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INFLUENCE OF HIGH TEMPERATURES ON PROPERTIES OF GEOPOLYMERS FILLED BY INORGANIC FIBROUS PARTICLES

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SUMMARY OF THE THESIS

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ABSTRACT

The presented research work deals with elevated temperature properties of inorganic fibrous particles filled geopolymers when exposed to 200, 400 and 800 ^{o}C. The basalt fibrous wastes and carbon fibrous particles (Carbiso) were chosen as source of inorganic fibrous particles due to their less cost and better thermal resistance properties. The high energy ball milling process was employed to prepare basalt microfibrils (BMF) and carbon microfibers (CMF) after 30 min dry pulverization of basalt fibrous wastes and carbiso powder, respectively. The prolonged pulverization was not continued because of rise in temperature of ball mill and sticking of particles to the surface of milling containers. Nevertheless, the longer grinding of carbiso powder showed less sticking tendency as compared to basalt fibrous wastes. Later, the milled particles were incorporated under 5, 10 and 15 wt % loading into geopolymers synthesized from calcined kaolin and shale clay residues. The prepared BMF/geopolymer composites or CMF/geopolymer composites were then evaluated for physical properties, micro-structural analysis and compression strength before and after exposure to elevated temperatures. As compared to BMF, the addition of CMF was found to maintain compact structure of geopolymers at elevated temperature exposures. This behavior was attributed to effective pore filling ability and better thermo-chemical resistance of CMF as compared to BMF. The geopolymer composite of 10 wt % BMF depicted the maximum compressive strengths of 34 MPa, 42 MPa, 23 MPa and 16 MPa at 30 ^{o}C, 200 ^{o}C, 400 ^{o}C and 800 ^{o}C, respectively. On the other hand, the maximum compressive strengths of 44 MPa, 49 MPa, 30 MPa and 21 MPa was recorded for the geopolymer composite of 10 wt % CMF at 30 ^{o}C, 200 ^{o}C, 400 ^{o}C and 800 ^{o}C, respectively. This indicated greater decrease in thermal stresses as well as more restriction on swelling of unreacted precursor phases after addition of CMF than BMF. Furthermore, the geopolymers filled by BMF and CMF showed higher compression strength values than the previously reported results on neat OPC when exposed to 800 ^{o}C. The 5, 10 and 15 wt% BMF filled geopolymers showed 22 %, 42 %, and 34 % increase over OPC respectively, whereas 5, 10 and 15 wt% CMF filled geopolymers showed 76 %, 88 % and 112 % increase over OPC respectively.

Keywords: Filled geopolymers, Basalt microfibrils, Carbon microfibers, Geopolymer composites, Compressive strength, Pore-filling ability, Thermal resistance

ABSTRAKT

Předložená práce se zabývá chováním geopolymerů plněných anorganickými vlákennými částicemi při zvýšené teplotě 200, 400 a 800 ºC. Pro výběr anorganických plniv byl zohledněn požadavek zvýšené tepelné odolnosti při přijatelné ceně a možnosti mechanického zjemňování. Byly vybrány částice na bázi čedičového vláknitého odpadu a uhlíkové vlákenné částice (Carbiso). Pro přípravu čedičových mikrofibril (BMF) a uhlíkových mikrovláken (CMF) bylo použito vysoce energetické mletí za sucha v kulovém mlýnku. Doba mletí 30 min. byla specifikována s ohledem na omezení vzrůstu teploty mlýnku a zabránění lepivosti mletých částic na jeho vnitřní povrch. Byly syntetizovány geopolymery z kalcinovaného kaolinu a břidlicového jílu s obsahem mletých částic BMF a CMF v rozmezí 5, 10 a 15% hmotnostních procent. Takto připravené kompozitní materiály geopolymer/CMF a geopolymer/BMF byly charakterizovány pomocí fyzikálních vlastností, mikrostrukturní analýzy a pevnosti v tlaku před a po vystavení zvýšeným teplotám. Bylo zjištěno, že přidání CMF udržuje lépe kompaktní strukturu kompozitních materiálů při zvýšených teplotních expozicích než přidání BMF. Tento rozdíl v chování obou plniv souvisí s jejich schopností efektivně plnit póry a termo chemickou degradací BFM za vysokých teplot. Kompozit s obsahem 10 hmotnostních procent BMF docílil pevnost tlaku 34 MPa, 42 MPa, 23 MPa and 16 MPa při teplotách 30 ºC, 200 ºC, 400 ºC and 800 ºC. Kompozit s obsahem 10 hmotnostních procent CMF docílil pevnost v tlaku 44 MPa, 49 MPa, 30 MPa a 21 MPa při teplotách 30 ºC, 200 ºC, 400 ºC a 800 ºC. Je patrné, že přídavek anorganických vláknitých částic přispívá ke snížení tepelného napětí a omezuje bobtnání nezreagované fáze prekurzoru. Geopolymrey plněné oběma typy částic vykazovaly zvýšené hodnoty pevnosti v tlaku v porovnání s geopolymery bez obsahu částicových plniv při teplotě 800 ºC. Geopolymery s obsahem 5, 10 a 15 hmotnostních procent BMF vykazovaly 22 %, 42 %, a 34 % nárůst pevnosti v tlaku ve srovnání s geopolymery bez obsahu částicových plniv. Geopolymery s obsahem 5, 10 a 15 hmotnostních procent CMF vykazovaly 76 %, 88 % and 112 % nárůst pevnosti v tlaku ve srovnání s geopolymery bez obsahu částicových plniv.

Klíčová slova: plněné geopolymery, čedičová mikrovlákna, uhlíková mikrovlákna, geopolymerní kompozity, pevnost v tlaku, schopnost plnění pórů, tepelná odolnost

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1 Introduction

There has been an increased environmental concern related to manufacture of ordinary Portland cement (OPC) as it results in significant release of CO_2 rapid depletion of landscape, dust production during transport, and generation of noise, etc [1]–[4]. Moreover, OPC has shown inferior performance in sulphate or acid environment due to easy dissolution of calcium compounds [5]. As a result, the research over alternative OPC binders gained importance to achieve environmental sustainability and durability in construction and building industry[1], [6]–[8]. Geopolymer is considered as the third generation cement after lime and OPC, and it is now emerged as an alternative to OPC due to superior durability and environmental performance [9].

2 Purpose and the aim of the thesis

Many researchers studied the mechanical properties of glass fiber reinforced geopolymer mortar at high temperatures, whereas only few studies were reported on the basalt fibers [10], [11]. The basalt fibers are easy to process, non-toxic, natural, eco-friendly and inexpensive as compared to other inorganic fibers. They are prepared from volcanic rocks produced from frozen lava, with a melting temperature comprised between 1500 and 1700 ^{o}C. They have extremely good modulus, high strength, improved strain to failure, high temperature resistance, excellent stability, good chemical resistance and reduced thermal and electrical conductivity [12], [13]. Various researches reported on continuous basalt fabric or basalt fiber as a strengthening material for cementitious concrete structures, though there are confined studies on the consequence of short basalt fibers on the properties of geopolymers. In recent times, carbon materials are treated as a potential candidate for reinforcement of geopolymers while exposed to higher temperature because of their remarkable thermal, mechanical and electrical properties [14], [15]. For this purpose, graphene, carbon nanofibers, carbon nanotubes etc were examined for enhancing the strength and ductility of geopolymer composites [16]. Furthermore, only some researchers also suggested the use of economical micro-size carbonized coconut shell, hemp hurds and bamboo particles particles over carbon nanotubes owing to their easier handling [17]. However, no research work is reported on the elevated temperature properties of carbiso particles filled geopolymers. The carbiso are 100 μm milled inexpensive carbon particles obtained from recycled carbon fibrous wastes. The thesis systematically investigated the effects of incorporation of inexpensive inorganic microfibers (basalt microfibrils and carbon microfibers) on the structure and thermal evolution of geopolymers synthesized from metakaoline. In particular, the following objectives were studied in detail

a) Effect of ball milling time on particle size distribution of BMF and CMF
b) Characterization of microstructure of geopolymer composites by SEM, EDS, Image analysis, XRD, TGA
c) Characterization of mechanical properties of geopolymer composites by measurement of compression strength, hardness, density, etc.
d) Evaluation of elevated temperature properties of geopolymer composites
e) Study of pore-filling ability of basalt microfibrils and carbon microfibers in geopolymer composites when exposed to elevated temperatures
f) Comparison of elevated temperature performance of geopolymer composites over previously reported traditional OPC based construction materials

3 Overview of the current state of the problem

Geopolymers-based materials are very attractive in construction industry as green concrete due to their corrosion resistance, cost efficiency, low permeability, low density, low shrinkage, rapid strength gain rate, chemical stability and freeze-thaw resistance, etc [18]. However, they have certain limitations over OPC. Geopolymers tend to be brittle, vulnerable to crack formation and undergo catastrophic failure because of

their cross-linked structure [19]. The inclusion of different fibers have shown to be effective in controlling crack propagation and enhancing the fracture energy of geopolymers, but the mechanical properties of geopolymers were found inadequate and non-consistent while exposed to elevated temperatures [20], [21]. During fire accidents, various fibers fail in providing effective reinforcements owing to lack of durability and structural strength at higher temperature. Furthermore, the thermal expansion mismatch between fiber and matrix can introduce thermal fatigue and stresses, and therefore affect the lifetime and dimensional stability of the composites [22], [23]. Moreover, the destruction of geopolymers can occur during the fire exposure due to evaporation of water adsorbed by sodium aluminosilicate N-A-S-H) gel, formation of anhydrous products, crystallization of stable anhydrous phases and melting [24] (see Figure 1). Hence, more research is necessary to identify alternative fibers which have better thermal resistance and sustain higher residual mechanical properties while exposed to elevated temperature [25].

$$\underset{\text{Geopolymer}}{M_n[-(SiO_2)_z - AlO]_n . wH_2O} \xrightarrow{\text{Dehydration } 125-160\ ^\circ C} \underset{\text{Anhydrous product}}{M_n[-(SiO_2)_z - AlO]_n}$$

$$\underset{\text{Anhydrous product}}{M_n[-(SiO_2)_z - AlO]_n} \xrightarrow{\text{Crystallization } 600-800\ ^\circ C} \underset{\text{(Nepheline)}}{Na_2O.Al_2O_2.2SiO_2} \xrightarrow[\text{Recrystallization}]{\text{Above } 800\ ^\circ C} \underset{\text{(Albite)}}{Na_2O.Al_2O_3.2SiO_2}$$

Figure 1. Geopolymer phase transformation during fire [24].

