



Thermal behavior and post-heating fracture characteristics of polypropylene microfiber-reinforced geopolymer binders

Mohammad R. Irshidat^{1,*}, Nasser Al-Nuaimi, Mohamed Rabie

Center for Advanced Materials (CAM), Qatar University, P.O. Box 2713, Doha, Qatar

ARTICLE INFO

Keywords:

Geopolymer
Polypropylene fiber
Elevated temperatures
Thermal
Fracture
Mechanical

ABSTRACT

This paper investigated the post-heating behavior of fly ash-based geopolymer binders reinforced with polypropylene (PP) microfibers. Geopolymer binders with different fiber contents including 0%, 0.05%, 0.1%, and 0.2% by fly ash weight were prepared and exposed to elevated temperatures of 200 °C, 400 °C, and 600 °C. The effect of fiber reinforcement on the mechanical strengths of unheated binders, residual fracture characteristics of heat-damaged binders, microstructure deterioration, and thermal properties of geopolymer specimens were investigated. The results revealed that PP microfibers had neglected effect on the compressive strength of the binders, but clearly enhanced their flexural strength. Maximum enhancement of 17% was observed in the flexural strength of binders with 0.05% PP fibers. Using PP microfibers enhanced the residual compressive but not the flexural strength of the heat-damaged binders. The fiber-reinforced specimens had higher residual compressive strength compared to the corresponding fibreless ones when heated up to 500 °C. The modulus of elasticity and the toughness of the binders decreased with heating. Fiber-reinforced specimens showed higher reduction in the toughness but lower reduction in the modulus due to heating compared to the fibreless specimens. Using PP microfibers reduced the thermal conductivity of geopolymer binders. The reduction due to the fiber addition was less pronounced for heated specimens compared to unheated ones.

1. Introduction

Alkali-activated or geopolymer binders are novel materials that have attracted a lot of attention for various construction applications. They are mainly produced by activating aluminosilicate source materials such as fly ash with alkali activators such as sodium or potassium hydroxide. With their low CO₂ footprint, comparable mechanical characteristics, high thermal resistance, and good durability, geopolymer composites represent environmental-friendly alternative to the OPC [1-4]. However, similar to OPC, geopolymer binders show brittle behavior and own low flexural and tensile strengths [5,6]. One of the common and effective way to enhance the flexural strength and toughness thus reduce the brittleness of the binders is to utilize short fibers in the mix. Addition of micro fibers in brittle matrices represents an efficient practice to mitigate the crack propagation thus improve the fracture toughness of the matrix when exposed to the load [7-9]. Different types of fibers have been used as reinforcement in cementitious and geopolymer composites to enhance their flexural capacity and toughness [7,8,10-12]. In this regards, polypropylene (PP) microfibers with their lightweight,

corrosion resistance, easy dispersion, and relatively low cost represent an attractive candidate [13-17].

The effect of PP microfibers on fresh and hardened properties of geopolymer composites has been examined extensively by previous studies. Nguyen et al. [18] reported that adding PP fibers to alkali-activated ladle slag mortars enhanced their fracture toughness, fracture energy, and flexural strength. Puertas et al. [19] reported that incorporating PP fibers in alkaline activated mortars improved their fracture resistance but not their mechanical strengths and modulus of elasticity. Bhutta et al. [20] reported that adding PP microfibers into geopolymer binders reduced their workability, increased their porosity, and insignificantly enhanced their mechanical strengths. Ranjbar et al. [9] showed that the incorporation of small amount (0.5 vol%) of PP fibers reduced the drying shrinkage and flexural strength of the geopolymer composites, but increased their energy absorption. Wang et al. [21] reported that the addition of PP fibers in the range of 0–1 wt% enhanced the thermal conductivity and the moisture absorption of lightweight fly ash-based geopolymer concrete. Mohseni [16] reported that using PP fibers enhanced the flexural strength of the metakaolin-

* Corresponding author.

E-mail address: mirshidat@qu.edu.qa (M.R. Irshidat).

¹ On leave from Jordan University of Science and Technology (JUST), Jordan.

Table 1
Chemical composition of fly ash.

Oxide	(%)
SiO ₂	49.90
Al ₂ O ₃	17.10
CaO	11.80
Fe ₂ O ₃	7.83
MgO	4.90
SO ₃	0.42
K ₂ O	0.28
Na ₂ O	0.14
Cl	0.01
LOI	7.62

based geopolymer concrete. Sukontasukkul et al. [22] reported that 0.5 vol% was the optimum PP fiber content of FA-based geopolymer mortar in terms of compressive strength. Chindaprasirt et al. [23] reported that 0.5% and 1.0% by weight of FA were the optimum fiber content for compressive and flexural strengths of high calcium FA geopolymer paste, respectively.

On the other hand, the performance of any material when exposed to elevated temperatures is significant, especially when it is to be utilized as construction material for structural application. Due to their inorganic framework, previous studies showed that geopolymer composites exhibited good thermal stability and fire resistance [24]. Jiang et al. [24] investigated behavior of geopolymer paste made of different fly ash classes when exposed to elevated temperatures. Their results showed that Class F fly ash-based geopolymer pastes had better mechanical properties than Class C when the heating temperature was below 500 °C, whereas the later showed better performance after being exposed to higher temperatures (>800 °C). Colangelo et al. [25] investigated the thermal behavior and stability of geopolymers mortars when exposed to elevated temperatures between 150 °C and 550 °C. Their results showed that the geopolymer mortar retained acceptable strength and remained thermally stable. Çelikten et al. [26] reported that the geopolymer mortar made of Class F fly ash were more durable at high temperatures up to 800 °C compared to the OPC mortar. Aygörmez et al. [27] reported that the compressive and flexural strengths of metakaolin-based geopolymer composites were reduced when exposed to elevated temperatures in the range of 600 °C to 900 °C. According to their results, the reduction in flexural strength was higher than that in the compressive

strength.

The aforementioned literatures indicated that geopolymer composites had good thermal behavior and fire resistance. In addition, positive impact of microfibers on the mechanical properties of the geopolymer composites has been reported. Reinforcing geopolymer binders with fibers has been able to provide higher resistance to crack formation and propagation thus overcome their brittleness issue. However, few studies investigated the influence of these fibers on the behavior of such composites when exposed to elevated temperatures. The novelty of this research is that it will help to understand the post-heating performance of fiber-reinforced geopolymer composites, and to explore the role of the fibers in mitigating the deterioration of the binder microstructure. The behavior of fly ash-based geopolymer binder with different dosages of PP microfibers ranged from 0.05% to 0.2% (by fly ash weight) were investigated at room temperature as well as after exposed to elevated temperatures up to 600 °C. The residual mechanical strengths and fracture characteristics of the binder were investigated after heating. In addition, the effect of the fibers on the thermal behavior of the geopolymer binders were investigated through thermal conductivity and thermogravimetric analysis. Finally, the microstructure deterioration of the binders due to heating was investigated using scanning electron microscopy (SEM) imaging.

2. Experimental program

2.1. Materials

The geopolymer binder used in this study was prepared using source materials, alkaline solution, silica sand, and superplasticizer. Class F fly ash received from local company was used as source material. Its moisture content and density were equal to 0.5% and 2.23 g/cm³,

Table 2
Geopolymer mix design (kg/m³).

Mix No.	Fly ash	Sand	NaOH (10 M)	Na ₂ SiO ₃	SP	PP fibers
M1	711	1956	171	256	7	–
M2						0.356
M3						0.711
M4						1.422

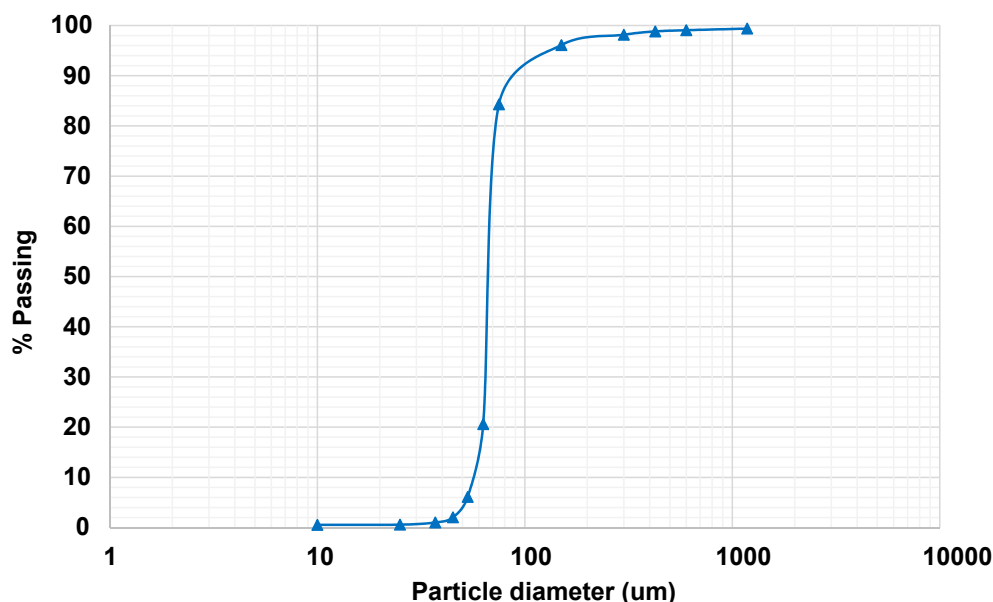


Fig. 1. Particle size distribution of fly ash used in this study.

