, a great deal of attention is being focused on the potential use of pozzolanic ashes in concrete. These pozzolanic materials are used in all corners of the world for their technical, economic and ecological benefits. One of the latest inclusions in the ash group is palm oil fuel ash (POFA), which is obtained by burning palm oil husks and palm kernel shells as fuel in palm oil mills [19]. The ash, which is disposed of without any profitable return, is now considered as a valuable material with good performance in improving the strength and durability of concrete mixtures [20,21].

Due to the importance of concrete performance at elevated temperatures and in fire, several studies have been previously carried out in regards to the subject of fibre-reinforced concrete at high temperatures. It was found that some fibres can develop the residual strength properties of concrete after exposure to high temperatures. Amongst them, the inclusion of polypropylene (PP) fibres in concrete mixtures was found to perform very efficiently. Kalifa et al. [5] and Poon et al. [17] stated that steel and polypropylene fibres could be used to decrease cracking and spalling in addition to enhancing the residual strength of concrete at elevated temperatures. According to the findings, it was ascertained that most properties of concrete reduced with an increase in temperature, especially for polypropylene fibre-reinforced concrete mixtures.

Concrete has been presented to have a number of benefits when used in constructions. Nonetheless, it suffers from a main weakness, which is its high brittleness. Concrete members, when exposed to elevated temperatures, exhibit more degradation such as cracking and spalling [22,23]. A past study proposed that concrete containing fibres [24,25] as well as concrete mixtures with pozzolanic materials such as fly ash [26], silica fume [27] and POFA [19] have demonstrated satisfactory performance at elevated temperatures.

As the addition of polypropylene fibres and POFA has been recommended for the possible decreasing of spalling of concrete at high temperatures [28,29], it paves the way for the application of waste carpet fibres and POFA to develop enhanced performance of concrete at elevated temperatures. However, research on the utilisation of such waste in concrete has not yet been conducted. Taking into account the availability of the waste materials and pozzolanic activities of the ash, POFA in particular, extensive research work has been carried out in the Department of Structure and Materials of Universiti Teknologi Malaysia (UTM) to explore the potential benefits of producing sustainable building materials.

Given the aforementioned argument, the purpose of this study was to investigate the combined effects of waste carpet fibres and POFA on the performance of concrete at elevated temperatures in addition to understanding the way carpet fibres contribute to the reduction in spalling in comparison with plain concrete without any fibres. In this study, a comparison was made amongst the physical and strength properties of both concrete mixtures containing carpet fibres and plain concrete after exposed to elevated temperatures.

2 Materials and Experimental Study

2.1 Materials

Type I ordinary Portland cement (OPC), which achieved the requirements of ASTM C 150-07, was used in this research. The palm oil fuel ash (POFA) was collected from a palm oil mill in Malaysia. The raw POFA was subsequently grounded finely in a Los Angeles milling device containing ten steel bars that were 800 mm long and 12 mm in diameter for a period of 2 h for each 4 kg of POFA. The ash conformed to the requirements of BS3892: Part 1-1992 and according to ASTM C618-15, may be categorised as in-between class C and F. However, considering the source and sort, the ash was neither of class C nor F. The specific gravity and Blaine fineness of the used POFA were 2.42 and 4930 (cm^2/g), respectively. The chemical analysis of both OPC and POFA was conducted using energy-dispersive spectrometry.

Table 1 Chemical composition and physical properties of OPC and POFA

Material	Chemical composition (%)								Physical properties	
	SiO_2	Al_2O_3	FeO_3	CaO	MgO	K_2O	SO_3	LOI	Specific gravity	Blaine fineness (cm^2/g)
OPC	20.4	5.2	4.19	62.39	1.55	0.0005	2.11	2.36	3.15	3990
POFA	62.6	4.65	8.12	5.7	3.52	9.05	1.16	6.25	2.42	4930

Table 2 Properties of waste carpet and typical virgin polypropylene fibres

Fibre	Length (mm)	Diameter (mm)	Density (kg/m^3)	Tensile strength (MPa)	Melting point (°C)	Reaction with water
	20	0.45	910	400	170	Hydrophobic

Table 3 Mix proportions and the properties of the different concrete mixes

Mix	Cement (kg/m^3)	POFA (kg/m^3)	Water (kg/m^3)	Fine agg. (kg/m^3)	Coarse agg. (kg/m^3)	V_f (%)	V_f (kg/m^3)	Slump (mm)	VeBe (sec)
A1	455	–	215	840	870	–	–	210	2.3
A2	455	–	215	840	870	0.50	4.550	70	6.6
B1	364	91	215	840	870	–	–	190	3.6
B2	364	91	215	840	870	0.50	4.550	60	7.1

The obtained results along with the physical properties are given in Table 1.

Mining sand with saturated surface dry condition passing through a 4.75-mm sieve, with fineness modulus, specific gravity and water absorption of 2.3, 2.6 and 0.70%, respectively, was used as the fine aggregate. On the contrary, crushed granite with a maximum size of 10 mm, specific gravity of 2.7 and 0.5% water absorption was used as the coarse aggregate. Throughout the study, supplied tap water was used for both mixing and curing purposes. In addition, a polymer-based superplasticizer (Rheobuild 1100) at 1.0% by weight of cementitious materials was employed to increase the concrete workability. The required waste carpet fibres were collected from ENTEX Carpet Industries SDN BHD, Selangor, Malaysia. The multifilament polypropylene carpet fibres were cut into lengths of 20 mm with an aspect ratio (l/d) of 44. The general properties of the carpet fibre used are presented in Table 2.

2.2 Mix Proportions

Four concrete mixtures were prepared in two series, with and without POFA contents, in addition to having 0 and 0.5% of carpet fibres contents. Series A mixes were made with OPC only, while series B mixes were made with POFA at the replacement level of 20% by weight of cement. The water/binder (w/b) ratio of 0.47 was kept constant in all mixes. The details of the mix proportions are summarised in Table 3.

2.3 Specimen Preparation and Test Methods

For each concrete mix, 100-mm cube specimens were cast and cured for 24 h in accordance with BS EN 12390-2:2009 and BS EN 12390-3:2009. Subsequently, the cube specimens were demolded and kept in a water tank until they are required on the day of the test. After 90 days of water curing, the fully saturated concrete specimens were taken out and dried at room temperature. Prior to testing, all cube samples were weighed. The control samples were tested for ultrasonic pulse velocity (UPV) following ASTM C597-09 and the compressive strength was set at the ambient temperature of 27 °C. The concrete cubes were heated in an electric furnace for temperatures of 200, 400, 600 and 800 °C at an average rate of 3 °C/min. The temperature was increased in line with the ISO-834 fire curve. The peak temperature was maintained for a period of 1 h. The time–temperature curve of the furnace is illustrated in Fig. 1. The temperature graph revealed similar trend to those of ISO 834 and ASTM E119. Previous studies by Awal et al. [19] as well as Xio and Falker [27] presented a comparable heating configuration.