4 Method Used, study material

4.1 Materials

The recycled carbon materials under trade name carbiso mil 100 μ were purchased from Easy composites, UK, whereas the short basalt fibrous waste was obtained from the VEBA Industries, Czech Republic. The basalt fibers had density of 2650 kg/m^3, initial modulus of 95 GPa, tensile strength of 4 GPa, elongation at break of 3 % and water absorption of less than 0.5 %. The chemical composition of basalt fibers as measured from elemental analysis is shown in Table 1.

Table 1.Elemental analysis of basalt fibers

Element	Oxygen	Sodium	Magnesium	Aluminum	Silicon	Potassium	Calcium	Iron
Weight %	42.41	0.56	1.04	5.39	14.79	0.97	5.72	10.27

The Baucis L110 alumino-silicate geopolymer binder based on metakaolin was obtained from Ceske Lupkove Zavody, Czech Republic along with sodium alkali activator. The metakaolin geopolymer was synthesized from calcined kaolin and shale clay residues with Si/Al ratio of 2.0. The kaolin was mainly composed of kaolinite with small amounts of quartz, whereas shale clay was composed of kaolinite with low amount of quartz and anatase. At first, kaolin and shale clay were passed in rotary klin to result in 30-70% loss of kaolinitic structure due to dehydroxylation. Later, it was converted to metakaolin by additional calcinations at 750 ^{o}C for 10 h in bath oven. The chemical composition of the metakaolin geopolymer was as follows (wt.%): SiO_2 47, Al_2O_3 24, LOI 0.5, Fe_2O_3 0.50, TiO_2 0.8, MgO 3.5, K_2O 0.40, CaO 17.50. The

mean particle size (d50) was 5 μm. The sodium alkali activator was mixture of Na_2SiO_3 and NaOH.

4.2 Preparation of carbon and basalt micro fibers

The short basalt fibrous waste was dipped in acetone for 24 h to remove the surface finish and impurities. For preparation of carbon and basalt microfibers, 30 min dry grinding was carried out by high-energy planetary ball mill of Fritsch Pulverisette 7, Germany in a sintered corundum container of 80 ml capacity using zirconium balls of 10 mm diameter [26], [27]. The ball to material ratio was kept at 10:1 and the speed was kept at 850 rpm. Later, Malvern zetasizer nano series based on dynamic light scattering principle of Brownian motion of particles was employed to characterize the particle size distribution of dry milled carbon/basalt particles. Deionized water was used as dispersion medium and it was ultrasonicated for 5 min with bandelin ultrasonic probe before characterization. In addition, microstructure of milled particles was observed on scanning electron microscope (SEM) of Hitachi–model TM-3000 at accelerated voltage of 15 kV.

4.3 Preparation of geopolymer composites

The four parts of sodium alkali activator and five parts of metakaoline based geopolymer were manually mixed for 10 min to ensure homogeneous preparation of geopolymer binders. For preparation of geopolymer composites, the carbon and basalt microfibers were initially pre-dried for 60 min at 70 ^{o}C in an oven. Next, both carbon/basalt micro fibers were added into the prepared geopolymer binder separately at 5 wt %, 10 wt % and 15 wt % loading. The mixing was homogeneously done in Hobart mixer for 5 min. Subsequently, the fresh prepared composite mortar was poured into 40 mm cubic-shaped moulds, vibrated for 2 minutes on the vibration table to remove air voids and wrapped using a thin plastic sheet to prevent water evaporation. The wrapped samples were demolded after 24 h of casting and then cured at room temperature (20 ± 2 ^{o}C) and a relative humidity of (70 ± 10 %) for 28 days.

4.4 Exposure to elevated temperature

The prepared geopolymer composites were exposed to elevated temperatures of 200, 400 and 800 ^{o}C at age of 28 days. The specimens were placed into a furnace (Elektrické Pece Svoboda, Czech Republic) and heated at fixed heating rate of 5 ^{o}C/min. As soon as the target temperature was attained, it was maintained for an additional 60 min. The furnace was then shut down to allow the specimens in the furnace to cool down to room temperature. Meanwhile, the unexposed specimens were left undisturbed at ambient condition.

4.5 Microstructure of geopolymer composites

The low vacuum scanning electron microscopy (SEM) of Hitachi–model TM-3000, coupled with X-rays microanalysis system of energy dispersive spectroscopy was employed to investigate the microstructure of geopolymer composites. It was carried out at 15 kV accelerated voltage. The samples were directly observed under the SEM without metallic coating due to low vacuum operations. The images were formed by acquisition of backscattered electrons at different magnifications.

4.6 Image analysis of geopolymer composites

It was employed to perform the pore area analysis on SEM images using IMAGEJ software. At first, the quality of images was improved by contrast enhancement and noise removal. Then, the images were segmented by proper thresholding method. In the current study, Otsu thresholding was suitably used to transform the images into binary form. The benefit of acquiring binary image is that it diminishes the difficulty of the data and simplifies the process of recognition and classification of porous and non porous area. Accordingly, the pore area (%) was evaluated by IMAGEJ software. Pore area (%) comprises the measurement of individual pore, summing up of all the individual pores and dividing the sum by the total

area of the image [28].

4.7 Phase composition of geopolymer composites

The X-ray diffraction (XRD) test was performed to investigate the phase composition of geopolymer composites when exposed to the elevated temperatures. The samples were prepared into powder form by cutting small geopolymer slices. The test was carried out using PANalytical X'pert PRO equipment in 2 h-range of 5 to 80 θ at operating conditions of 40 kV and 30 mA using a Cu ka X- ray source.

4.8 Physical properties of geopolymer composites

The hardness of geopolymer composites was measured on the Rockwell H scale using an Avery Rockwell hardness tester. The samples were polished with emery paper to achieve flat and smooth surfaces before the measurement. The test was repeated for 5 samples. The average of measurements and 95% confidence interval limits were taken. Furthermore, the values of bulk density was determined in accordance with the ASTM-C948 2014 using the Eq. (1) [29]. The test was repeated for 5 samples and an average of measurements was taken.

$$\text{Bulk density} = \frac{W_d}{W_a - W_i} \times \rho \quad (1)$$

Where W_d is dry specimen's mass after 24 h at 105 °C, W_i is specimen's mass immersed in water, W_a is saturated specimen's mass with a dry surface and ρ is the bulk density of water (kg m^{-3}). The average of measurements and 95% confidence interval limits were taken for measurements of 5 readings.

4.9 Compression strength of geopolymer composites

The geopolymer composites were tested for compression testing using Labor Tech universal testing machine, Czech Republic with load cell capacity of 2000 kN. The 40 mm cubes were tested for the determination of compression strength according to ASTM C109 standard. The test was repeated for 5 samples. The average of measurements and 95% confidence interval limits were taken.

4.10 Thermal stability of geopolymer composites

The thermo gravimetric analysis (TGA) was performed to know the thermal stability of geopolymer composites from weight loss with increase in temperature. It was conducted using TGA/SDTA 851 METLER TOLEDO analyzer. Samples with 10 mg were placed in an alumina crucible and tests were carried out in air atmosphere with a heating rate of 10 °C/min from 30 to 1000 °C.

5 Summary of the results achieved

5.1 Characterization of carbon and basalt micro fibers

Figure 2 (a) shows the particle size distribution results of basalt particles obtained after 30 min of dry milling. It can be observed that short basalt fibrous waste was transformed into basalt particles of micro to nano scale in multimodal distribution. With longer milling time, the basalt particles were found to deposit onto the walls of milling containers. This behavior was attributed to increase in temperature of ball mill and following cold welding of basalt particles on milling container [26]. For more homogeneous refinement of basalt particles to nano scale, it is essential to pulverize them for prolonged duration by overcoming the rise in temperature of ball mill. Figure 2 (b) showed the SEM image of microstructure of basalt particles after 30 min of dry milling. The shape of basalt particles was observed largely in the form of microfibrils with few particles below 10 μ scale.