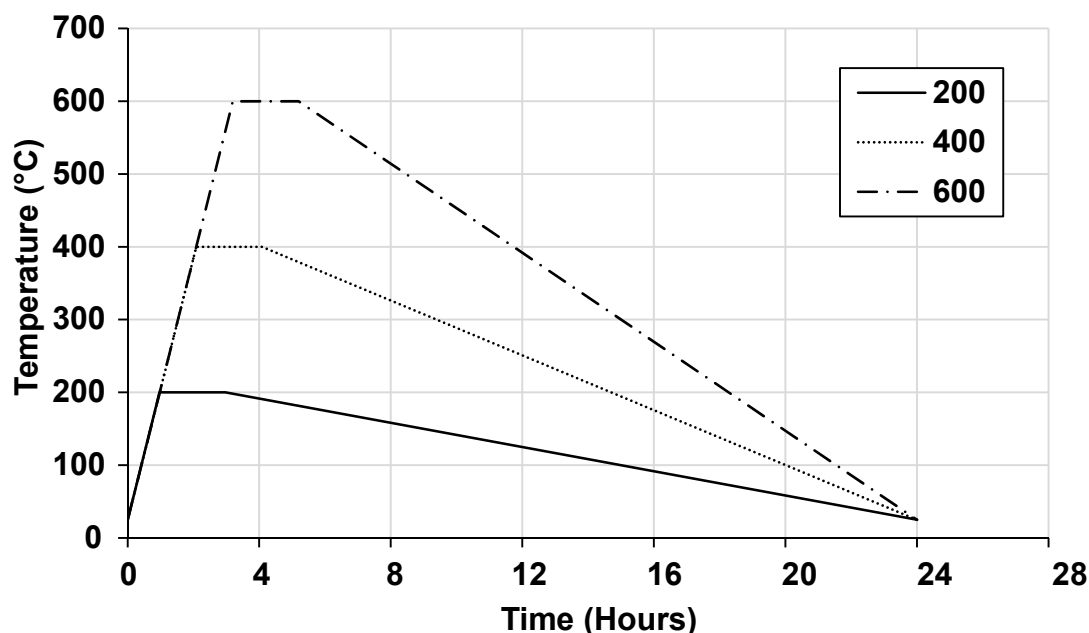


Fig. 2. Temperature-time profile.

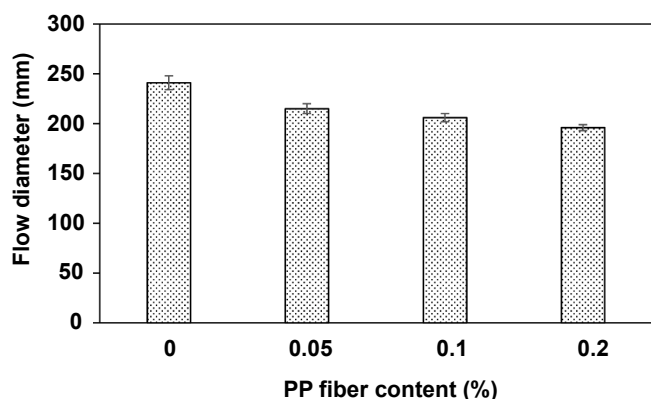


Fig. 3. Flow table results of geopolymer binder with PP-fibers.

respectively. The chemical composition (ASTM C618-12a) and particle size distribution of the fly ash are shown in Table 1 and Fig. 1, respectively. Alkaline solution consisted of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were used to initiate the polymerization process. The sodium silicate solution was obtained from local company with chemical composition of 27% SiO_3 , 13% Na_2O , and 60% water. The NaOH solution with a concentration of 10 Molar was prepared by adding 400 g of NaOH pellets into distilled water until the total solution quantity becomes 1 L. The solution was left to cool down then it was stored in an airtight plastic container to be used later to produce the geopolymer binder. The silica sand used in this study was locally available with specific gravity, fineness modulus, and water absorption of 2.56, 2.31, 1.87%, respectively. Polycarboxylate ether-based superplasticizer (PC 485, EPSILONE) was used for all mixes. Commercially available monofilament polypropylene (PP) microfibers (from Sika®) were added into the mixes with different quantities. The fibers owned a length of 12 mm, diameter of 34 μm , density of 910 kg/m^3 , and melting point of 160 °C.

2.2. Geopolymer mix design and binder preparation

Four batches of geopolymer binder were prepared in this study: one

control batch with no fibers and three other batches contained PP fibers with dosages of 0.05%, 0.1%, and 0.2% by weight of fly ash. Based on a previous study published by the authors [28], the sodium silicate to sodium hydroxide solution ratio and fluid to binder ratio were 1.5 and 0.6, respectively. The fly ash to sand ratio was selected to be 1:2.75. The mix proportions are detailed in Table 2. The geopolymer binder was prepared by dry mixing the fly ash and PP fibers for 60 s to ensure homogeneous distribution of the fibers. The previously made activator solution and the superplasticizer were mixed together for 120 s, and then the solution was placed in the mixing bowl. The fly ash and PP fibers were added along with the activator solutions to the mixing bowl and mixed for 30 s. Then the sand was added and mixed for another 150 s. The prepared geopolymer mortar was cast in 5 cm cubes for compression test and prisms with dimensions of 4 cm \times 4 cm \times 16 cm for the flexural test. The casted specimens were cured at 80 °C for 24 h. The specimens were named by a letter G refers to geopolymer and two numbers. The first number refers to the fiber content as a weight percentage of the fly ash, and the second number refers to the elevated temperature.

2.3. Heating regime

After curing, number of the prepared geopolymer binder specimens were exposed to elevated temperatures of 200 °C, 400 °C, and 600 °C. Electrical furnace was used with heating profile shown in Fig. 2 with a rate of 3 °C/min. After reaching the desired elevated temperature, the temperature was fixed for two hours then the furnace was turned off. After heating, the specimens were left at room temperature until the day of testing.

2.4. Test procedures

2.4.1. Workability

ASTM C1437 standard was followed to perform the flow table test in order to investigate the effect of the PP microfibers on the workability of geopolymer binders. The flow diameter was measured for geopolymer mixes with 0%, 0.05%, 0.1%, and 0.2% of PP microfibers. Four readings for each mix were measured, and then the average was calculated and reported.

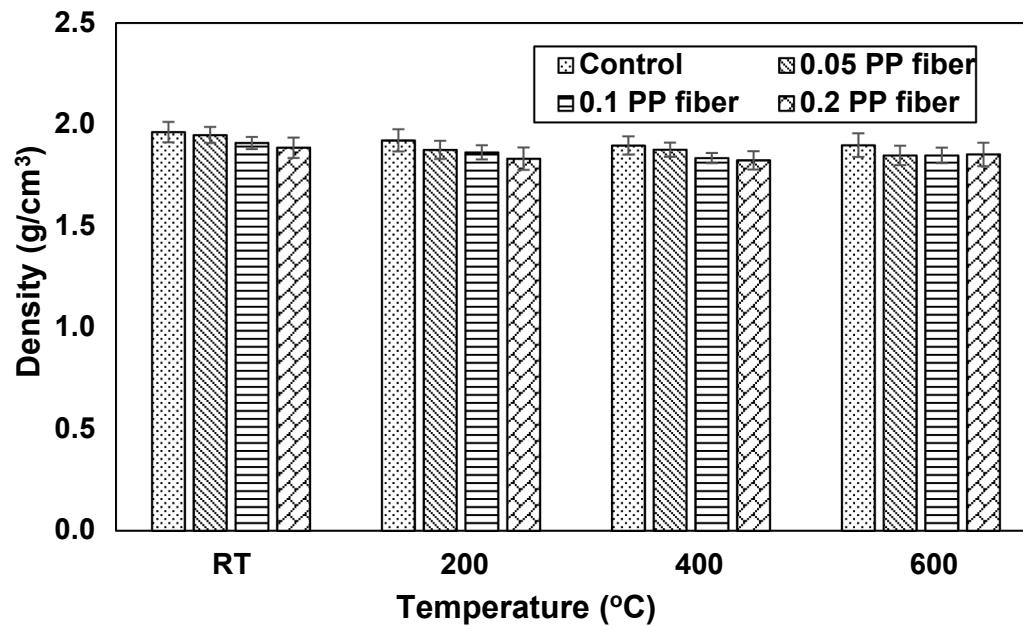


Fig. 4. Density of heated fiber-reinforced geopolymer binders.