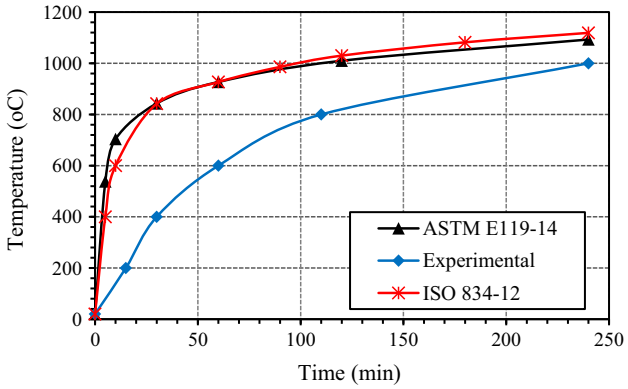

Fig. 1 Time–temperature curve of the electrically controlled furnace

The concrete samples were allowed to cool naturally in the air at the laboratory temperature of 25 ± 2°C. The residual weight, UPV values and cube compressive strength were then recorded for all specimens. Scanning electron microscopy (SEM), X-ray diffraction (XRD) and differential thermal analysis (DTA) were used to investigate the morphology and microstructure of the concrete samples at room temperature and elevated temperatures.

3 Experimental Results and Analysis

3.1 Spalling and Surface Colour

No notable explosive spalling was observed in the concrete cubes containing carpet fibres throughout the fire testing. Thus, this finding reinforced the notion that carpet fibres is able to enhance the resistance of concrete against spalling at elevated temperatures significantly. The main cause of concrete spalling at high temperatures is related to the internal pore pressure buildup, which is due to the evaporation of both free and bound water [30]. In the plain concrete specimens without carpet fibres, this inner pressure was not released and thus resulted in explosive spalling of the concrete surface.

As aforementioned, spalling was not observed in the concrete samples with carpet fibres at different temperatures. This phenomenon could be due to the low melting point of carpet fibres. Polypropylene carpet fibres melt at approximately 170 °C while spalling occurs beyond 190 °C [28,31]. When the fibres melt and are partly absorbed by the matrix, the bed of the fibres acts as an additional pathway for gases. Therefore, the fibres contribute to the formation of a network along the matrix, which subsequently permits the outward migration of gases and as a consequence, the decrease in pore pressure.

At the ambient temperature, the surface colour of the concrete samples was grey and dim grey for OPC and POFA concrete mixtures respectively, with smooth surfaces (Fig. 2). These appearances were retained up to a temperature of 200 °C. However, at 800 °C, a whitish grey colour for OPC and light grey colour for POFA concrete specimens were observed. Additionally, hairy cracks began to develop at 800 °C in both OPC and POFA mixtures for both air- and water-cooled samples. The colour variation of the concrete specimens could be attributed to the changes in the composition and texture as well as both development and crystal destruction while firing [27].

The said observation demonstrated that the variation in the surface colour of the concrete specimens had no clear relationship with the inclusion of the carpet fibres. This could be because the melting point of the carpet fibres is at a temperature of below 200 °C. However, the alterations in the concrete containing POFA were more obvious at elevated temperatures. This observation probably owed to chemical transformations, which took place in the specimens at high

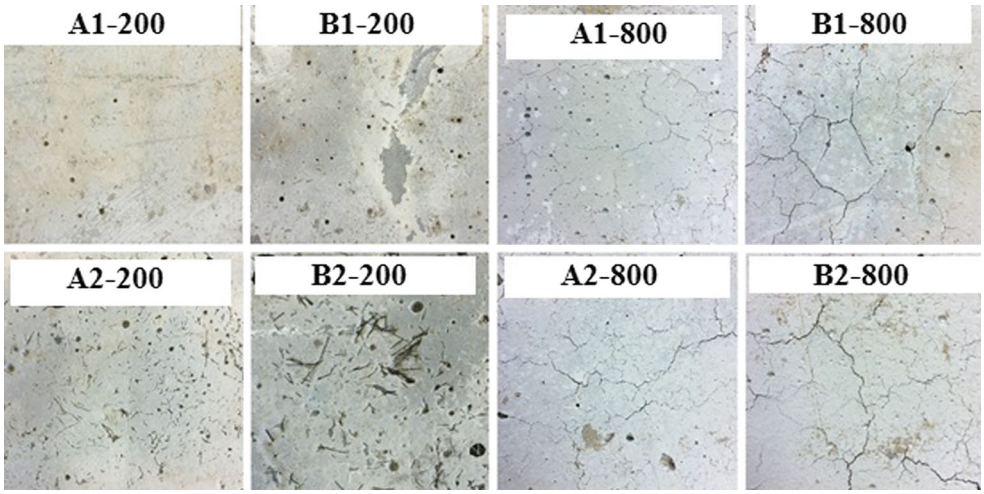

Fig. 2 Surface texture of the concrete specimens exposed to high temperatures

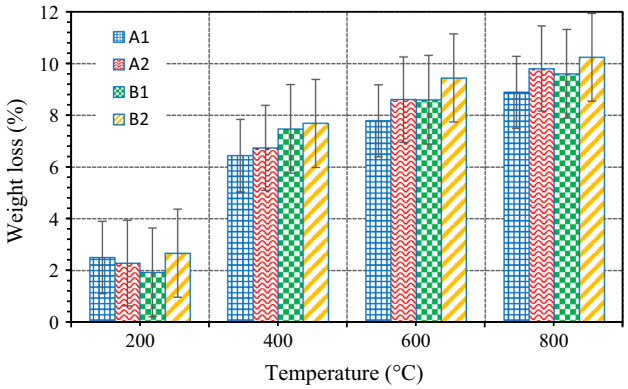

Fig. 3 Mass loss of different concrete mixtures

temperatures. The amount of Fe_2O_3 in the amorphous state of POFA was higher than that of OPC. Moreover, the iron oxide in POFA oxidised at temperatures beyond 250 °C and therefore created an appearance with a severe variety of colours, as the heating increased [32].

3.2 Mass Loss

For weight loss assessment, the weights of the concrete cubes were measured before and after the exposure to elevated temperatures. The impact of high temperature on the mass loss of both plain concrete and concrete containing carpet fibres is shown in Fig. 3. The mass loss of all the investigated specimens is expressed as a percentage of the original mass at the ambient temperature to the mass after exposure to a specific elevated temperature. Figure 3 further displays that at different temperatures, the weight loss of the concrete mixtures containing carpet fibres and POFA had the tendency to increase.

Temperature influence can be separated into three phases, in accordance with the difference in the residual mass obtained at high temperatures. In the first phase, 27–200 °C, small mass loss was observed for all mixes, as the extra amount of free water was present in the concrete samples. Given that the melting point of fibres is at approximately 170 °C, this range of temperature did not significantly affect the inner fibres in the concrete specimens. Nonetheless, the outer fibres, which were exposed to temperatures up to 200 °C, melted. In the second phase, where the temperature increased from 200 to 400 °C, the weight loss was considerable due to the complete melting of the fibres as well as the release of both gel and capillary water.