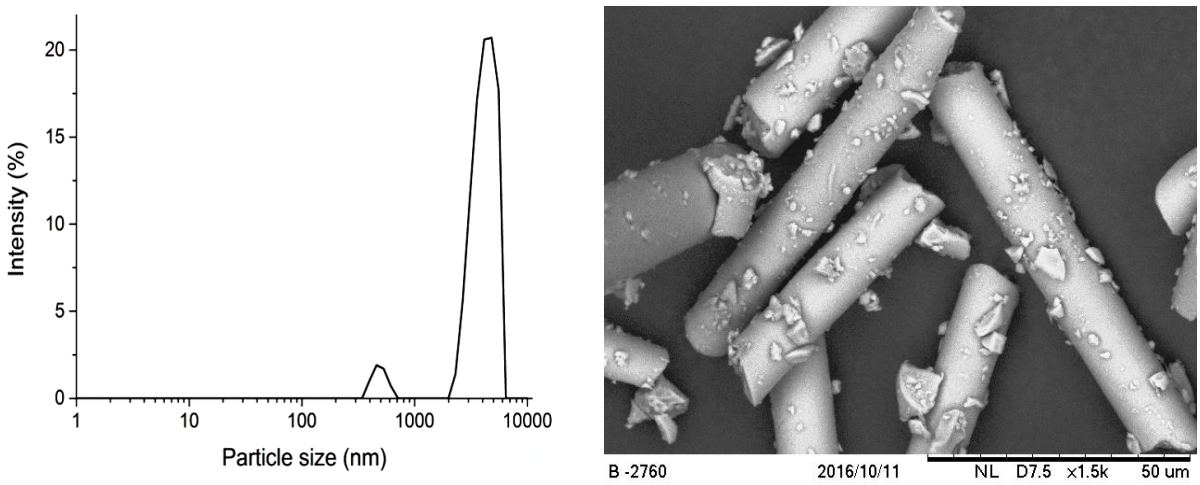


Figure 2. (a)Particle size distribution of basalt particles after 30 min dry milling (b). SEM image of basalt fibers after 30 min dry milling

Likewise, for uniform dispersion of carbiso mil 100 µ particles in geopolymer system, their surface was mechanically activated using 30 min dry pulverization. Figure 3 shows the particle size distribution results of carbiso mil 100 µ particles after dry milling. It can be seen that carbiso mil 100 µ particles were converted into fine carbon micro structures having multimodal distribution after 30 min dry milling. Further, the morphology of carbon particles was investigated with the help of SEM images shown in Figure 4. The shape of carbon particles was observed predominantly in the form of microfibers with few of microparticles below 10 µ scale. Unlike basalt particles, the deposition of carbon particles was found less severe with longer milling time. Therefore, the relative percentage of CMF or microparticles can be altered based on the duration of the milling action. The shorter milling time can produce more of microfibers and longer milling time can produce more of microparticles. The milling time of 30 min was fixed in this study because of the requirement of higher aspect ratio of CMF for effective reinforcement in composites.

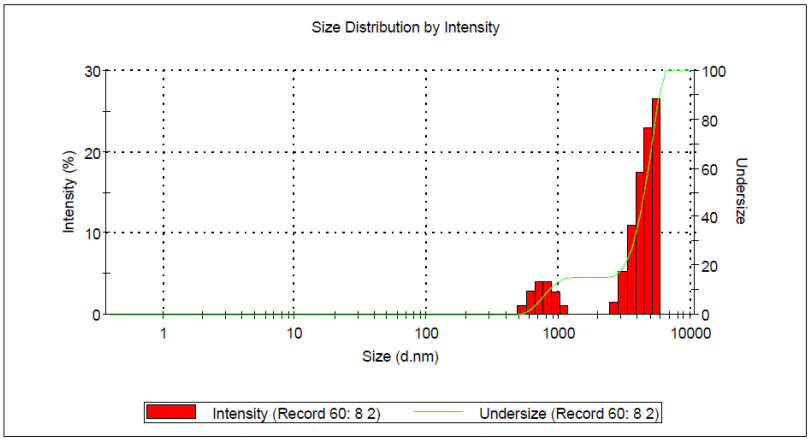
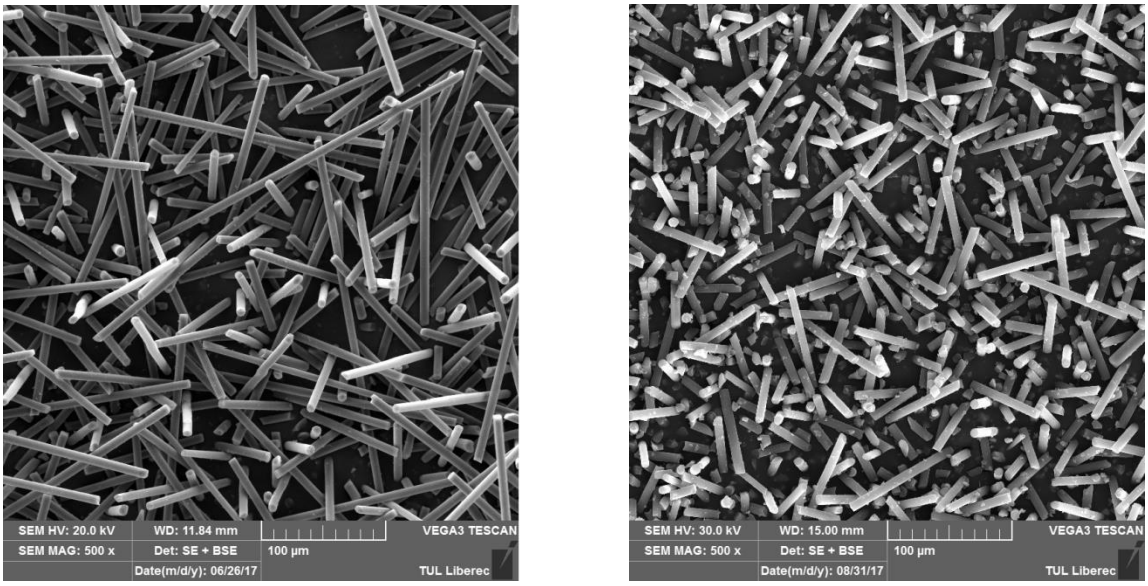


Figure 3. Particle size distribution of carbiso particles after 30 min dry milling



(a) before milling (b) after milling

Figure 4. Microstructure of carbiso powder

5.2 Microstructure analysis of geopolymer composites

The SEM micrographs of the neat geopolymer and BMF/geopolymer composites before and after exposure to the elevated temperatures are shown in Figure 5. The typical microstructure of homogeneous and dense matrix consisting mostly of alumino-silicate gel was viewed before exposure to the elevated temperatures. The micrographs of geopolymer composites demonstrated the smooth surfaces of BMF in the geopolymer matrix, which pointed out no degradation of basalt fibers owing to action of alkali in the activating solution. The BMF appeared to have reacted with the geopolymer matrix to some extent. The majority of the microfibrils were covered by the geopolymer, which pointed out possible physical bonding of geopolymer matrix with basalt fibers. In addition, the geopolymer composites exposed the chances of ductile failure from observations of indistinct cross-sections of basalt fiber ends. When the samples exposed to elevated temperatures, the development of higher bright crystals content, wider micro- cracks, and the relatively large voids were noticed. The compact microstructure of geopolymers became more porous at 800 ^{o}C, which might be caused by weight loss, matrix decomposition and phase transformations [30], [31]. The geopolymer composites revealed lesser microstructural deterioration at elevated temperatures than neat geopolymers and hence eventual less strength loss. This showed the formation of dense microstructure by BMF, which gave resistance to the penetration of heat. This can be attributed to the mechanical percolation along with pore filling effects of BMF at elevated temperatures [32], [33]. Further, the thermal resistance characteristics of BMF were identified from appearance of fibers in micrographs of samples exposed to 800 ^{o}C. The loose interface layer attributable to enlarged space between the matrix and microfibrils resulted in the strength reduction of geopolymer composites at increased temperature [34].