Table 3
Mechanical characteristics.

Specimen	Residual compressive strength (MPa)	Residual flexural strength (kN)	Residual Elastic Modulus (GPa)	Residual fracture energy (N. mm)
G-0-25	37.9	2.46	9.87	1991
G-0-200	31.8	1.53	6.25	1168
G-0-400	26.7	0.71	3.24	556.9
G-0-600	12.7	0.45	1.90	334.4
G-0.05-25	39.1	2.89	7.84	5054
G-0.05-200	36.3	1.70	6.14	2660
G-0.05-400	30.6	0.73	4.20	1286
G-0.05-600	11.0	0.41	1.60	315.3
G-0.1-25	38.7	2.65	6.83	4379
G-0.1-200	32.5	1.49	5.56	2346
G-0.1-400	29.4	0.58	3.10	993.3
G-0.1-600	10.4	0.39	1.10	286
G-0.2-25	38.0	2.48	6.56	4221
G-0.2-200	33.7	1.46	5.29	2367
G-0.2-400	27.7	0.55	2.50	717
G-0.2-600	11.6	0.32	0.75	422.2

2.4.2. Density

The effect of the PP fibers on the density of geopolymer binders was investigated according to the ASTM C642 standard. Three 50 mm cube specimens were tested for each batch and the average was reported. The specimens were heated at 110 ± 5 °C for 24 h using electrical oven, then weighted when cooled at room temperature. The procedure was repeated until a constant mass was achieved. The density was measured by dividing the dry weight of each specimen by its volume.

2.4.3. Thermal conductivity

Thermal conductivity of fiber-reinforced geopolymer binders was investigated. Two specimens with size of 40 mm × 40 mm × 80 mm were used to measure the conductivity of the binders using hot disk thermal constant analyzer. The data was acquired during the test using transient plane source (TPS) method.

2.4.4. Mechanical strengths

In order to investigate the mechanical performance of geopolymer binders with PP fibers, compression and flexural strength tests were performed with loading rates of 1.3 kN/s and 0.044 kN/s using universal testing machine according to the ASTM C109 and ASTM C348 standards, respectively. Three samples were tested for each specimen and the average strength was reported.

2.4.5. Thermogravimetric analysis

Thermogravimetric analysis (TGA) test was performed to study the thermal stability of the geopolymer binders. Total mass loss of selected binder specimens due to gradual temperature exposure from 30 °C and 730 °C was monitored using TGA 4000 PerkinElmer device. Small fragments with a size of 45 μm were extracted from the selected specimens and used in the test.

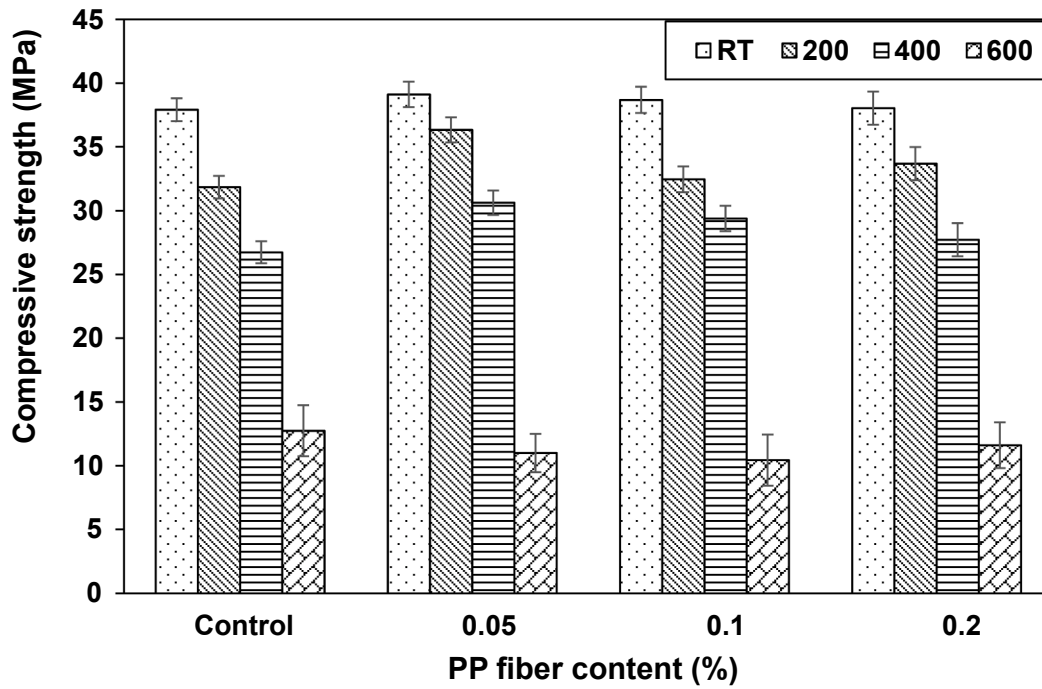
2.4.6. Microstructural investigation

SEM imaging was conducted to investigate the role of the PP fibers in maintaining the integrity of the geopolymer specimens when exposed to elevated temperatures. Small fragments of the binder were extracted from selected specimens and coated with gold to improve the conductivity of the samples. Then, the SEM imaging was conducting in accordance with ASTM C1723, using NOVA NanoSEM 450 device.

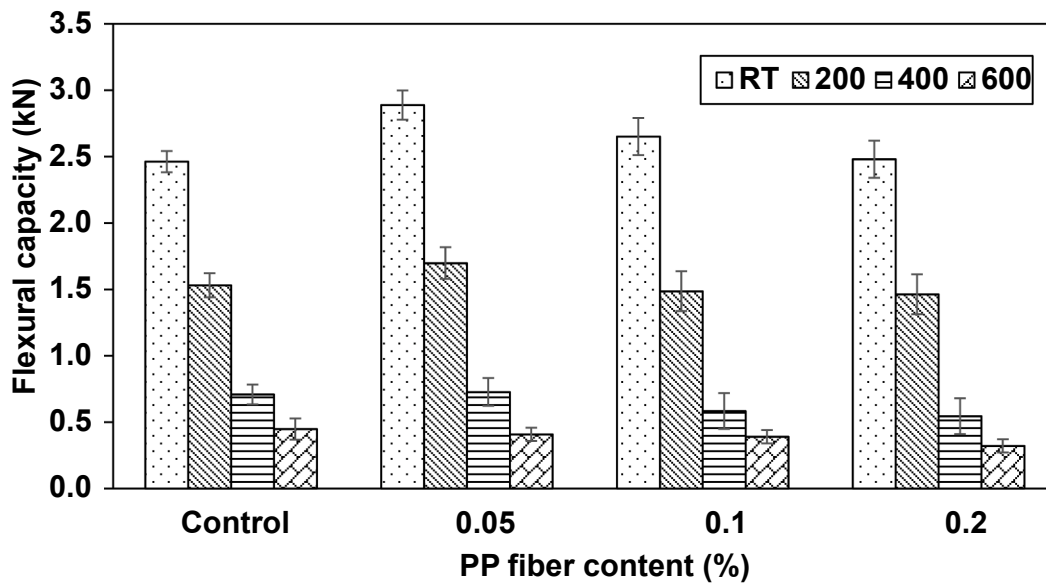
3. Results and discussion

3.1. Workability

Fig. 3 shows the flow table results of geopolymer binders with various fiber contents. The figure revealed that adding PP fibers negatively affected the workability of the mixes. The flowability of the fiber-reinforced geopolymer binder decreased with increasing the fiber content. Similar trend was reported in the literatures [7,9,20]. The



(a)



(b)

Fig. 5. (a) Compressive strength and (b) flexural strength of fiber-reinforced geopolymer binders exposed to elevated temperatures.

reduction in the workability could be attributed to the increase in yield stress of the fresh binder with the increase of fibers' content [7], or to the high shear resistance due to the presence of the fibers [9].