Beyond 400 °C, the rate of weight loss slowed down moderately. The highest loss of 10.24% was found in the concrete specimens containing 0.5% fibres and 20% POFA. The mass loss in POFA-based concrete mixtures could be due to the higher moisture content absorbed by the ash. In theory, the mass loss in the concrete samples at high temperatures could be attributed to the decomposition of calcareous aggregates, liberation of carbon dioxide (CO_2) and sloughing off of the concrete surface, which therefore altered the mechanical properties of the concrete [33,34]. Xiao and Falkner [27] previously interpreted the structural integrity of fibre-reinforced concrete in terms of mass loss. They perceived that the cement matrix losses its binding properties owing to the vaporisation of free water in the calcium silicate hydrate (C–S–H) gel and decomposition of calcium hydroxide $Ca(OH)_2$.

3.3 Residual Ultrasonic Pulse Velocity (UPV)

The ultrasonic pulse velocity (UPV) test is a nondestructive test that measures the quality and homogeneity of concrete specimens to determine the existence of pores and cracks. Figure 4 displays the variations in the UPV of the concrete mixtures containing carpet fibres and POFA exposed to the designated temperatures. It can be seen that the polypropylene carpet fibres produced no notable effects on the UPV values of the concrete. At the ambient temperature, the UPV values of the concrete mixtures were high while at higher temperatures, lower values were recorded in all the test samples. At room temperature, the UPV values of OPC concrete, for instance, were 4570 and 4580 m/s for 0 and 0.5% carpet fibres content respectively, which were excellent in terms of concrete quality [35]. UPV values of 4559 and 4540 m/s were also found in POFA concrete samples.

Higher UPV values of between 4100 and 4550 m/s were found at temperatures of 200–600 °C in the specimens containing carpet fibres for both OPC and POFA mixtures in contrast to that of the plain concrete mixture without any fibres, which could be classified as good quality concrete, as stated by Neville [35]. However, at a temperature of beyond 600 °C, the UPV values recorded for the concrete mixes containing carpet fibres significantly dropped.

The decrease in the UPV values could be due to the melting of the fibres, which creates an additional porous network along the bed of the melted fibres. In theory, it could also be a result of the deterioration of microstructure of the matrix. Awal et al. [19] and Zheng et al. [36] indicated that the said form of variation is due to the degradation of C–S–H at temperatures beyond 450 °C, which increases the volume of pores in the concrete and therefore reducing the UPV values of the concrete specimens.

3.4 Residual Compressive Strength

The experimental results and the ratio of residual compressive strength to original compressive strength of the concrete mixtures at the ambient temperature and upon heating to 200, 400, 600 and 800 °C in addition to exposure to air-cooled regime are illustrated in Figs. 5 and 6. The results showed that the room temperature compressive strength of

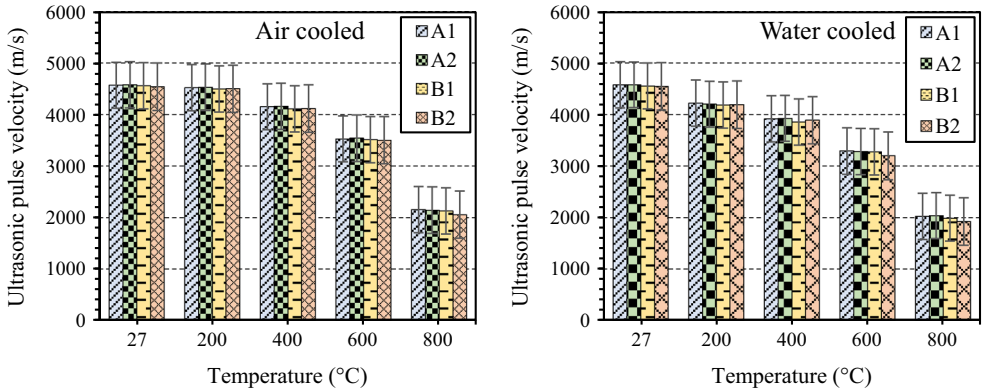

Fig. 4 Variation in UPV values of the concrete mixtures exposed to high temperatures

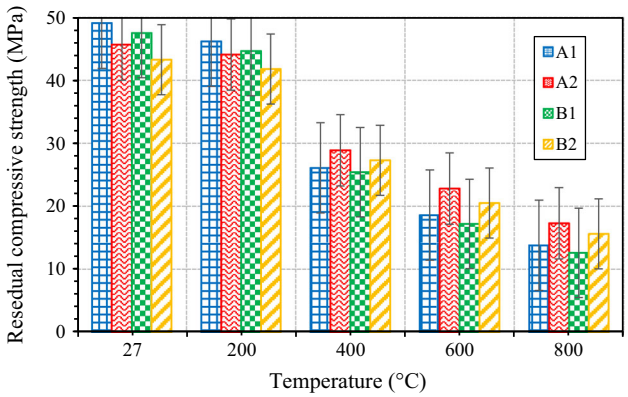

Fig. 5 Residual compressive strength of the concrete mixtures

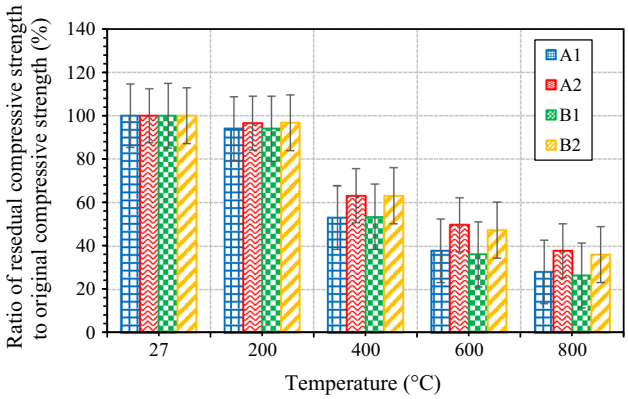

Fig. 6 Ratio of residual compressive strength to original compressive strength

the concrete decreased with the addition of the carpet fibres. Comparing the value of the control mix (concrete without any fibres or POFA), the addition of fibres at 0.5% volume fraction decreased the compressive strength by 6.9%. Further decrease in compressive strength of 3.3% in contrast to OPC concrete was observed in the concrete containing 20% POFA. In addition, in the fibrous mixtures containing POFA and 0.5% fibre, the compressive strength value decreased by 5.4% in comparison with that of OPC concrete, which possessed the same amount of fibres. The said reduction was attributed to the slow hydration and low pozzolanic activity of POFA, which negated the increase in compressive strength [11].

After heating up to 200 °C, the cube compressive strength of all four mixtures reduced by 3.35–9.76% compared to the strength values at the ambient temperature. The strength loss in this phase could be attributed to the initial moisture loss in the concrete mixtures. Higher compressive strength losses were observed in both the OPC and POFA concrete specimens without carpet fibres. Given this, the positive effects of carpet fibres on the residual compressive strength of concrete mixtures was clearly shown. As aforementioned, carpet fibres melt at temperatures between 160 and 180 °C. As such, the melted fibres, which are in liquid form, fill the holes and subsequently contribute to better performance under loads. The high residual compressive strength of the concrete mixtures containing carpet fibres was therefore attributed to its dense microstructure in comparison with that of the plain concrete [24].