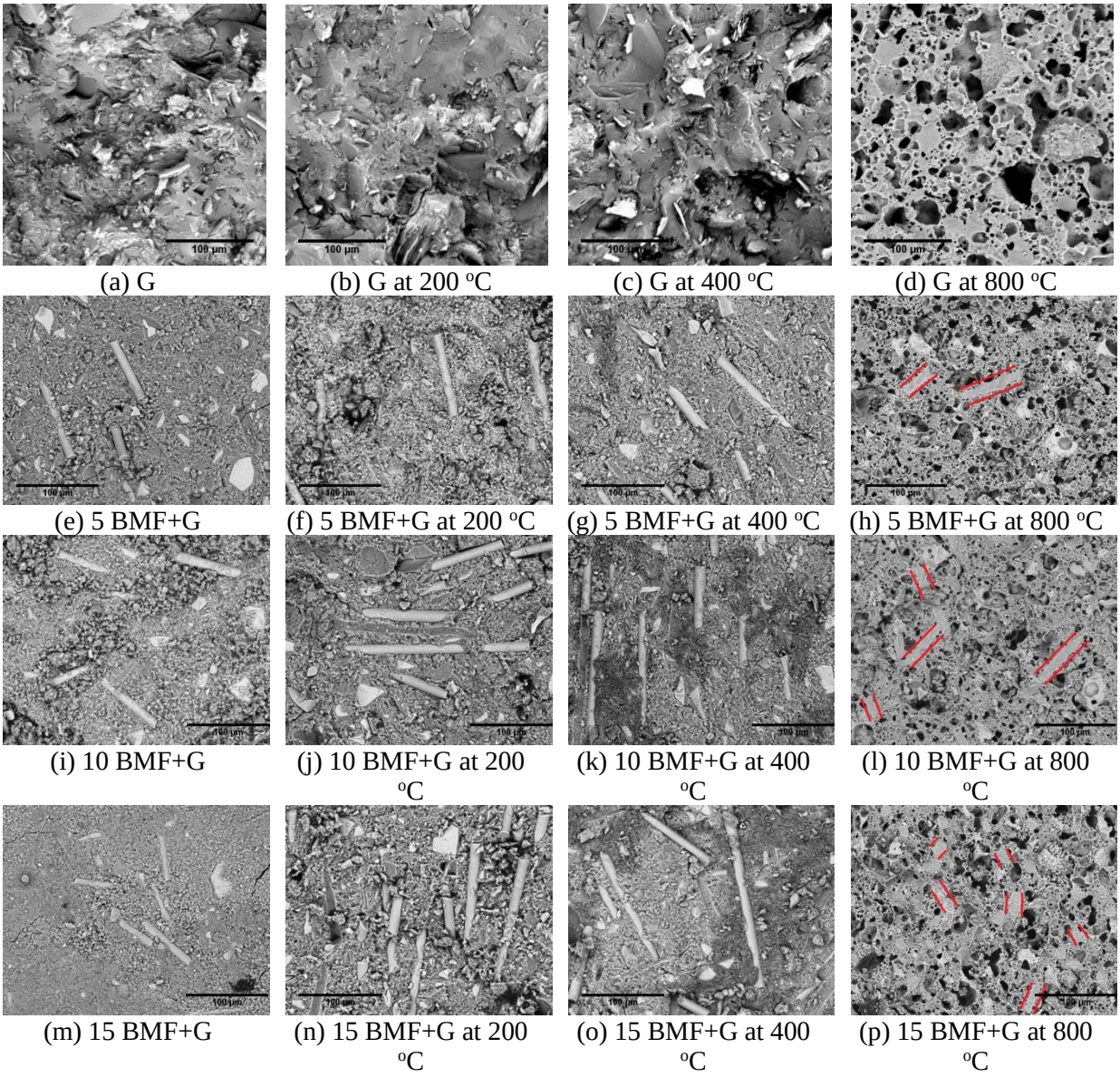


Figure 5. Microstructure of basalt microfibril/geopolymer composites at elevated temperature

The SEM micrographs of neat geopolymer and CMF/geopolymer composites at different temperature exposure are shown in Figure 6. The smooth surfaces of carbon fibers in the geopolymer matrix indicated no degradation of carbon fibers under action of alkali in the activating solution. The strong adhesion between the geopolymer gel and the surface of the fiber can be confirmed based on presence of geopolymer layer on fiber ends pulled out from the matrix and more striations on fiber surfaces [31]. Furthermore, the fractured surfaces of neat geopolymer showed straight cracks, whereas more number of curvilinear small cracks was found in case of geopolymer composites due to crack deflections by CMF. Therefore, it can be concluded that the addition of CMF ensured the effective toughening mechanism to prevent the catastrophic fracture of geopolymers. When the samples exposed to elevated temperatures, the geopolymer composites showed lower micro structural deterioration than neat geopolymers due to possible mechanical percolation along with pore filling effects of carbon micro fibers [32], [33]. This observation was further investigated by image analysis. The development of wider micro- cracks, higher bright crystals content and the relatively large voids were observed with increased temperature exposure. As discussed previously, this might be caused by weight loss, matrix decomposition and phase transformations in

geopolymers at higher temperature [30], [31]. The CMF did not exhibit any observable degradation after elevated temperature exposure. However, previous studies highlighted the significant degradation of polymeric fibers, glass fibers, basalt fibers, etc after such temperature exposure [35]. This indicated the thermal resistance characteristics of CMF that can continue to provide the reinforcement to geopolymers when exposed to higher temperatures and therefore less strength loss. Nevertheless, the development of loose interface layer caused by enlarged space between fibers and the matrix at elevated temperatures can possibly reduce the strength of geopolymers to some extent [34].

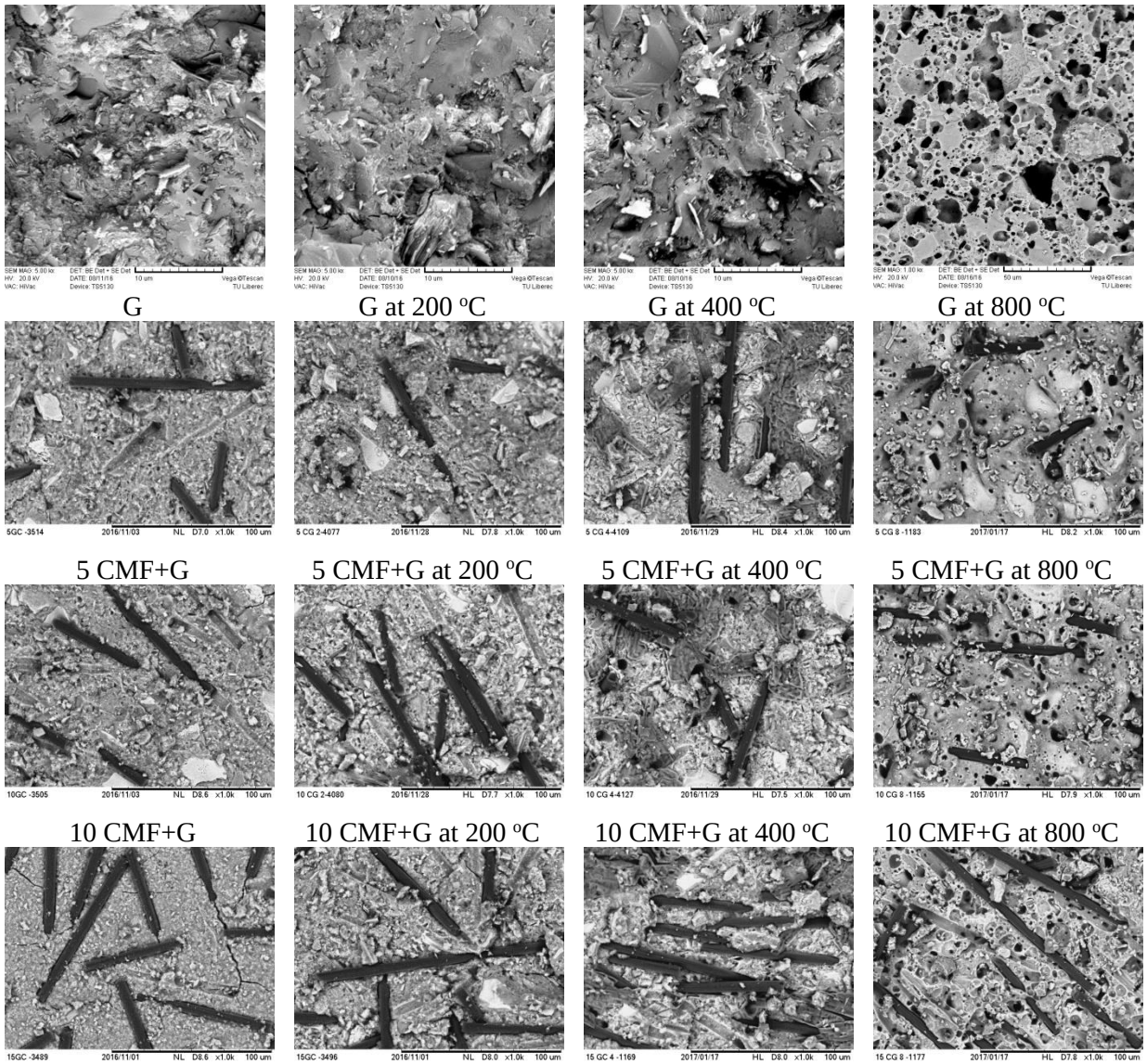


Figure 6. Typical fracture surface microstructure of carbon microfiber/geopolymer composites after exposure to elevated temperature

5.3 Image analysis of geopolymer composites

The quantitative analysis of the pore area is important to establish the relationships between microstructure and mechanical properties of geopolymer composites after exposure to elevated temperatures. In present work, image analysis was used for estimation of pore area analysis by observation

of large capillary pores and voids in binary images of Figure 7 and Figure 8. At first, SEM images were carefully converted into binary images by segmentation of Otsu thresholding method. The pore area was represented by black color in binary images and it was calculated in pixels by IMAGEJ software. Such 20 images of each sample were analyzed and average of pore area was determined (see Figure 9(a) and 9(b)). The pore area was found to reduce with increase in loading of BMF or CMF, which supported the previous observation of pore filling ability. However, the BMF/geopolymer composites depicted greater pore area than CMF/geopolymer composites across all range of to elevated temperature exposures. This indicated greater pore filling ability of CMF than BMF due to their thermal resistance properties across all elevated temperatures. Therefore, the better mechanical properties were expected from geopolymers filled with CMF as compared to BMF.