3.2. Density

The variation in the density of fiber-reinforced geopolymer binders when exposed to elevated temperatures was monitored and plotted in

Fig. 4. It was clear that for all exposed temperatures, the density of the binders insignificantly decreased with increasing the fiber content. The reduction in the density might be ascribed to (1) the fact that the density of the fibers was less than that of the geopolymer matrix; (2) the ability of air entrainment by fibers [7,29].

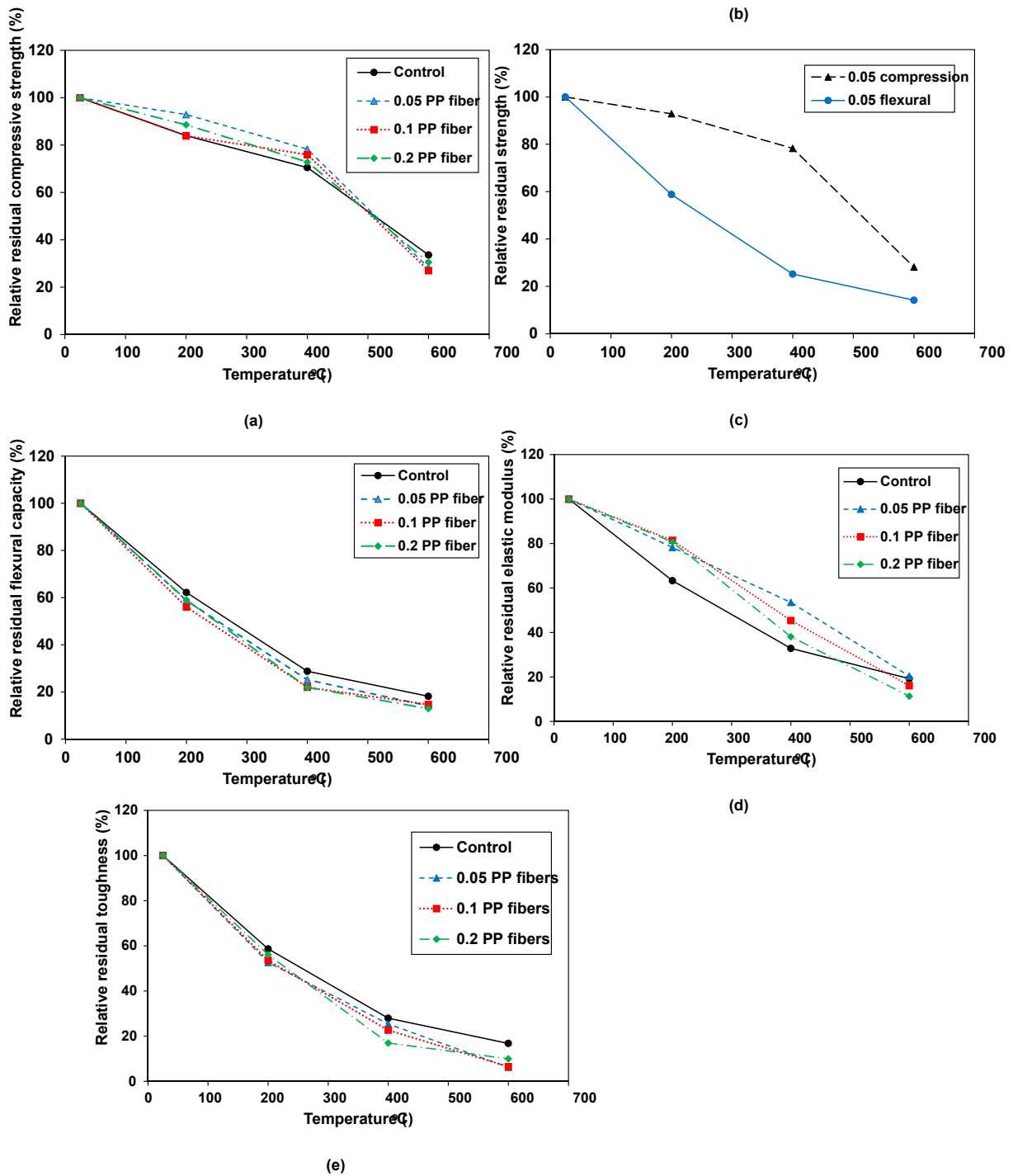


Fig. 6. Relative residual values of fiber-reinforced geopolymer binders exposed to elevated temperatures (a) compressive strength (b) flexural capacity (c) comparison of compression and flexural (d) elastic modulus (e) toughness.

3.3. Mechanical strengths of unheated geopolymer

Compressive and flexural strength tests were conducted for unheated geopolymer binder specimens with various fiber contents. The results are summarized in Table 3. Compressive strength of unheated fibreless geopolymer specimen (G-0-25) equals to 38 MPa. For unheated specimens, adding PP microfibers had neglected effect on the compressive strength of the geopolymer binder regardless of the fiber content as shown in Fig. 5a. Similar observation was reported in the literatures [9,27]. Flexural strength of unheated fibreless geopolymer specimen (G-0-25) equals to 2.5 kN. The flexural strength of unheated specimen was

enhanced by adding PP microfibers as shown in Fig. 5b. The maximum enhancement of 17% was observed for specimen with 0.05% fiber content. Similar observation was reported in the literatures [27,30]. Polypropylene micro fibers were effective in stopping the microcracks caused by tensile stresses in the lower region of the specimens during the flexural strength test. After the formation of the cracks at microscale, the fibers played a major role in preventing these cracks to convert into macroscale, thus kept the integrity of the specimens and enhanced their flexural strength [11,17,27,31].

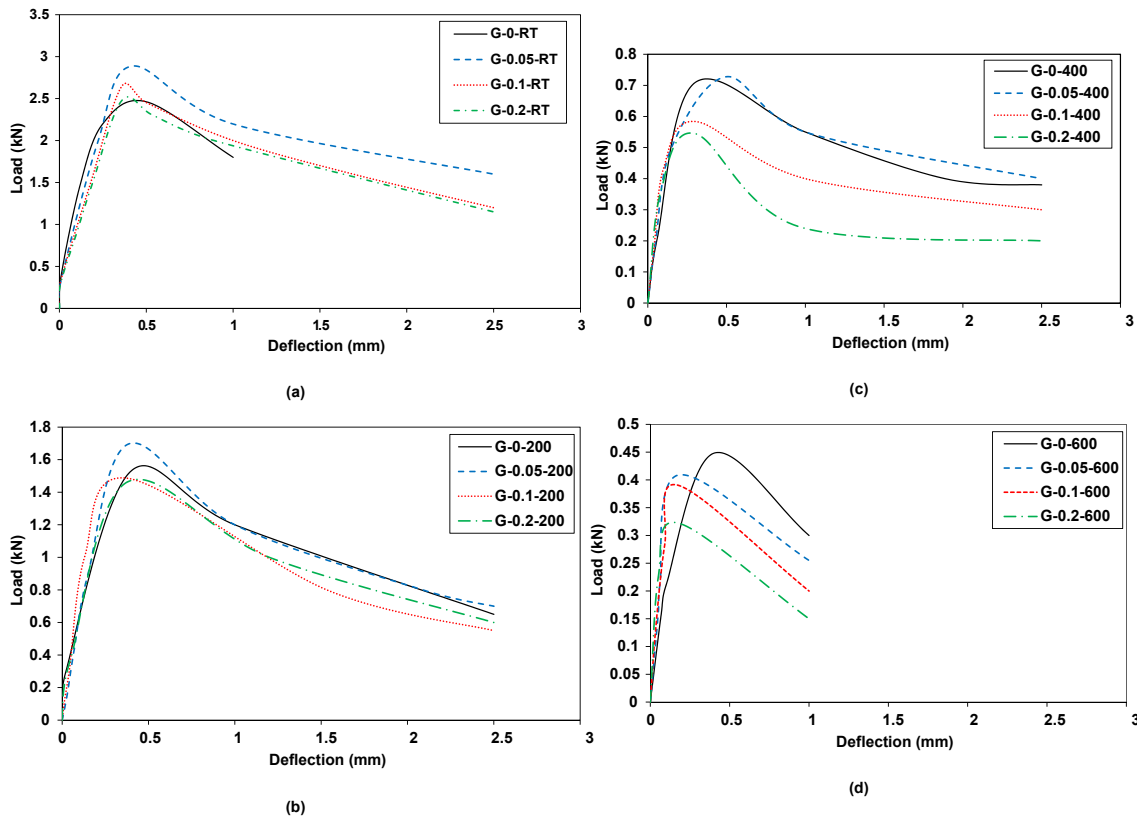


Fig. 7. Flexural load–deflection curves of fiber-reinforced geopolymer heated at (a) room temperature (b) 200 °C (c) 400 °C (d) 600 °C.