The significant influence of moisture in the concrete specimens at elevated temperatures was therefore established [28]. For partial loss of moisture until 200 °C, owing to the vaporisation of free water, the loss in compressive strength was not significant. However, with full water loss, the compressive strength dropped sharply at 400 °C. Furthermore, beyond 400 °C, the compressive strength loss became gradual with the increase in temperature for all mixes. The steady degradation of compressive strength could be a result of the complete melting process of fibres as well as slow evaporation of chemically bound water owing to the disintegration and dehydration process of C–S–H gel and decomposition of $Ca(OH)_2$, which occurs at temperatures beyond 400 °C in concrete [5].

For higher temperatures, the residual compressive strength of the concrete mixtures containing carpet fibres was higher

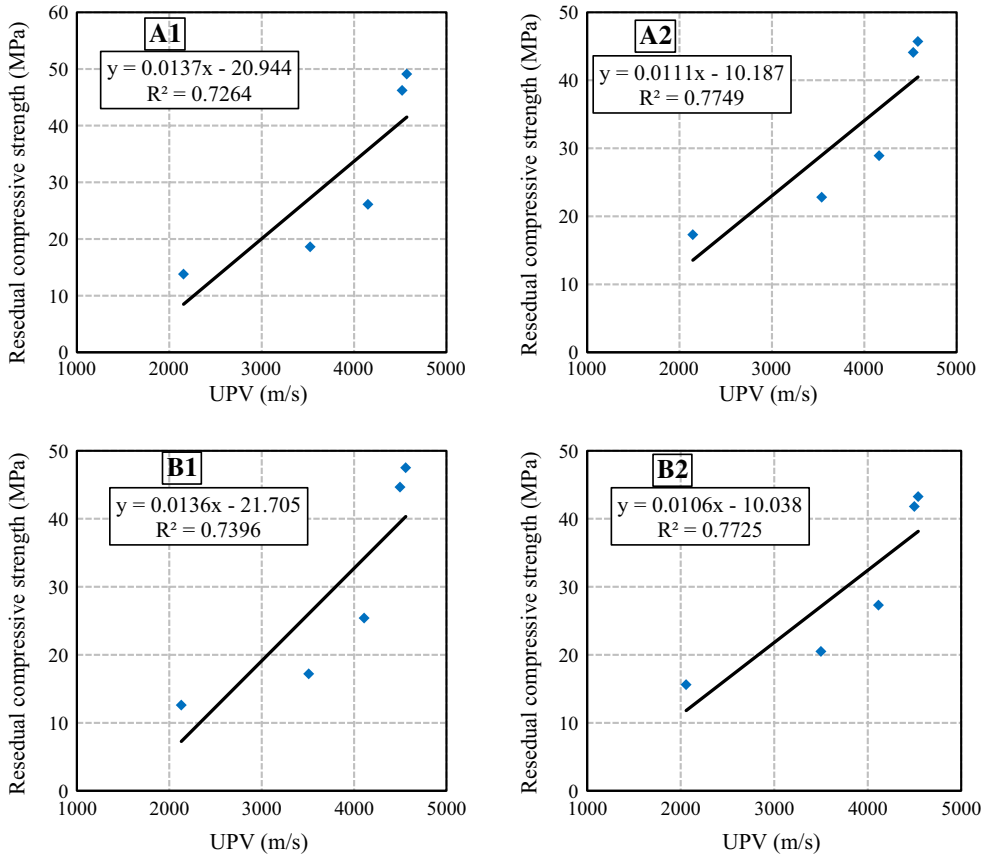

Fig. 7 Correlation amongst the residual UPV and compressive strength

than that of OPC and POFA mixtures without any fibres. This was due to the presence of the fibres that resulted in the reduction of spalling on the concrete specimens. Moreover, at elevated temperatures, the addition of discontinuous carpet fibres in the concrete mixtures decreased the uneven propagation of macrocracks and thus supported a ductile performance. Accordingly, the fibres were capable of providing adequate loads to repress cracks opening and redistribute the stresses against the neighbouring matrix [27,28]. In addition, the concrete mixtures containing carpet fibres were more ductile than that of the plain concrete with a slow decrease in strength. Therefore, the results indicated that the addition of the carpet fibres resulted in the increase of ductility of concrete, with a higher energy absorption and a well-distributed cracking.

In their study, Xiao and Falkner [27] ascertained that the residual compressive strength of concrete mixtures containing polypropylene was slightly higher than that of the mixtures without fibres. They observed that PP fibres melt at high temperatures and create networks to release thermally induced pressures and consequently, avoid excessive loss of strength. Similar results were also reported by Behnood and Ghandehari [24].

3.5 Relationship Amongst the Residual Compressive Strength and Ultrasonic Pulse Velocity

It was observed that the ultrasonic pulse velocity (UPV) values could be correlated with the corresponding residual cube compressive strength. Figure 7 displays a good relationship amongst the residual compressive strength and UPV values of all four concrete mixtures at high temperatures. To explain further, Fig. 7 illustrates the relationships between the compressive strength and UPV values of the concrete mixtures containing carpet fibres and POFA.

The obtained residual cube compressive strength values were used as a response factor with the UPV values as their predicator parameter. To correlate the experimental data, linear regression method was applied, resulting in Eqs. (1)–(4), with R^2 values of between approximately 0.72 and 0.77 for all samples, which signified good confidence for the relationships. They are as the following:

$$f_{\mathrm{rcu}} = 0.0137V - 20.944 \quad \left(R^2 = 0.7264\right) \tag{1}$$

$$f_{\mathrm{rcu}} = 0.0111V - 10.187 \quad \left(R^2 = 0.7749\right) \tag{2}$$

$$f_{\mathrm{rcu}} = 0.0136V - 21.705 \quad \left(R^2 = 0.7396\right) \tag{3}$$

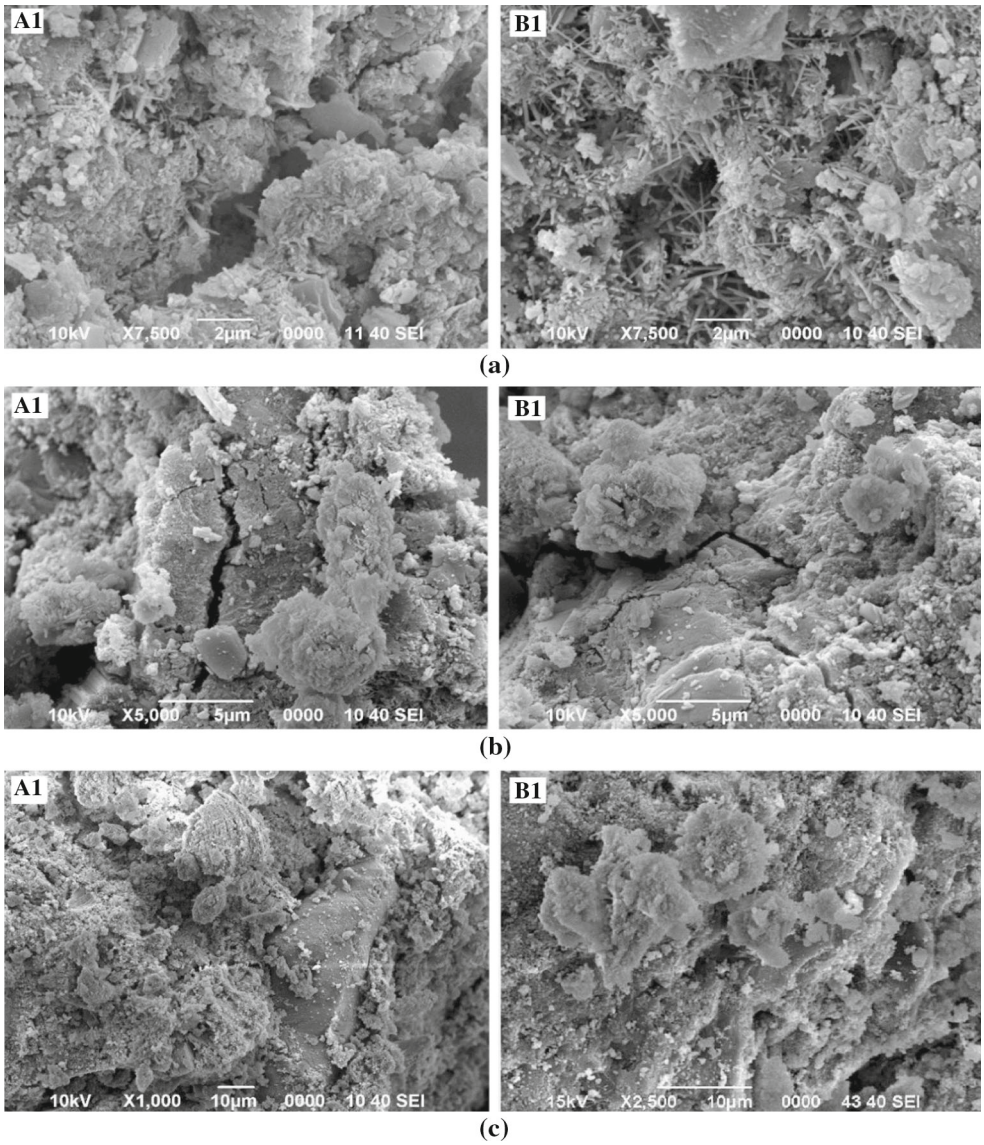

Fig. 8 SEM micrographs for plain OPC (*A1*) and POFA (*B1*) concrete specimens at **a** 27 °C; **b** 200 °C; and **c** 800 °C temperatures

$$f_{\mathrm{rcu}} = 0.0106V - 10.038 \quad \left(R^2 = 0.7725\right) \tag{4}$$

where f_{rcu} is the residual cube compressive strength (MPa) and V signifies the residual UPV (m/s) at high temperatures.

The empirical parameters of the equations attained in this study were almost comparable to those stated by Suhaendi and Horiguchi [37] for concrete containing polypropylene fibres and Awal et al. [19] for plain concrete containing POFA. The correlations in this study showed that the application of UPV measurement could be applied in the inspection of the properties of fire-damaged concrete in terms of compressive strength in a faster and more efficient way. However, the establishment of solid correlations would first require more essential experimental data from the process.

3.6 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) investigations demonstrated different variations in the morphology of OPC and POFA concrete mixtures with and without carpet fibres at designated temperatures. Figures 8 and 9 reveal the SEM of unheated and heated concrete specimens exposed to 200 and 800 °C. The SEM of OPC and POFA concrete mixtures at the ambient temperature (27 °C) showed the C–S–H gel formation and a continuous structure without microcracks and pores. As seen in Fig. 8a, on the 90-days curing period, the C–S–H gel was more evenly spared in POFA concrete in comparison to OPC. The finely spared of C–S–H gel and development of extra C–S–H gel due to the consumption of portlandite by pozzolanic action of POFA result in better per-