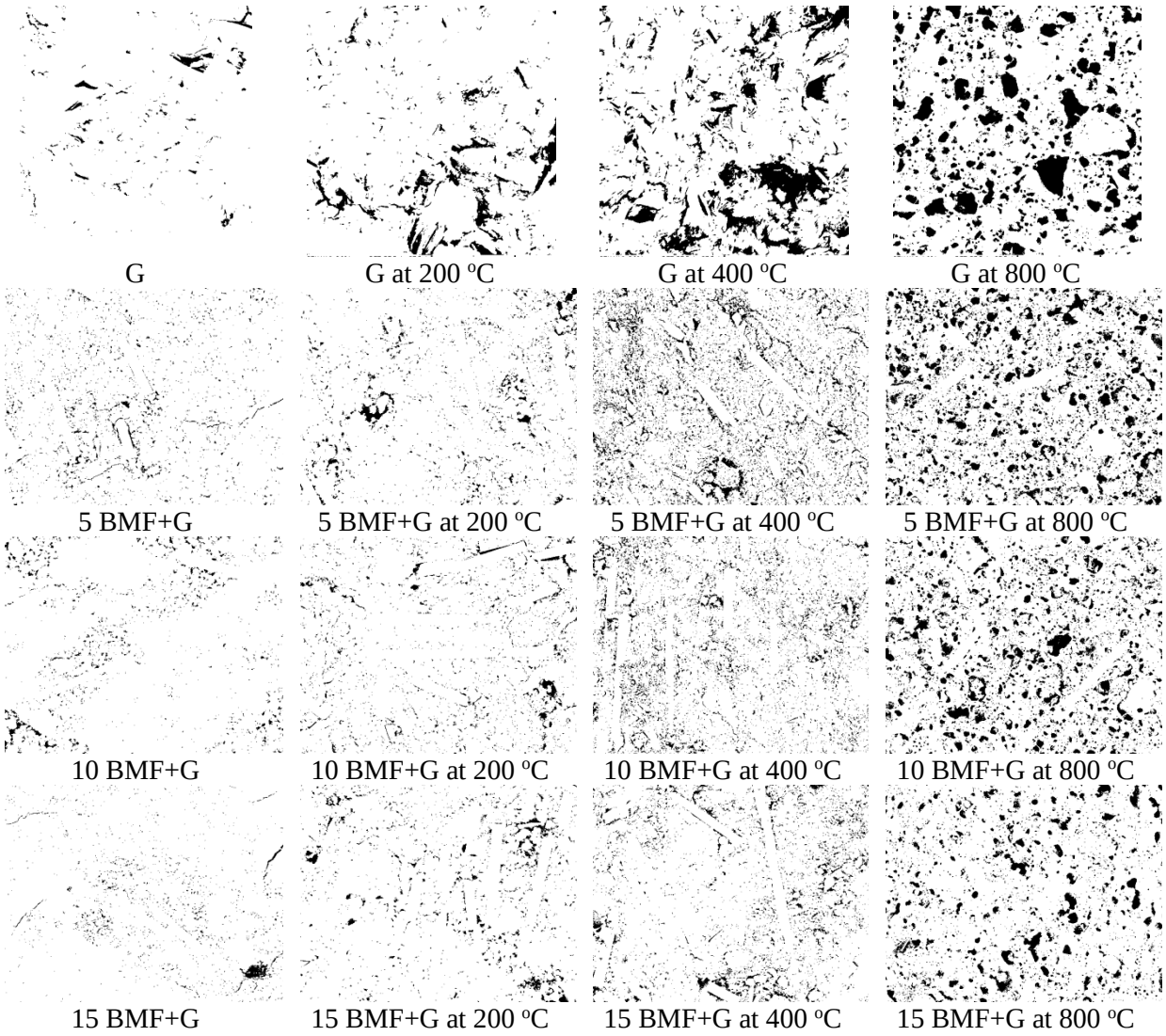


Figure 7. Estimation of pore area in basalt microfibril/geopolymer composites by image analysis

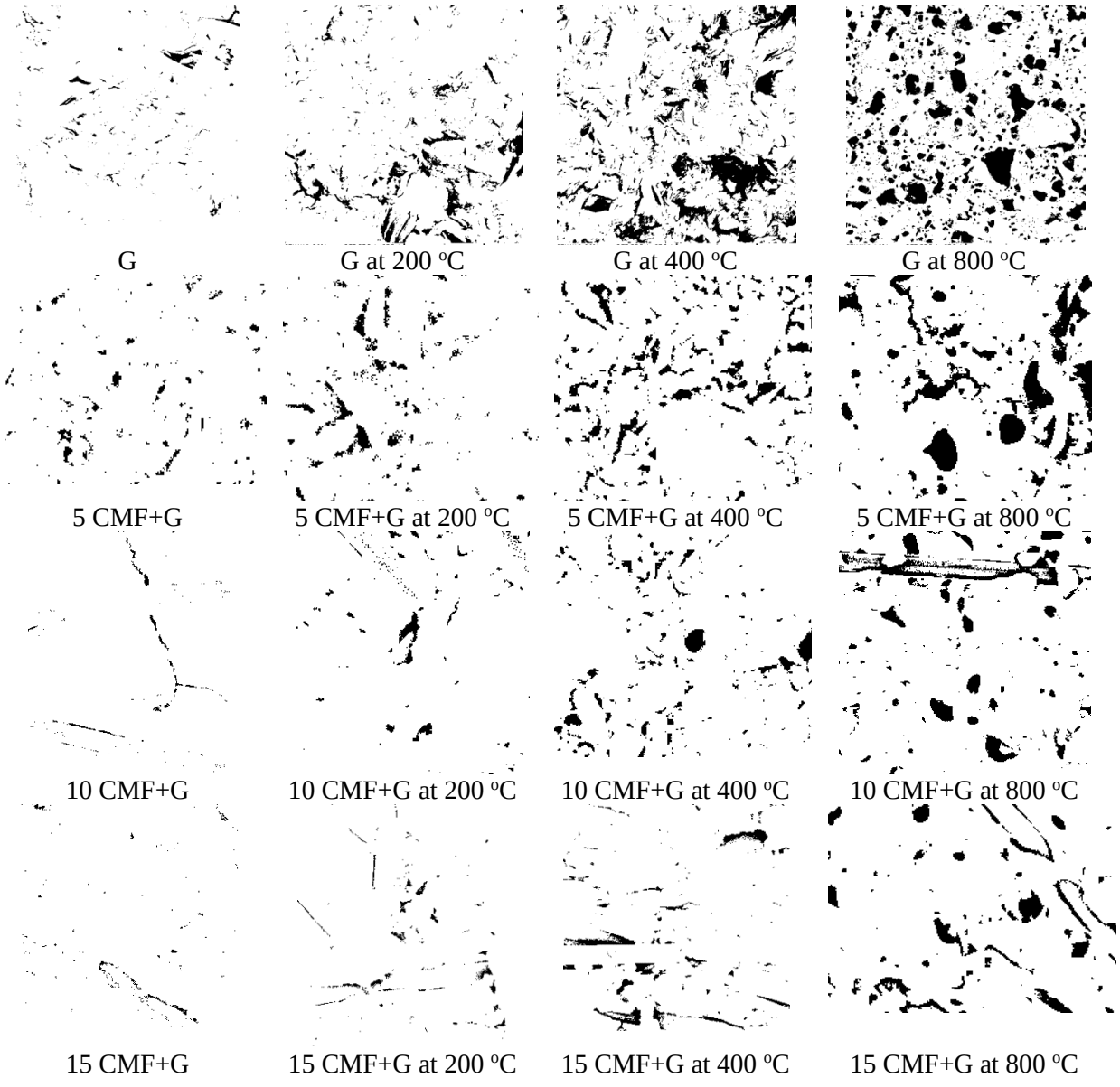
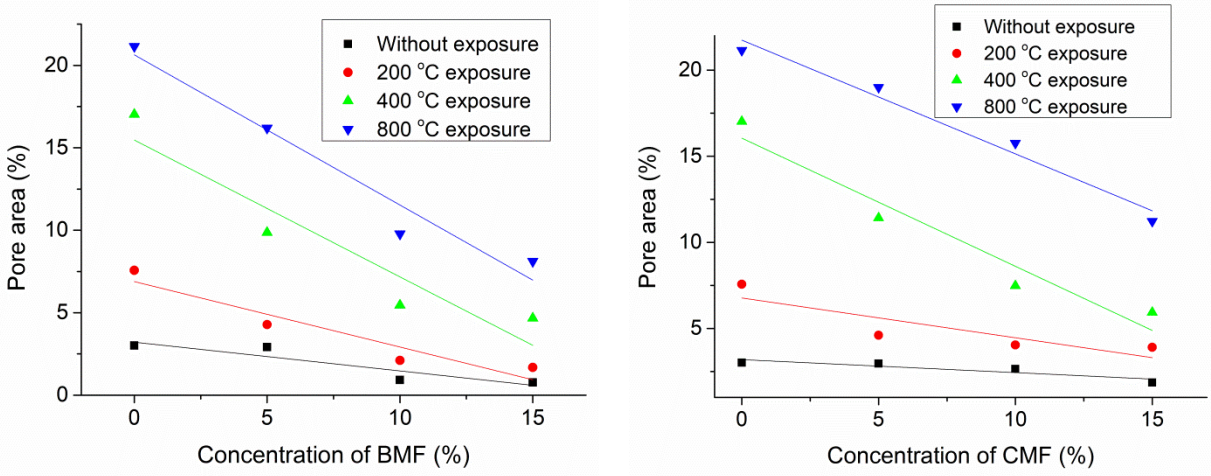


Figure 8. Estimation of pore area in carbon microfiber/geopolymer composites by image analysis



(a) BMF/geopolymer composites (b) CMF/geopolymer composites

Figure 9. Estimation of pore area of geopolymer composites

Further, the temperature (T) sensitivity in pore area (P) changes after addition of BMF or CMF in geopolymers was estimated by using method of least squares for linear regression Equation (2).

$$P = a + (b \times T) \qquad (2)$$

Table 2 shows the calculated parameters where slope indicates the sensitivity in changes of pore area. As compared to CMF/geopolymer composites, the slope of BMF/geopolymer composites was found to change significantly. This indicated better stability of CMF/geopolymer composites and greater thermal resistance of CMF than BMF. Further, the sensitivity in changes of pore area was found less at lower concentration of fillers; however it increased with increased concentration of BMF or CMF.