3.4. Post-heating behavior of fiber-reinforced geopolymer

3.4.1. Residual compressive strength

Residual compressive strength of heat-damaged fiber-reinforced geopolymer binder specimens are summarized in Table 3. The results revealed that the presence of PP microfibers had significant effect on the strength of heated specimens. The compressive strength of specimens with 0.05% fiber content heated at 200 °C and 400 °C were 14% and 15% higher than the strength of the corresponding fibreless specimens. To focus on the effect of elevated temperature exposure on the compressive strength, relative residual compressive strength was calculated by dividing the residual strength after heating by the original strength of the corresponding unheated specimens. The results were presented in Fig. 6a. The relative residual compressive strengths of fibreless geopolymer binders when exposed to elevated temperatures of 200 °C, 400 °C, and 600 °C were equal to 84%, 70%, and 34% of the original strength of the unheated specimen. The reduction in the strength could be attributed to the thermal shrinkage from the vapor effect [24,32]. During the heating process, the water inside the geopolymer matrix evaporated and caused an internal pressure. The vapor pressure increased with the exposure temperature, and caused thermal stresses thus initiated thermal cracks. For fiber-reinforced specimens, the compressive strength was also decreased due to heating. However, the fiber-reinforced specimens heated up to 400 °C had higher residual strength compared to the corresponding fibreless specimens. After that, the fibreless specimens had higher residual strength when heated at 600 °C as shown in Fig. 6a. Similar trend was reported in [27]. This finding could be attributed to the fact that when exposed to elevated temperature above the melting point of the PP fibers (160 °C–170 °C), the fibers melted and created a path to release the pore pressure thus reduced the thermal stresses [13,15]. It was noted that specimens with 0.05% fiber content had the higher relative residual compressive strength among all other specimens. The reason beyond that could be

attributed to the fact that specimen with 0.05% fiber content had the lowest amount of fibers, thus when melted less voids were created compared to specimens with higher fiber content.

3.4.2. Flexural load–deflection curves

During the flexural test, the load and the midspan deflection values were monitored for all specimens. For unheated geopolymer binder specimens, the values were plotted in Fig. 7a. The unheated fibreless specimen (G-0-25) showed brittle failure without post-failure performance. Adding PP microfibers clearly enhanced the post-failure performance and significantly improved the ductility of the specimens. Similar trend was reported in the literatures [7,8,33]. This finding could be attributed to (1) the bridging ability of the fibers which restricted the initiation and propagation of the cracks within the matrix [29,33] (2) the role of the PP fibers in the formation of many microcracks rather than few macrocracks [7,29]. The load–deflection curves for fiber-reinforced specimens heated at 200 °C, 400 °C, and 600 °C are presented in Fig. 7b–d. The curves were characterized in terms of flexural capacity (maximum load), elastic modulus (slope of initial linear part), and fracture energy or toughness (area under the curve). The effect of elevated temperature on these characteristics are discussed in the following sections.

3.4.3. Residual flexural capacity

Flexural capacities of all tested specimens were extracted from the load–deflection curves as the maximum load, and presented in Table 3. The flexural strength of unheated fibreless geopolymer specimen (G-0-25) equals to 2.5 kN. The flexural strength of unheated geopolymer specimens was enhanced by adding PP microfibers. The maximum enhancement (17%) was observed for specimen with 0.05% fiber content. Similar observation was reported in the literatures [27,30]. Polypropylene micro fibers were effective in stopping the microcracks caused by tensile stresses in the lower region of the specimens during the

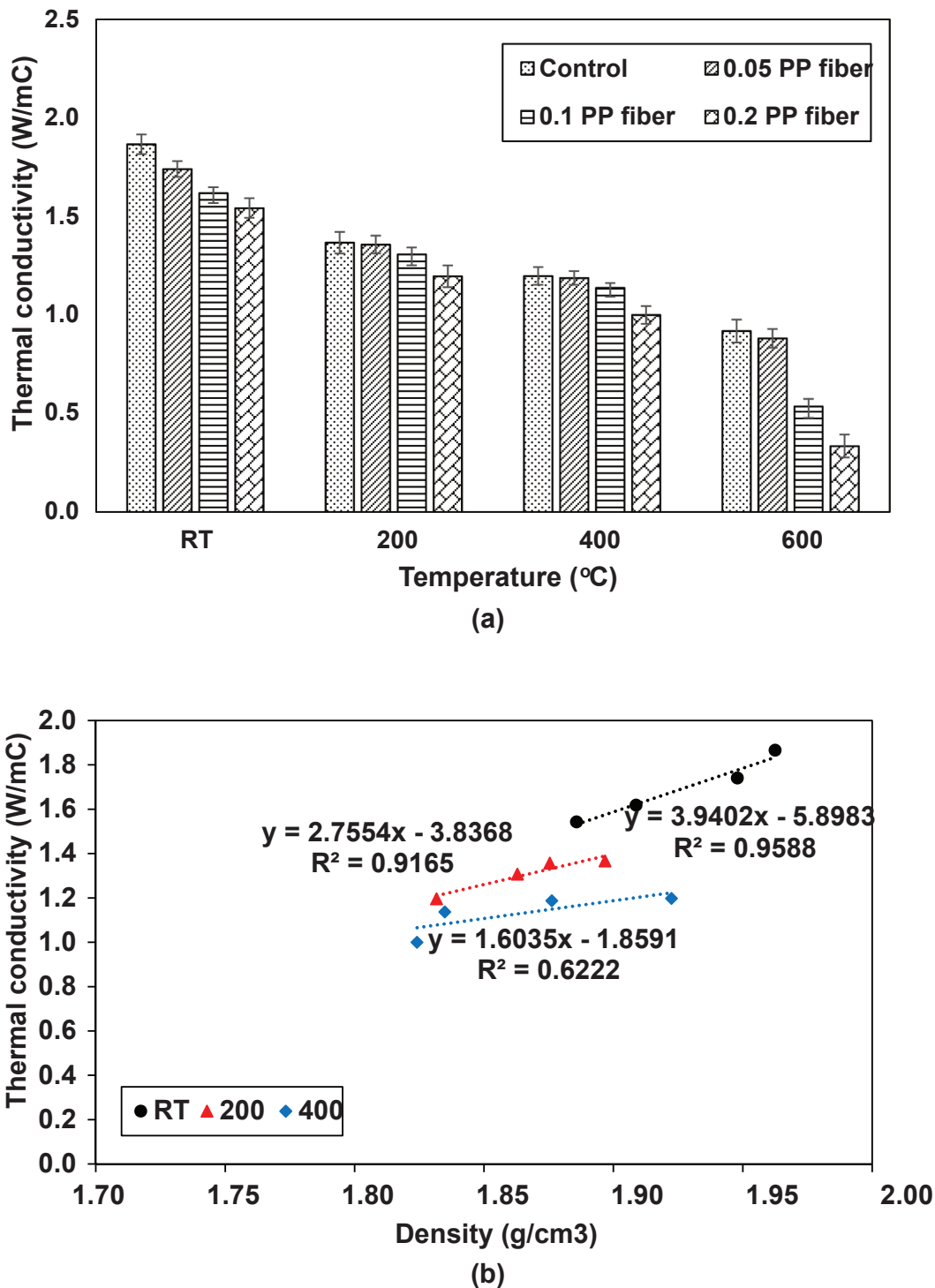


Fig. 8. (a) Thermal conductivity (b) thermal conductivity-density relationship of heated fiber-reinforced geopolymer binders.

flexural strength test. After the formation of cracks at microscale, the fibers played a major role in preventing these cracks to convert into macroscale, thus kept the integrity of the specimens and enhanced their flexural strength [11,17,27,31].

The flexural strength of the fibreless geopolymer binder decreased as the temperature increased. When exposed to elevated temperatures of 200 °C, 400 °C, and 600 °C, the fibreless geopolymer binders' flexural capacities were significantly reduced by 38%, 71%, and 82%, respectively, as shown in Fig. 6b. The reduction in the flexural strength of the geopolymer binder due to heating could be attributed to the thermal

shrinkage from the vapor effect [24,32]. During the heating process, the water inside the geopolymer matrix evaporated and caused an internal pressure. The vapor pressure increased with the exposure temperature, and caused thermal stresses thus initiated intensive thermal cracks.