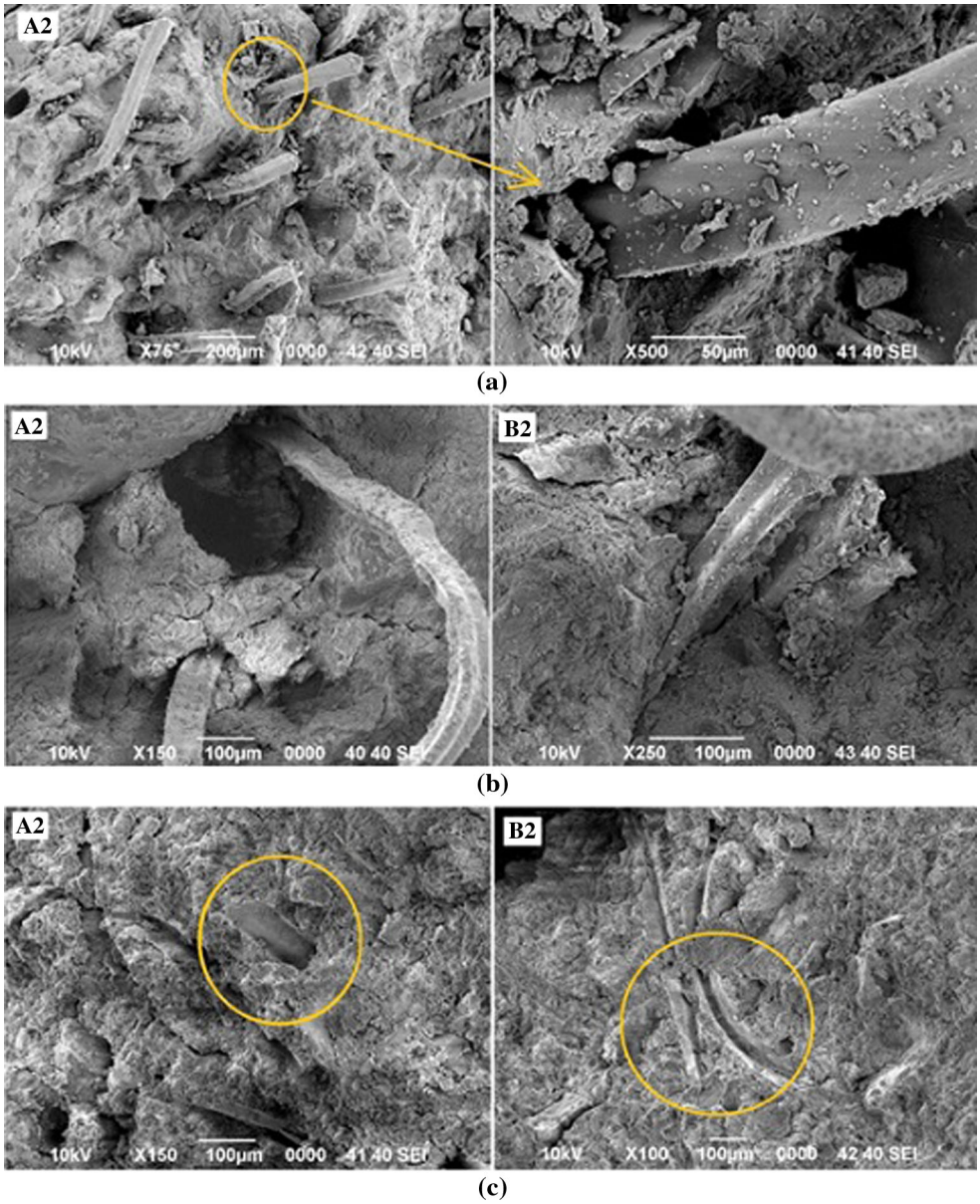

Fig. 9 SEM micrographs of the bonding interface between carpet fibres and matrix as well as melted fibres at **a** 27 °C; **b** 200 °C; and **c** 800 °C temperatures

formance in the concrete mixtures. This owed to the fact that POFA modified the concrete matrix through the pozzolanic reaction and reduced the $Ca(OH)_2$ content [19].

Figure 8b shows a slight number of microcracks, which were detected at 200 °C, while a fairly large amount of microcracks occurred in the cement matrices at the temperature of 800 °C. At the latter temperature, the OPC and POFA matrices turned into an amorphous structure, and thus, large amount of cracks appeared throughout the concrete specimens as illustrated in Fig. 8c. It can be seen that at elevated temperatures, the matrices of POFA was more compact than that of OPC. However, at the highest temperature of 800 °C, the microstructures of all specimens were extremely damaged, which led to the deterioration of C–S–H. The results of the present work were in agreement with that described by Noumowe [28].

Figure 9 displays the general views of the carpet fibres dispersed in unheated concrete mixtures. SEM analysis demonstrated that the carpet fibres acted as bridges across cracks and pores. It was also shown that the fibres were tightly wrapped by the C–S–H gels. Figure 9a also reflects a strong bond between the carpet fibres and cement matrix. The SEM image further revealed that the carpet fibres along with cement paste provided a strong interfacial bonding, which

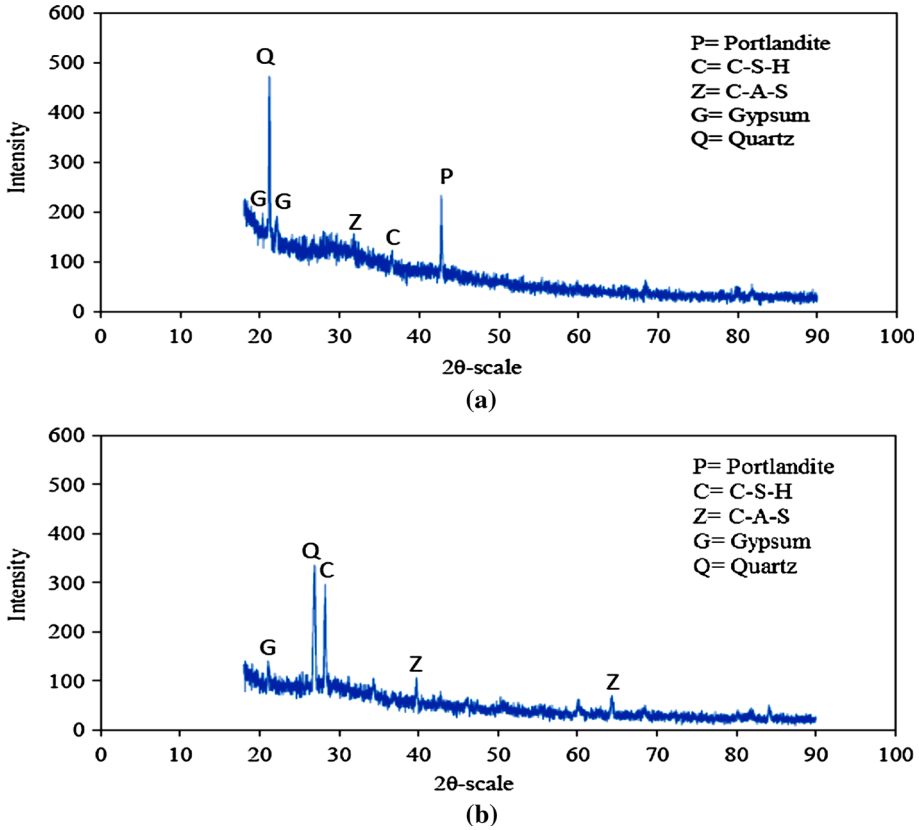

Fig. 10 XRD patterns of **a** OPC and **b** 20% POFA paste at the ambient temperature

resulted in smaller crack size on the interface. In addition, Fig. 9b shows the concrete containing carpet fibres after exposure to high temperatures. At the ambient temperature, it can be seen that the fibres had star cross section. However, at 200 °C, the carpet fibres had lost their solid structure in both OPC and POFA concrete mixtures.

A significant change in the bond between the carpet fibres and cement matrix of the concrete mixtures upon exposure at 200 °C was therefore found. The said finding could be attributed to the formation of the microcracks and therefore reduction in the bonding between the fibres and cement matrix [28]. Upon heating the concrete specimens up to 800 °C, the carpet fibres melted and evaporated and thus formed an additional network in the mixture that could act to release high internal pressures. Figure 9c presents the effects of the melted fibres. The use of PP fibres clearly affected the porosity of the concrete at elevated temperatures. In addition, it could even reduce the pore pressure inside the concrete [18].

3.7 X-Ray Diffraction (XRD)

The results of XRD of the OPC and POFA concrete mixtures exposed to high temperatures of 27, 200 and 800 °C are illustrated in Figs. 10, 11 and 12. The XRD analysis shows that at the high temperatures the composition of concrete was significantly affected by fire. From the data given in Fig. 11, it can be seen at 200 °C, the presence of ettringite and gypsum in both OPC and POFA specimens. The intensity of quartz and C–S–H was significantly lower than those of unheated specimens shown in Fig. 10. Portlandite was at very low rate in OPC specimen and almost negligible for POFA matrix. At 200 °C, the XRD peaks are comparatively lower than that of unheated specimens which indicates a decomposition of concrete at high temperatures. The breakdown of the hydration products at 200 °C has slight impact on the interface performance. Furthermore, the mechanical properties of FRC are not significantly affected [32,40].

Figure 12 illustrates the XRD analysis for the OPC and POFA concrete specimens at temperature of 800 °C. The dehydroxylation of the calcium hydroxide can be observed, which resulted in full collapse of the concrete composition and degraded the calcium hydroxide from the matrix and also reduction in the intensity of C–S–H gel. The reduction was related to the dehydroxylation of portlandite in the cement matrix which is higher in OPC specimens. Therefore, this degradation led to reduction in performance of concrete at elevated temperatures and loss in strength. However, due to the lower amount of portlandite in the POFA matrix, it can be seen that the intensity of components are significantly higher than that of OPC matrix. It indicates that the presence of POFA in matrix can significantly improve the performance of concrete at elevated temperatures and prevent the dehydroxylation of cement matrix [19].