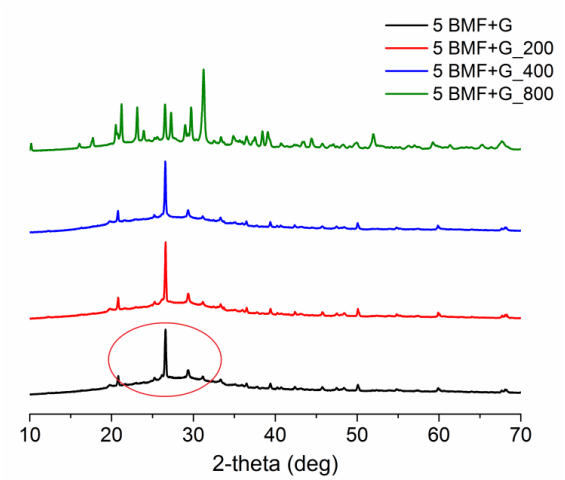
Table 2.Estimation of sensitivity of pore area changes by linear regression method

	Sample	Intercept	Slope	R^2
BMF geopolymer composites	Without exposure	3.20±0.49	-0.17±0.05	0.76
	200 °C exposure	6.88±0.84	-0.39±0.09	0.85
	400 °C exposure	15.47±1.88	-0.82±0.20	0.84
	800 °C exposure	20.63±1.27	-0.91±0.13	0.93
CMF geopolymer composites	Without exposure	3.18±0.22	-0.07±0.02	0.75
	200 °C exposure	6.76±0.87	-0.23±0.09	0.62
	400 °C exposure	16.04±1.20	-0.74±0.12	0.91
	800 °C exposure	21.73±0.70	-0.66±0.07	0.96

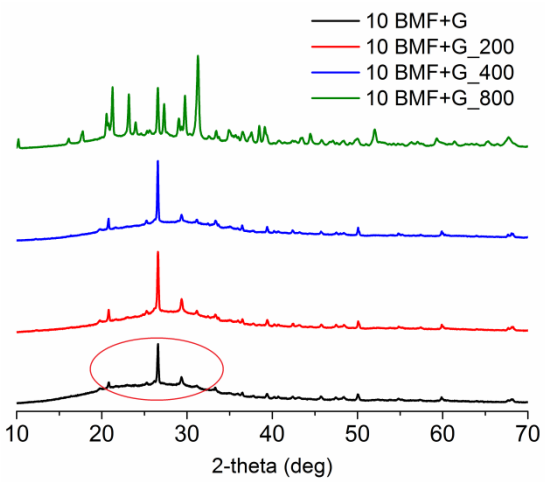
5.4 XRD analysis of geopolymer composites

Figure 10 shows the XRD patterns of samples when exposed to the elevated temperature of 200, 400, and 800 °C. A broad hump at 20–40° 2-theta can be found, which indicated the formation of amorphous gels of geopolymerization [36]. The formation of N-A-S-H gel was found in greater quantity than the C-A-S-H and (C, N)-A-S-H. As the formation of C-A-S-H and (C, N)-A-S-H depend on the availability of calcium ions and pH of the system [37], therefore the extra precipitation of calcium alumina silicate hydrates formation can be attributed to nucleating sites present on BMF. The calcium alumina silicate hydrates were not detected in XRD spectra due to its astable phase. The consistent appearance of broad hump from room temperature to 400 °C suggested the thermal resistance characteristics of prepared geopolymer composites. The geopolymer composite samples represented their original structural

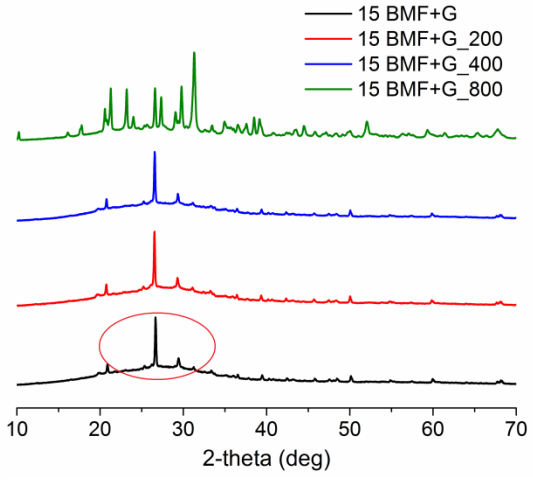
characteristics and there was no any new crystalline phase generated when exposed to elevated temperature upto 400 °C. At room temperature, the several characteristic peaks observed in the XRD pattern were identified as quartz, zeolite, thomsonite, goethite and semicrystalline hillebrandite. The better durability and thermal stability of geopolymers was ascribed to their zeolite-like structure characteristics [37]. The occurrence of these several characteristic peaks depend on type of aluminosilicate source, type of alkali activator, type of fillers, their mix-design in geopolymer composite, remaining unreacted silica or alumina in geopolymer, remaining unreacted other impurities in geopolymer, etc [38]. Nevertheless, on further increase in elevated temperature to 800 °C, the diffuse peaks disappeared and new Bragg peaks corresponding to new crystalline phases (i.e. akermanite, nepheline, gehlenite) were detected [39], [40]. The mechanism of crystallization at elevated temperature can be explained from the reaction of released calcium, silicon and aluminum from geopolymer gel and unreacted traces of metakaolin/basalt microfibril to form these intermediate products. However, the maximum retention of dimensional stability and strength of geopolymer composites can be expected due to formation of more nepheline phase at increased BMF loading [41].



(a) 5 BMF/geopolymer



(b) 10 BMF/geopolymer

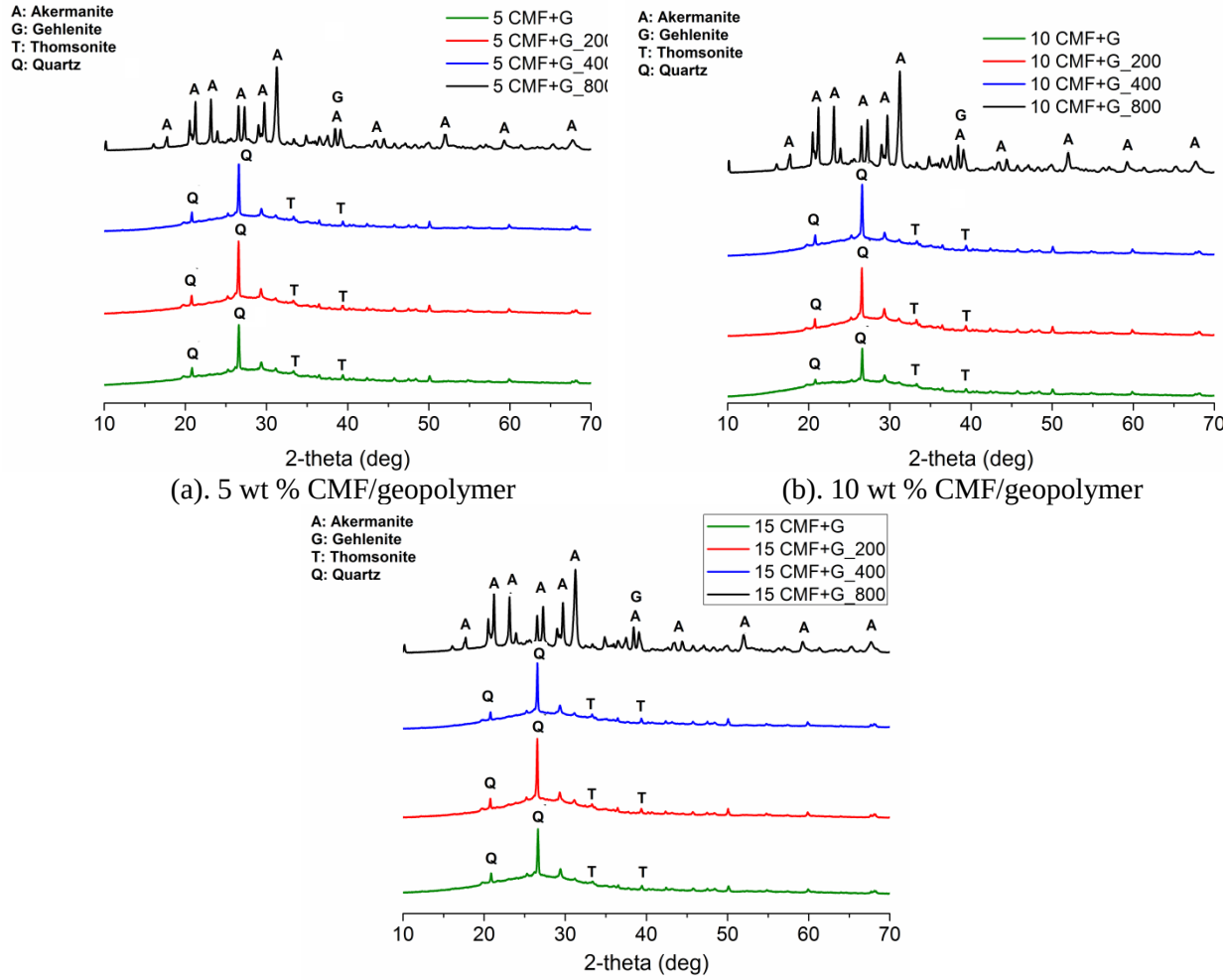


(c) 15 BMF/geopolymer

Figure 10. XRD analysis of basalt microfibril/geopolymer composites at elevated temperature