To focus on the effect of elevated temperatures on the flexural capacity of fiber-reinforced geopolymer binders, the relative residual flexural strength was calculated as the ratio between the residual flexural strength of heated specimens and the flexural strength of the corresponding unheated ones. The results are plotted in Fig. 6b. It was clear that the reduction in the flexural capacity of fiber-reinforced

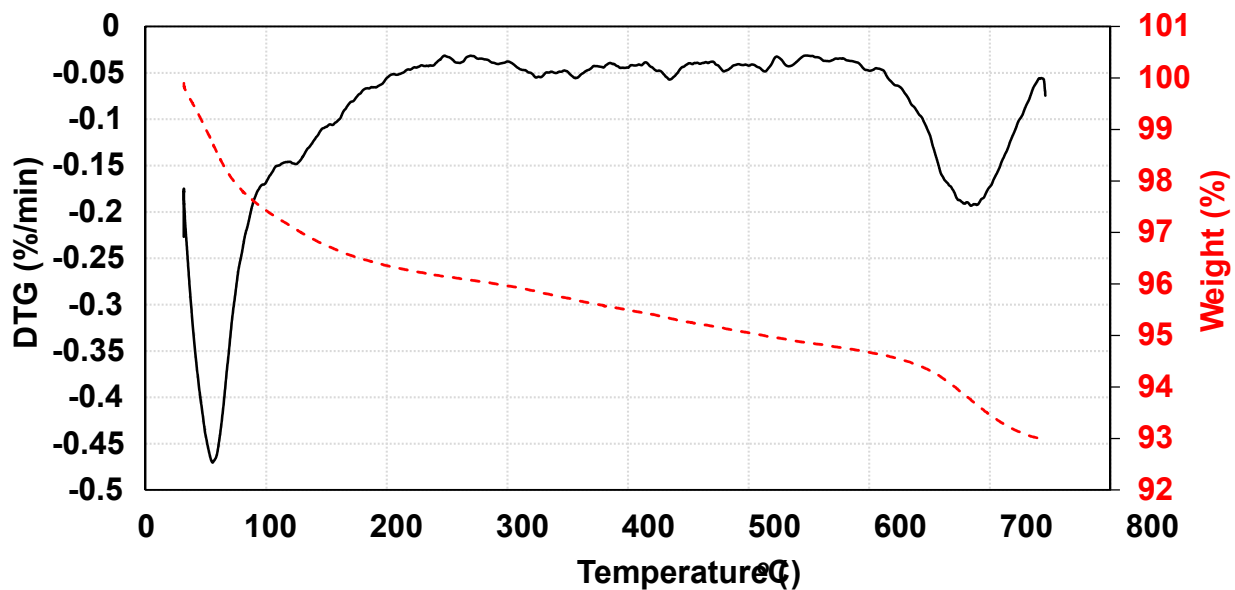


Fig. 9. Thermogravimetric analysis results.

geopolymer was higher than that of fibreless geopolymer for all exposed temperatures. Similar trend was reported in [27]. The relative residual flexural strength of fibreless specimens due to heating at 200 °C, 400 °C, and 600 °C equal to 62%, 29%, and 18%, respectively. These values reduced to be 59%, 25%, and 14% for specimens with 0.05% PP fiber content. In addition, the reduction in the flexural capacity of the fiber-reinforced geopolymer specimens when exposed to elevated temperatures was higher than that for the compressive strength as shown in Fig. 6c. This trend could be attributed to the more sensitivity of the flexural strength to the development and propagation of the internal microcracks at high temperatures [27,34].

3.4.4. Residual elastic modulus

The modulus of elasticity of unheated fibreless specimen was equal to 9.88 GPa. The results revealed that the modulus of elasticity of the geopolymer binder decreased with the inclusion of the fibres. The reduction increased with fiber content. The reduction in the modulus was equal to 21%, 33%, and 34% in the case of specimens with 0.05%, 0.1%, and 0.2% of PP fibers, respectively. Similar trend was reported in the literature [2,7,17,35]. In general, the elastic modulus can be mainly affected by the stiffness of the fibers and porosity of the matrix [7]. The reduction in the modulus of the binders due to the fiber addition could be ascribed to (1) the more pores through the matrix due to the presence of the fibers and (2) the lower value of modulus of elasticity of the PP fibers compared to the geopolymer paste.

On the other hand, the results revealed that the modulus of elasticity of geopolymer binder decreased with heating. However, the presence of the fibers decreased the reduction in the modulus due to heating as shown in Fig. 6d. To highlight that, the relative residual modulus of elasticity was calculated as the ratio between the modulus of elasticity of heated and the corresponding unheated specimens. It was clear in the figure that the fiber-reinforced specimens showed lower rate of modulus of elasticity loss due to heating compared to the fibreless specimens. The relative residual modulus of fiber-reinforced geopolymer was higher than that of fibreless geopolymer for almost all studied temperatures as shown in Fig. 6d. It was also noticed that the drop in the modulus increased with fiber content when the specimens exposed to temperatures between 200 °C and 400 °C which exceed the melting point of the fibers (160 °C).

3.4.5. Residual fracture energy (toughness)

It was clear in Table 3 that the toughness of the unheated geopolymer binder significantly enhanced due to the fiber addition. The enhancement in the toughness could be attributed to the huge improvement in the ductility of the binder in the presence of the fibers. During the test, the PP microfibers mitigated the propagation of the cracks, which allowed the specimens to absorb more energy and increased their toughness. These results are consistent with the literatures [7,22,29].

On the other hand, the toughness of all specimens decreased with increasing the exposure temperature. The reduction in the toughness due to heating was attributed to the reduction in the maximum strength as shown in the load–deflection curves (Fig. 7). The toughness values of fiber-reinforced specimens were higher than that of fibreless specimens for all exposure temperatures as listed in Table 3. The reason of that could be attributed to the enhancement in the post-failure behavior of the geopolymer in the presence of the fibers. The relative residual toughness was calculated as the ratio between the toughness of heated specimens and the corresponding unheated ones. It was noted that the fiber-reinforced specimens showed higher reduction in the toughness due to heating compared to the fibreless specimens as shown in Fig. 6e. This finding might be attributed to the melting of the PP fibers due to heating.

3.5. Thermal behavior characterization

3.5.1. Thermal conductivity

Effect of fiber addition and heating level on the thermal conductivity of geopolymer binders was investigated. Fig. 8a shows that thermal conductivity of geopolymer binder decreased with the presence of the fibers. The reduction increased with fiber content due to the lower value of the thermal conductivity of the PP fibers compared to the geopolymer matrix, or to the ability of air entrainment by fibers. Similar results were reported in [13,15] for cementitious binders. On the other hand, the thermal conductivity of fiber-reinforced geopolymer binder reduced when exposed to elevated temperatures. The reduction increased with temperature as shown in Fig. 8a. This finding could be attributed to the deterioration in the microstructure of the binder due to heating as discussed later in the microstructural analysis section, and to the melting of the fibers due to heating.