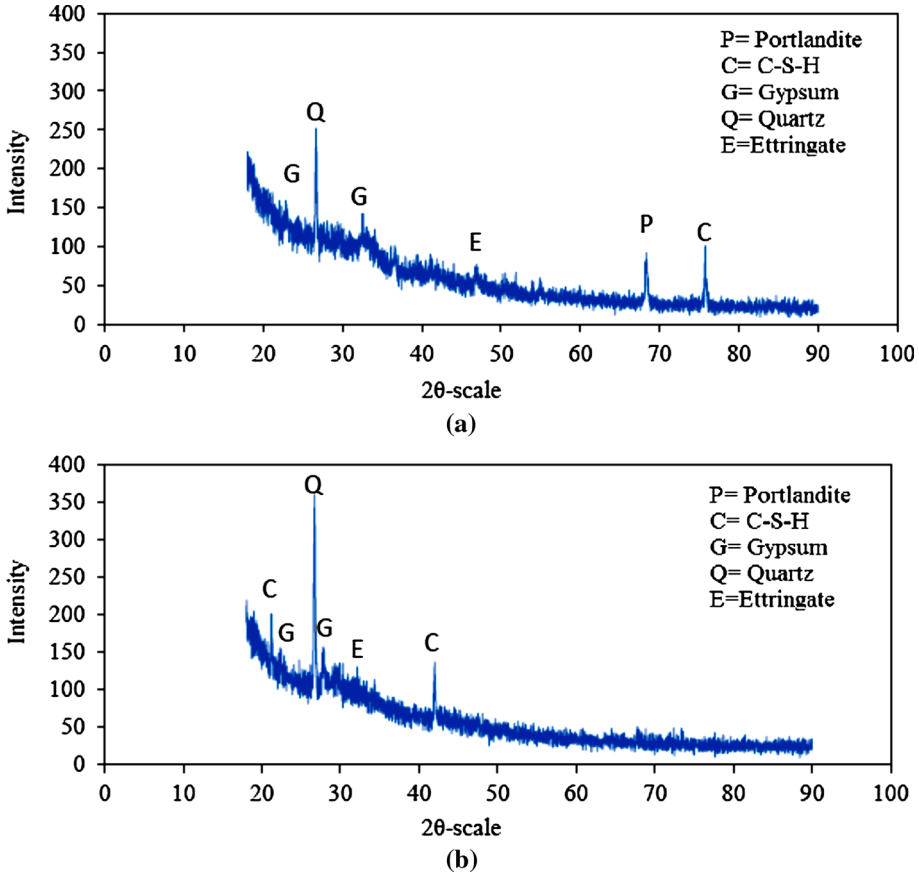

Fig. 11 XRD patterns of **a** OPC and **b** POFA specimens after being exposed to temperature of 200 °C

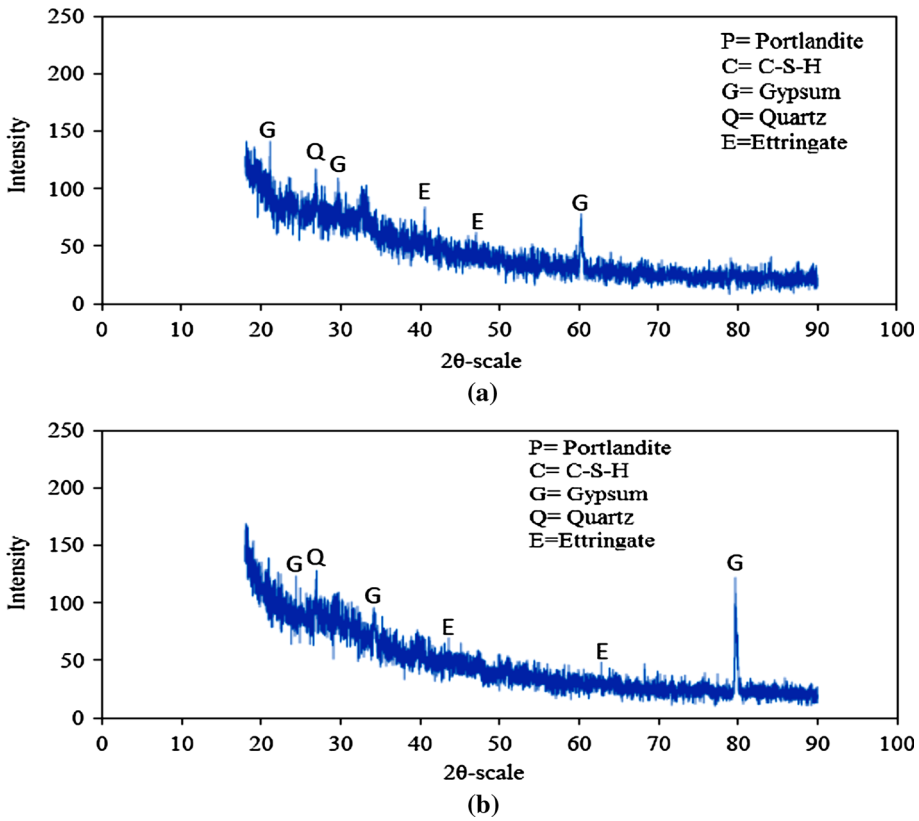

Fig. 12 XRD patterns of **a** OPC and **b** POFA specimens after being exposed to temperature of 800 °C