Similarly, the nature and composition of reaction products in CMF/geopolymer composites were investigated from XRD analysis. Figure 11 shows the XRD patterns of samples when exposed to the elevated temperature of 200, 400, and 800 °C. The formation of amorphous gels of geopolymerization can

be confirmed from the broad hump at 20–40° 2-theta [36], [37]. The more widening of this peak at higher carbon microfiber loading indicated the increased calcium silicate hydrates and more amorphous gel formation. Furthermore, the consistent appearance of this diffuse peak from room temperature to 400 °C suggested the thermal resistance characteristics of prepared geopolymer composites. When exposed to elevated temperature upto 400 °C, the geopolymer composite samples represented their original structural characteristics and there was no any new crystalline phase generated. The several characteristic peaks were identified as quartz, zeolite, thomsonite, goethite and semicrystalline hillebrandite till 400 °C. As mentioned in previously, the better durability and thermal stability of geopolymers was ascribed to their zeolite-like structure characteristics [37]. Nevertheless, on further increase in elevated temperature to 800 °C, the diffuse peaks disappeared and new Bragg peaks corresponding to new crystalline phases (i.e. akermanite, nepheline, gehlenite) were detected [39], [40]. This indicated the decomposition and crystallization of geopolymers at 800 °C, which can subsequently deteriorate their mechanical properties.



(a). 5 wt % CMF/geopolymer

(b). 10 wt % CMF/geopolymer

(c). 15 wt % CMF/geopolymer

Figure 11. XRD analysis of carbon microfiber/geopolymer composites

5.5 Physical properties of geopolymer composites

Table 3 illustrates the physical properties (i.e. hardness and bulk density) of the neat geopolymer and BMF/geopolymer composites before and after exposure to elevated temperature. The hardness describes the ability of a material to resist plastic deformation under indentation. Across all range of

temperature exposures, the geopolymers showed improved hardness with increased loading of BMF. This explained the uniform distribution of the load on the BMF, which reduced the penetration of the test ball at the surface of the geopolymer. Further, the higher hardness could be attributed to the extra precipitation of calcium alumina silicate hydrates formation due to nucleating sites present on BMF [24]. However, when the samples were exposed to elevated temperature of 200, 400 and 800 ^{o}C, all the samples showed reduction in bulk density and hardness values. This behavior was attributed to evaporation of water and change in Si/Al ratio as temperature increased [42], [43]. A similar phenomenon was observed previously which resulted in foam like structures by formation and growth of bubbles with increasing the Si/Al ratio [44]. At 800 ^{o}C of elevated temperature exposure, the neat geopolymer showed 13 % reduction in density, whereas 10 wt % basalt microfibril filled geopolymer composites showed 8 % density reduction.

Table 3. Physical properties of basalt microfibril/geopolymer composites at elevated temperature

Temperature (^{o}C)	G		5 BMF+G		10 BMF+G		15 BMF+G	
	Hardness (HV)	Density (kg/m^3)	Hardness (HV)	Density (kg/m^3)	Hardness (HV)	Density (kg/m^3)	Hardness (HV)	Density (kg/m^3)
30	536±46	1510±94	578±58	1560±108	567±60	1570±110	563±64	1550±106
200	395±32	1490±89	402±41	1500±112	419±45	1520±113	483±52	1520±112
400	290±23	1402±86	300±35	1440±109	306±38	1450±115	325±41	1440±113
800	330±37	1310±93	-	-	-	-	-	-

Similarly, the physical properties of neat geopolymer and CMF/geopolymer composites before and after exposure to elevated temperature are illustrated in Table 4. The density was found to reduce with increase in carbon microfiber loading. The carbon microfiber filled geopolymers exhibited significant increase in viscosity due to high aspect ratio and smooth light surfaces of microfibers. This subsequently resulted into the entrapment of more air and thus possible reduction in density of geopolymer composites than neat geopolymers [31]. From Table 4, the hardness of geopolymer was found to increase with increased loading of CMF across all range of temperature exposures. The similar explanation of uniform distribution of the load on the CMF which reduced the penetration of the test ball at the surface of the geopolymer can be given for enhancement in hardness values. Likewise in the case of BMF, all the CMF/geopolymer composites showed reduction in bulk density and hardness values when exposed to elevated temperature of 200, 400 and 800 ^{o}C. However, drop in hardness of CMF/geopolymer composites was less as compared to BMF/geopolymer composites. This showed intact structure of CMF/geopolymer composites at elevated temperatures due to effective pore-filling effect of carbon micro fibers as compared to BMF.

Table 4. Physical properties of carbon microfiber/geopolymer composites

Temperature (^{o}C)	G		5 CMF+G		10 CMF+G		15 CMF+G	
	Hardness (HV)	Density (kg/m^3)	Hardness (HV)	Density (kg/m^3)	Hardness (HV)	Density (kg/m^3)	Hardness (HV)	Density (kg/m^3)
30	536±46	1510±94	558±52	1480±102	569±51	1490±106	562±55	1480±104
200	395±32	1490±89	489±48	1440±108	494±46	1510±103	482±42	1480±109
400	290±23	1402±86	435±45	1400±111	482±49	1360±113	577±45	1350±112
800	330±37	1310±93	367±41	1270±107	371±45	1260±110	379±43	1220±108

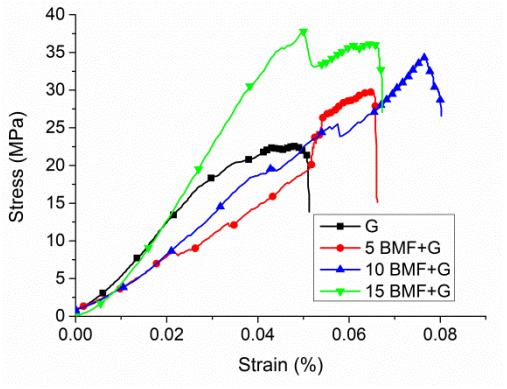
5.6 Compression strength of geopolymer composites

Figure 12 shows the compression strength results of geopolymer and BMF/geopolymer composites before and after exposure to elevated temperatures. The geopolymer composites showed higher compression strength than neat geopolymers over all range of temperature exposures. From stress-strain curve, the neat geopolymer indicated a typical brittle failure mode, whereas geopolymer composites exhibited an extended period of plastic deformation (i.e. pseudoplastic behavior) unlike short drop at the point of maximum load. This non-linear behavior of geopolymer composites can be explained from the fiber-bridging and sliding after debonding and pulling-out of BMF from the geopolymer matrix. This further indicated more favorable interaction between BMF and the matrix possibly due to a combination of physical and chemical bonding. All samples showed increase in compression strength with increase in temperature till 200 ^{o}C. This behavior was attributed to the formation of discontinuous nano-pores and dehydration shrinkage of geopolymers due to expel of free water at 200 ^{o}C [43]. Nevertheless, with further increase in elevated temperature at 400 and 800 ^{o}C, all samples showed deterioration in compression strength. This phenomenon resulted due to the thermal incompatibility (i.e. differential thermal expansion between geopolymer and BMF), pore pressure effects (i.e. movement of free water and hydroxyls) and possible phase transition in geopolymers [38], [42]. At elevated temperature exposure, several events such as evaporation of water adsorbed by N-A-S-H gel, formation of anhydrous products, crystallization of stable anhydrous phases and melting (sintering) occurred, which subsequently deteriorated the mechanical properties [38]. The less deterioration for geopolymer composites indicated the thermal resistance characteristics of geopolymers after the addition of basalt microfibril. This behavior can be further explained from the results of pore area (see Figure 9 (a)), where basalt microfibril acted as effective pore filling agents and exhibited a very limited development of macro-cracks. This decreased the thermal stresses on geopolymer composite pastes at elevated temperature exposure and maintained higher residual mechanical properties [45]. The geopolymer composite of 10 wt % basalt microfibril maintained the residual compressive strengths of 23.13 and 16.08 MPa at 400 ^{o}C and 800 ^{o}C, respectively and thus recording a minimum strength loss of 32 and 43 %, respectively (Table 5). On the other hand, the neat geopolymers exposed to 800 ^{o}C crumbled into fine particles rather than small broken blocks after the compression strength testing. This indicated the loss of bonding capacity of geopolymers in absence of BMF when exposed to elevated temperatures. Furthermore, the filling of basalt microfibers showed higher compression strength values than the previously reported results on neat OPC when exposed to elevated temperatures [46] (see Figure 13). The significant improvement was found for 800 ^{o}C exposure, where filling of BMF showed higher values of compression strength compared to OPC. The percentage increase over cement was calculated from the Equation (3).