The relationship between thermal conductivity and density of fiber-

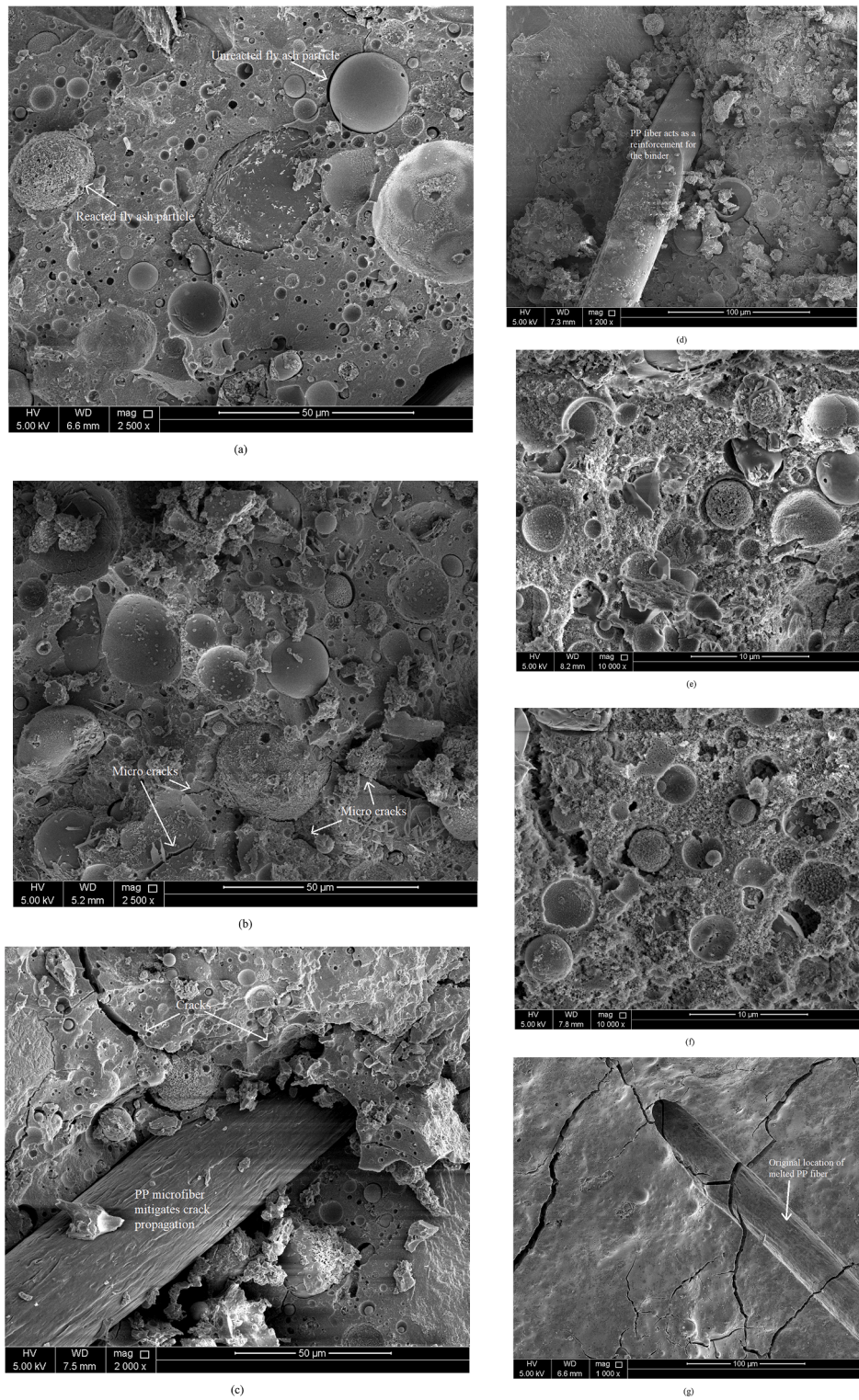


Fig. 10. Representative SEM images show (a) homogeneous geopolymerization matrix with reacted and unreacted fly ash (unheated specimen) (b) thermal microcracks distributed through the matrix (heated at 200 °C) (c) PP microfibers bridging the cracks (heated at 200 °C) (d) PP fibers acted as micro reinforcements (heated at 200 °C) (e) and (f) fragmented matrix combined with more voids and cracks (heated at 400 °C and 600 °C) (g) original location of melted PP fibers (heated at 600 °C).

reinforced geopolymer binders heated at different levels were presented by Eq. (1) and plotted in Fig. 8b.

$$\lambda = \begin{cases} 3.9402\rho - 5.8983RT \\ 2.7554\rho - 3.8368200^\circ C \\ 1.6035\rho - 1.8591400^\circ C \end{cases} \quad (1)$$

Where λ and ρ are the thermal conductivity and the density of geopolymer binder, respectively. It was clear that the thermal conductivity increased linearly with increasing the density of the binder for all exposure temperatures. However, the accuracy of the equations decreased with increasing the exposure temperatures as the values of R^2

decreased.

3.5.2. Thermogravimetric analysis (TGA)

In this study, TGA analysis was carried out for fibreless geopolymers binder under temperature ranging from 20 °C to 750 °C. The goal was to investigate the deterioration in the microstructure of the geopolymer matrix in terms of the total mass loss when subjected to elevated temperatures. The results are presented in Fig. 9 in terms of TGA and its corresponding derivative function (DTG) curves. The TGA profile showed that the total weight loss of the geopolymer binder equal to 7%. According to the literatures [4,24,25,36], the total weight loss of fly ash-based geopolymer binders usually does not exceed 10%. In addition, the figure revealed that three main weight losses occurred during the heating process. The first one occurred between 25 °C and 95 °C. According to the literatures [4,34], this loss referred to the evaporation of free or physically bonded water. The physically bonded water is usually produced by the geopolymerization reaction, and evaporates after exposure to elevated temperatures in the range of 0–120 °C [24]. The second mass loss occurred between 100 °C and 250 °C. This loss was referred to the evaporation of chemically bound water. The chemically bonded water is usually evaporated when the specimens exposed to temperatures in the range of 100–300 °C [4,24,37]. The third mass loss occurred between 600 °C and 730 °C. This loss was referred to the dehydroxylation of the OH groups, which usually occurred when the specimens exposed to temperatures in the range of 600–750 °C [4,23,24].

3.6. Microstructure deterioration

Microstructure deterioration of geopolymer binders due to elevated temperatures exposure was investigated using scanning electron microscopy (SEM) analysis. The role of the PP microfibers in mitigating the microstructure deterioration was also explored. For fibreless unheated specimen, the SEM image captured homogeneous geopolymerization matrix produced by aluminosilicate gel with reacted and unreacted FA particles as shown in Fig. 10a. Small microcracks were noticed in the dense matrix, which resulted from water evaporation during the preparation process [24]. When exposed to elevated temperatures, deterioration to the binder microstructure combined with thermal microcracks have been noticed. The microcracks started to initiate and propagate through the microstructure as shown in Fig. 10b. However, the PP microfibers bridged the cracks and mitigated their propagation as shown in Fig. 10c. In addition, the fibers distributed within the matrix as micro reinforcements as shown in Fig. 10d. Good bond between the fibers and the binder matrix was noticed in Fig. 10d, which enhanced the stress transfer process thus improved the mechanical properties. For fibreless specimens heated at 400 °C, more cracks were monitored within fragmented matrix as shown in Fig. 10e. Exceeding the exposed temperature to be 600 °C resulted in wider cracks distributed within crumbled matrix as shown in Fig. 10f. For fiber-reinforced binder heated at a temperature greater than the melting point of the PP fibers, the fibers melted which resulted in more voids as shown in Fig. 10g, which referred to the original location of the melted fibers. Generating these extra voids might be one reason beyond the reduction of the mechanical strengths of the binder.

4. Conclusions

Thermal and post-heating behavior of fiber-reinforced geopolymer binders were experimentally investigated in this research. The following conclusions could be drawn:

1. The workability of the fly ash-based geopolymer binders declined with increasing the PP fiber content. Maximum reduction of 19% was reported for 0.2% PP fibers.
2. Using PP microfibers had neglected effect on the compressive strength of the unheated fly ash-based geopolymer binders, but clearly enhanced their flexural strength. Maximum enhancement of 17% was observed in the flexural strength of unheated binders with 0.05% PP fibers.
3. Heating fly ash-based geopolymer binders caused degradation in their mechanical strengths. The reduction in compressive and flexural strengths of the binders reached 63% and 82%, respectively, when heated up to 600 °C. Using PP microfibers enhanced the residual compressive but not the flexural strength of the heat-damaged binders.
4. PP microfibers reduced the modulus of elasticity of the unheated geopolymer binders, but significantly improved the ductility and the toughness of the specimens. Specimen with 0.05% fiber content owned the best performance among all other percentages.
5. The modulus of elasticity of fly ash-based geopolymer binders decreased with heating. Fiber-reinforced specimens showed lower rate of modulus of elasticity loss due to heating compared to the fibreless specimens.
6. The toughness of fly ash-based geopolymer binders decreased with increasing the exposure temperature. Fiber-reinforced specimens showed higher reduction in the toughness due to heating compared to the fibreless specimens.
7. Using PP microfibers reduced the thermal conductivity of geopolymer binders. The reduction increased with increasing the fiber content. For mixes with up to 0.1% PP microfibers, the reduction due to fiber addition was less pronounced for heated-specimens compared to unheated ones.
8. Good bond between PP microfibers and the geopolymer matrix was noticed in the SEM images, which enhanced the stress transfer process thus improved the mechanical properties.
9. Deterioration to the geopolymer binder microstructure combined with thermal microcracks have been noticed in the SEM images for heated specimens. The PP microfibers mitigated the propagation of the cracks.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

Thermal analysis was performed at the Center for Advanced Materials (CAM) and the SEM imaging was conducting at the Central Labs Unit at Qatar University.