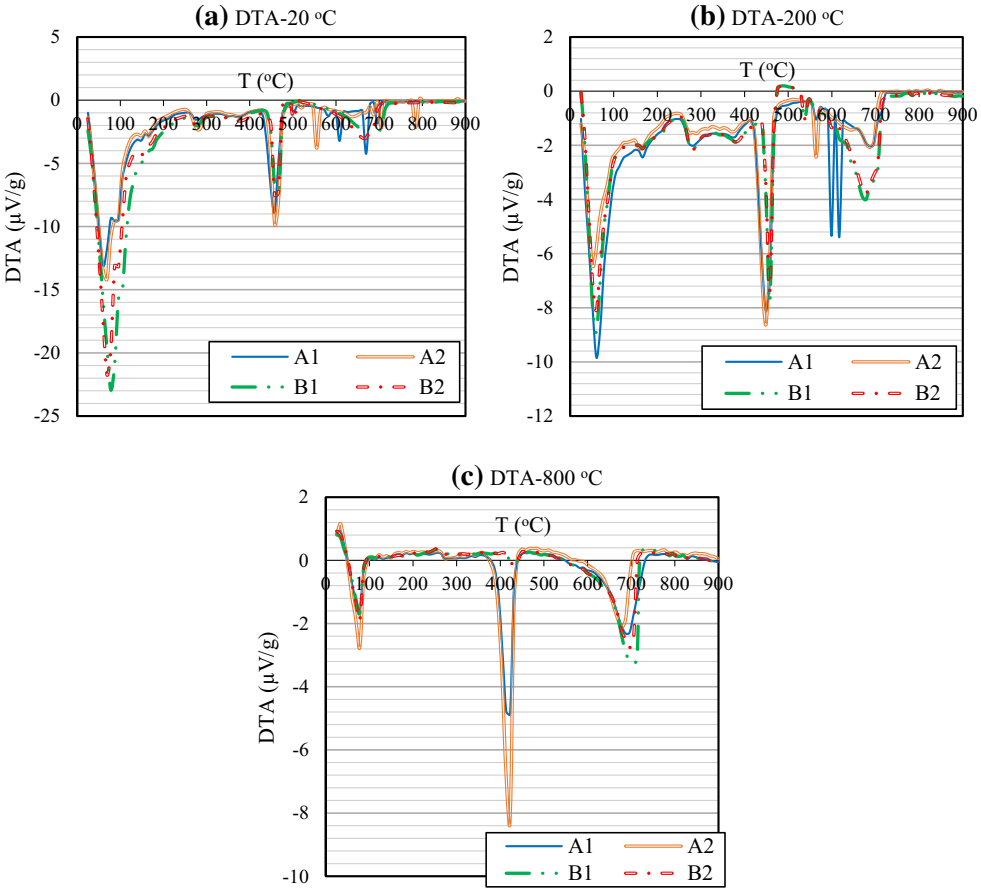

Fig. 13 DTA results for the concrete mixtures at temperatures of **a** 20, **b** 200 and **c** 800 °C

3.8 Differential Thermal Analysis (DTA)

When a composite material such as concrete is exposed to elevated temperatures of between 100 and 900 °C, numerous chemical and physical phenomena occur. The reactions take place throughout the heating of the concrete mixtures. Figure 13a reveals DTA results of the concrete specimens at the ambient temperature. It can be seen that some physical phenomena had occurred in the temperature series between 80 and 180 °C. The evaporation of water in the cement matrix below 110 °C, C–S–H dehydration as well as shrinkage and melting of fibres at approximately 170 °C were the most important issues during the said phase. The observations made in this study were comparable to those found by Noumowe [28] and Fares et al. [32].

Few endothermic peaks were observed in the unheated specimens, which were 85–130, 470, 555, 660 and 750 °C. The detected peaks of heat flow were related to the temperatures of phase transition of the hydrates in the cement paste as well as the melting of carpet fibres. There was no significant distinction between OPC and POFA concrete mixtures comprising waste carpet fibres for the entire heating process. A dual peak at 80–130°C was also observed, which was attributed to the vanishing of free and bound water in hydrates like C–S–H gel [32].

Noumowe [28] stated that the small variation of heat flow between 170 and 480 °C could be attributed to a continuous dehydration of the C–S–H gel and melting of PP fibres. According to Sun and Xu [38], the said difference owes to the decomposition of fibres at 200–300 °C, release of free water of hydrates, first phase of dehydration and failure of C–S–H gel structure. Sun and Xu [38] also found that between 600 and 700 °C, the hydroluminate decomposes and forms $\beta - C_2S$ and at approximately 720 °C, the calcium carbonate ($CaCO_3$) decomposes, thus permitting CO_2 to liberate from the concrete. There were some differences observed in OPC and POFA concrete mixtures during the heating process. Particularly, at below 110 °C, both B1 and B2 mixtures containing POFA offered a higher peak than both A1 and A2 specimens with OPC only. This could be attributed to the lower unit weight of POFA compared to OPC that resulted in the increased volume of mixtures and therefore, more free water in the specimens [19].

Figure 13b, c displays DTA and TGA results of the concrete specimens exposed to high temperatures of 200 °C and 800 °C. Comparing the results of both unheated and heated

specimens, several peaks were observed to have changed. Figure 13b reveals that after heating the concrete specimens up to 200 °C, bound water of C–S–H, free water, ettringite and hydrated calcium monocarboaluminate were removed [4,28]. For heating up to 800 °C, the peak at 440 °C in Fig. 13c decreased notably in contrast to that of at 20°C, particularly for POFA concrete mixtures. The reduction was associated with the dehydroxylation of portlandite in the cement matrix. Moreover, the peak at 680 °C was related to the allotropic conversion of quartz-α in quartz-β kept unchanged. As this transformation was reversible, quartz-α reformed after cooling [39,40].

4 Conclusions

Based on the experimental results and observations made, the following conclusions could be drawn:

- The mass loss in concrete mixtures with carpet fibres was higher. The mass loss in the concrete composites could be classified into three different classes. For up to 200 °C, the mass loss was moderately small, as the fibres did not fully melt. This scenario became more significant when the temperature increased to 400 °C. Beyond 400 °C, the rate of weight loss slowed down.
- At the ambient temperature, the ultrasonic pulse velocity (UPV) values of 4200–4600 m/s were observed and could be categorised as good quality concrete for specimens with carpet fibres and POFA. However, when exposed to high temperatures, the residual UPV of both concrete mixtures with and without fibres dropped slightly.
- The concrete mixtures containing carpet fibres exhibited better performance in terms of ductility due to the bridging action of the fibres than that of the plain concrete.
- At the room temperature, the compressive strength of the fibre matrix decreased with the addition of carpet fibres due to the lower strength properties of PP fibres. Unlike mass loss, the overall compressive strength of both OPC and POFA concrete composites containing carpet fibres was not significantly affected, particularly at temperatures beyond 400 °C. The decrease in strength, nevertheless, was more prominent in mixtures heated up to temperatures beyond 600 °C.
- SEM images showed that cracks had the tendency to appear more regularly in OPC concrete specimens in comparison with POFA concrete mixtures when exposed to elevated temperatures. SEM images also presented clear indications of the carpet fibres melting and formation of an additional porosity. A notable change in the bond between the carpet fibres and cement matrix of concrete mixtures after exposure at 200 °C was found due to the formation of microcracks after evaporation free water from the specimens. At 800 °C, there was a noteworthy modification between the porosity of the carpet fibres concrete and plain concrete. This could be a consequence in lesser vapour pressure in the concrete mixtures containing fibres exposed to high temperatures. This would therefore result in a lower risk of concrete spalling in case of an accident.
- DTA analyses of concrete showed little difference between both the unheated and heated concrete specimens. At the temperature range of between 20 and 130 °C, no sensible degradation was observed. Only the departure of both free and bound water contained in C–S–H were observed, which subsequently led to a small variation of the porosity. Between temperatures of 130 and 400 °C, cracks were observed, particularly within the paste due to the fibres melting as well as the evaporation of water of C–S–H gel. Beyond 400 °C, the mass loss decreased. The reduction was less profound with respect to the concrete comprising of fibres in contrast to the plain concrete mixtures for both OPC and POFA content.
- The XRD results showed the variation in the mineralogy of the concrete specimens at elevated temperatures. It is due to the decomposition of concrete at high temperatures.

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