References

- [1] F. Colangelo, I. Farina, M. Travaglini, C. Salzano, R. Cioffi, A. Petrillo, Eco-efficient industrial waste recycling for the manufacturing of fibre reinforced innovative geopolymer mortars: Integrated waste management and green product development through LCA, *J. Clean. Prod.* 312 (2021) 127777.
- [2] S.K. John, Y. Nadir, K. Girija, Effect of source materials, additives on the mechanical properties and durability of fly ash and fly ash-slag geopolymer mortar: A review, *Constr. Build. Mater.* 280 (2021) 122443.
- [3] A. Hassan, M. Arif, M. Shariq, A review of properties and behaviour of reinforced geopolymer concrete structural elements- A clean technology option for sustainable development, *J. Clean. Prod.* 245 (2020) 118762.
- [4] M.R. Irshidat, N. Al-Nuaimi, M. Rabie, Sustainable utilization of waste carbon black in alkali-activated mortar production, *Case Stud. Constr. Mater.* 15 (2021) e00743.
- [5] P. Zhang, Y. Zheng, K. Wang, J. Zhang, A review on properties of fresh and hardened geopolymer mortar, *Compos. Part B Eng.* 152 (2018) 79–95.
- [6] D.S.d.T. Pereira, F.J.d. Silva, A.B.R. Porto, V.S. Candido, A.C.R.d. Silva, F.D. C. Garcia Filho, S.N. Monteiro, Comparative analysis between properties and microstructures of geopolymeric concrete and portland concrete, *J. Mater. Res. Technol.* 7 (4) (2018) 606–611.
- [7] N. Ranjbar, M. Zhang, Fiber-reinforced geopolymer composites: A review, *Cem. Concr. Compos.* 107 (2020) 103498.

- [8] A. Noushini, M. Hastings, A. Castel, F. Aslani, Mechanical and flexural performance of synthetic fibre reinforced geopolymer concrete, *Constr. Build. Mater.* 186 (2018) 454–475.
- [9] N. Ranjbar, S. Talebian, M. Mehrali, C. Kuenzel, H.S. Cornelis Metselaar, M. Z. Jumaat, Mechanisms of interfacial bond in steel and polypropylene fiber reinforced geopolymer composites, *Compos. Sci. Technol.* 122 (2016) 73–81.
- [10] A.C. Ganesh, M. Muthukannan, Development of high performance sustainable optimized fiber reinforced geopolymer concrete and prediction of compressive strength, *J. Clean. Prod.* 282 (2021) 124543.
- [11] K. Chen, D. Wu, HaiXiang Chen, G. Zhang, R. Yao, C. Pan, Z. Zhang, Development of low-calcium fly ash-based geopolymer mortar using nanosilica and hybrid fibers, *Ceram. Int.* 47 (15) (2021) 21791–21806.
- [12] F. Şahin, M. Uysal, O. Canpolat, Y. Aygörmec, T. Coşgun, H. Dehghanpour, Effect of basalt fiber on metakaolin-based geopolymer mortars containing rilem, basalt and recycled waste concrete aggregates, *Constr. Build. Mater.* 301 (2021) 124113.
- [13] M.R. Irshidat, N. Al-Nuaimi, M. Rabie, Hybrid effect of carbon nanotubes and polypropylene microfibers on fire resistance, thermal characteristics and microstructure of cementitious composites, *Constr. Build. Mater.* 266 (2021) 121154.
- [14] M.R. Irshidat, N. Al-Nuaimi, M. Rabie, Microstructure and mechanical behavior of cementitious composites with multi-scale additives, *Adv. Concr. Constr.* 11 (2021) 163–171.
- [15] M.R. Irshidat, N. Al-Nuaimi, M. Rabie, The Role of Polypropylene Microfibers in Thermal Properties and Post-Heating Behavior of Cementitious Composites, *Materials* 13 (2020) 2676.
- [16] E. Mohseni, Assessment of Na₂SiO₃ to NaOH ratio impact on the performance of polypropylene fiber-reinforced geopolymer composites, *Constr. Build. Mater.* 186 (2018) 904–911.
- [17] A. Karimipour, M. Ghalehnavi, J. de Brito, Effect of micro polypropylene fibres and nano TiO₂ on the fresh- and hardened-state properties of geopolymer concrete, *Constr. Build. Mater.* 300 (2021) 124239.
- [18] H. Nguyen, V. Carvelli, E. Adesanya, P. Kinnunen, M. Illikainen, High performance cementitious composite from alkali-activated ladle slag reinforced with polypropylene fibers, *Cem. Concr. Compos.* 90 (2018) 150–160.
- [19] F. Puertas, T. Amat, A. Fernández-Jiménez, T. Vázquez, Mechanical and durable behaviour of alkaline cement mortars reinforced with polypropylene fibres, *Cem. Concr. Res.* 33 (12) (2003) 2031–2036.
- [20] A. Bhutta, M. Farooq, N. Banthia, Performance characteristics of micro fiber-reinforced geopolymer mortars for repair, *Constr. Build. Mater.* 215 (2019) 605–612.
- [21] Y. Wang, T. Zheng, X. Zheng, Y. Liu, J. Darkwa, G. Zhou, Thermo-mechanical and moisture absorption properties of fly ash-based lightweight geopolymer concrete reinforced by polypropylene fibers, *Constr. Build. Mater.* 251 (2020) 118960.
- [22] P. Sukontasukkul, P. Pongsopha, P. Chindaprasirt, S. Songpiriyakij, Flexural performance and toughness of hybrid steel and polypropylene fibre reinforced geopolymer, *Constr. Build. Mater.* 161 (2018) 37–44.
- [23] P. Chindaprasirt, U. Rattanasak, S. Taebuanhuad, Resistance to acid and sulfate solutions of microwave-assisted high calcium fly ash geopolymer, *Mater. Struct.* 46 (3) (2013) 375–381.
- [24] X. Jiang, Y. Zhang, R. Xiao, P. Polaczyk, M. Zhang, W. Hu, Y. Bai, B. Huang, A comparative study on geopolymers synthesized by different classes of fly ash after exposure to elevated temperatures, *J. Clean. Prod.* 270 (2020) 122500.
- [25] F. Colangelo, R. Cioffi, G. Roviello, I. Capasso, D. Caputo, P. Aprea, B. Liguori, C. Ferone, Thermal cycling stability of fly ash based geopolymer mortars, *Compos. Part B Eng.* 129 (2017) 11–17.
- [26] S. Çelikten, M. Sarıdemir, İ. Özgür Deneme, Mechanical and microstructural properties of alkali-activated slag and slag + fly ash mortars exposed to high temperature, *Constr. Build. Mater.* 217 (2019) 50–61.
- [27] Y. Aygörmec, O. Canpolat, M.M. Al-mashhadani, M. Uysal, Elevated temperature, freezing-thawing and wetting-drying effects on polypropylene fiber reinforced metakaolin based geopolymer composites, *Constr. Build. Mater.* 235 (2020) 117502.
- [28] M.R. Irshidat, N. Al-Nuaimi, W. Ahmed, M. Rabie, Feasibility of recycling waste carbon black in cement mortar production: Environmental life cycle assessment and performance evaluation, *Constr. Build. Mater.* 296 (2021) 123740.
- [29] K.Z. Farhan, M.A.M. Johari, R. Demirboğa, Impact of fiber reinforcements on properties of geopolymer composites: A review, *J. Build. Eng.* 44 (2021) 102628.
- [30] M.M. Al-mashhadani, O. Canpolat, Y. Aygörmec, M. Uysal, S. Erdem, Mechanical and microstructural characterization of fiber reinforced fly ash based geopolymer composites, *Constr. Build. Mater.* 167 (2018) 505–513.
- [31] Y. Xu, G. Xing, J. Zhao, Y. Zhang, The effect of polypropylene fiber with different length and dosage on the performance of alkali-activated slag mortar, *Constr. Build. Mater.* 307 (2021) 124978.
- [32] O.A. Abdulkareem, A.M. Mustafa Al Bakri, H. Kamarudin, I. Khairul Nizar, A. A. Saif, Effects of elevated temperatures on the thermal behavior and mechanical performance of fly ash geopolymer paste, mortar and lightweight concrete, *Constr. Build. Mater.* 50 (2014) 377–387.
- [33] A. Karimipour, J. de Brito, Influence of polypropylene fibres and silica fume on the mechanical and fracture properties of ultra-high-performance geopolymer concrete, *Constr. Build. Mater.* 283 (2021) 122753.
- [34] H.Y. Zhang, V. Kodur, B. Wu, L. Cao, S.L. Qi, Comparative Thermal and Mechanical Performance of Geopolymers derived from Metakaolin and Fly Ash, *J. Mater. Civ. Eng.* 28 (2016) 04015092.
- [35] A.M. Rashad, A comprehensive overview about the influence of different additives on the properties of alkali-activated slag – A guide for Civil Engineer, *Constr. Build. Mater.* 47 (2013) 29–55.
- [36] M. Sivasakthi, R. Jeyalakshmi, N.P. Rajamane, Fly ash geopolymer mortar: Impact of the substitution of river sand by copper slag as a fine aggregate on its thermal resistance properties, *J. Clean. Prod.* 279 (2021) 123766.
- [37] H.Y. Zhang, V. Kodur, B. Wu, L. Cao, F. Wang, Thermal behavior and mechanical properties of geopolymer mortar after exposure to elevated temperatures, *Constr. Build. Mater.* 109 (2016) 17–24.