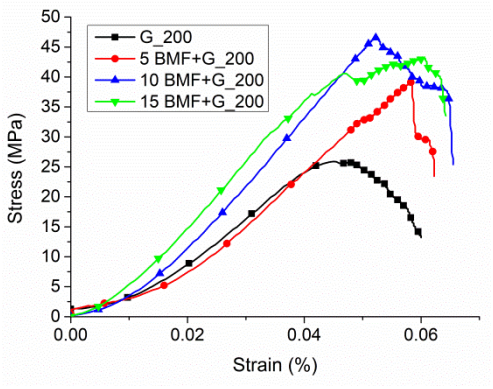
$$\text{Percentage increase over cement} = \frac{\sigma_{gc} - \sigma_{opc}}{\sigma_{opc}} \times 100 \qquad (3)$$

Where σ_{gc} is compression strength of geopolymer composites and

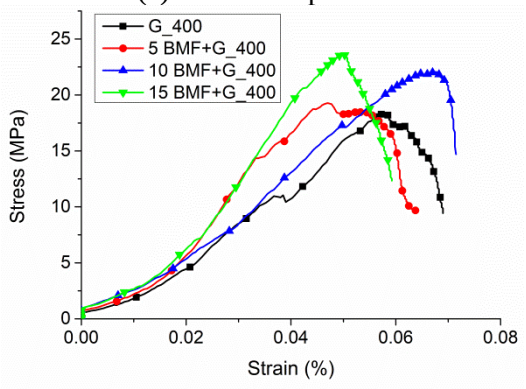
σ_{opc} is compression strength of OPC



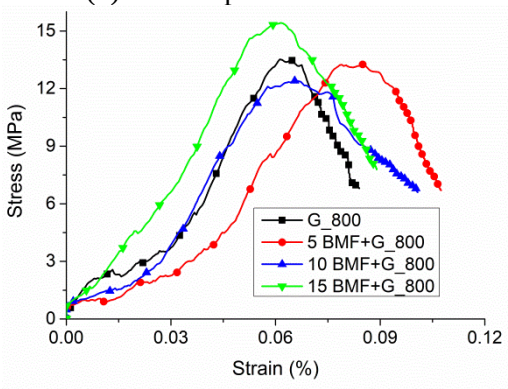
(a) Without exposure



(b) After exposure to 200 ºC



(c) After exposure to 400 ºC

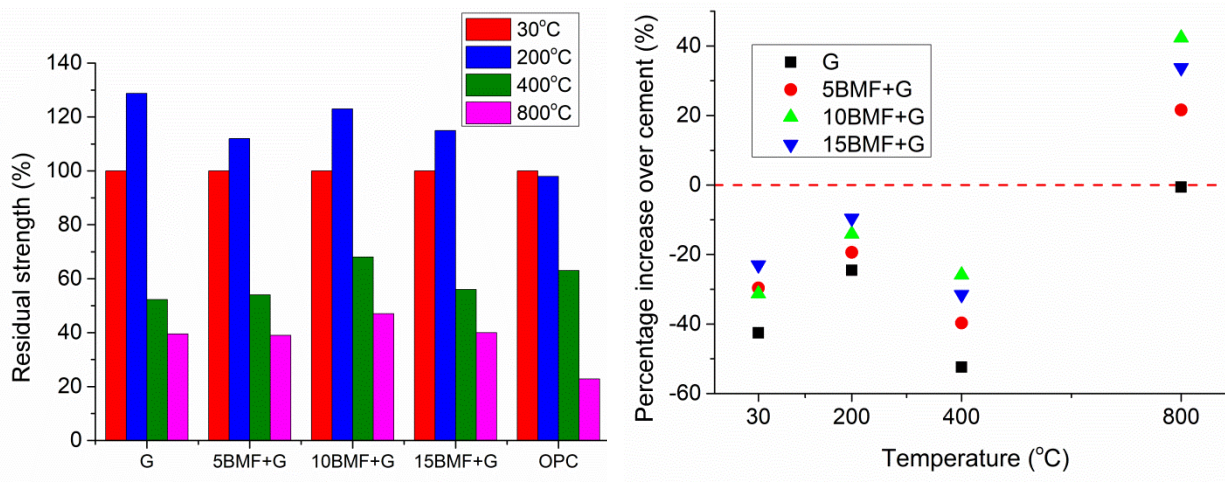


(d) After exposure to 800 ºC

Figure 12. Stress-strain curve for BMF/geopolymer composites

Table 5. Compression strength of basalt microfibril/geopolymer composites at elevated temperature

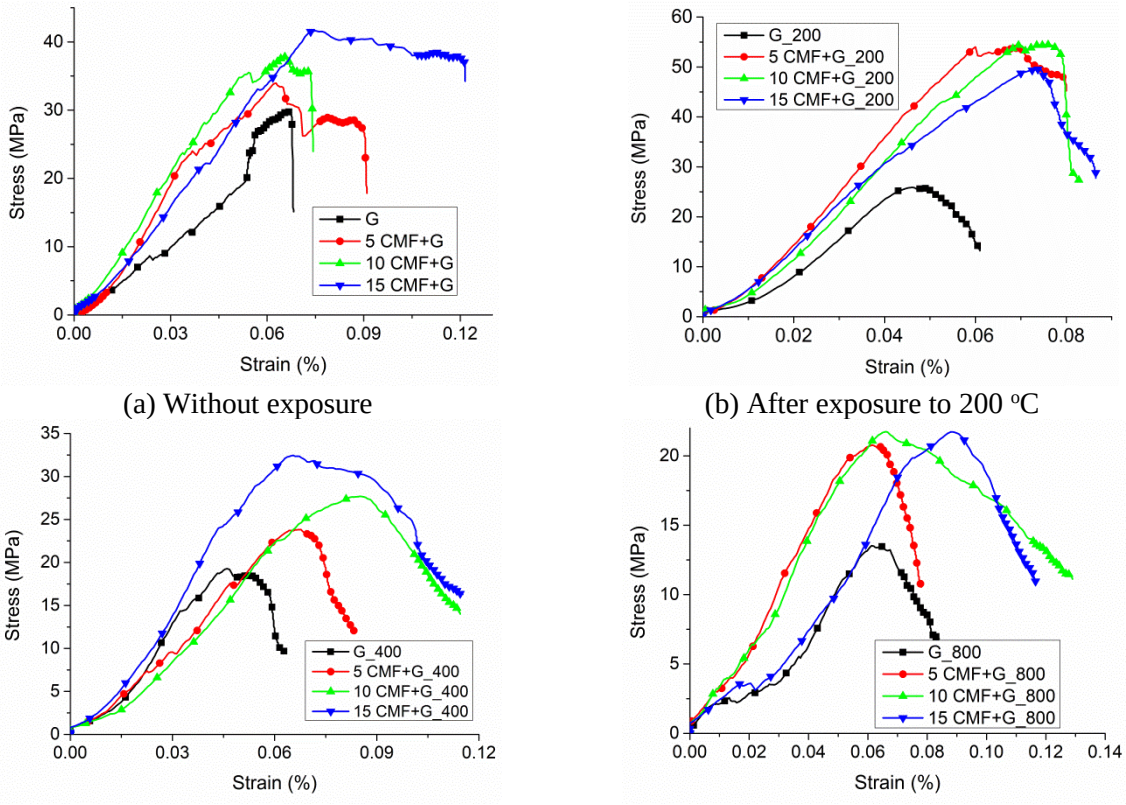
Temperature (ºC)	G	5 BMF+G	10 BMF+G	15 BMF+G	OPC
	Compressive strength (MPa)	Compressive strength (MPa)	Compressive strength (MPa)	Compressive strength (MPa)	Compressive strength (MPa)
30	28.43±2.5	34.82±3.1	34.00±3.2	38.10±3.5	49.5
200	36.61±3.2	39.11±3.5	41.65±3.9	43.85±4.4	48.5
400	14.85±1.9	18.82±2.1	23.13±2.7	21.36±2.5	31.2
800	11.23±2.2	13.74±2.4	16.08±2.5	15.11±2.6	11.3



(a) Residual strength (b) Percentage increase

Figure 13. Compression strength comparison of BMF/geopolymer composites with OPC

Similarly, figure 14 showed the compression strength results of geopolymer and CMF/geopolymer composites before and after exposure to elevated temperatures. The stress-strain curve of CMF/geopolymer composites showed larger strain values than BMF/geopolymer composites. This indicated more pseudoplastic behavior in CMF/geopolymer composites and somewhat brittle behavior of BMF/geopolymer composites. The compression strength of CMF/geopolymer composites was found greater than the compression strength of BMF/geopolymer composites over all range of temperature exposures. The geopolymer composite of 15 wt % carbon micro fiber kept up the residual compressive strengths of 33.55 MPa and 23.96 MPa at 400 ºC and 800 ºC, respectively and therefore recording a minimum strength loss of 19 and 42 %, respectively (Table 6). This proved more favorable interaction of CMF with geopolymer as compared to BMF with geopolymer. Likewise, CMF/geopolymer composites depicted higher compression strength values than the previously reported results of neat OPC when exposed to elevated temperatures (Figure 15). As compared to BMF/geopolymer composites, the percentage increase over OPC strength was found higher in case CMF/geopolymer composites.



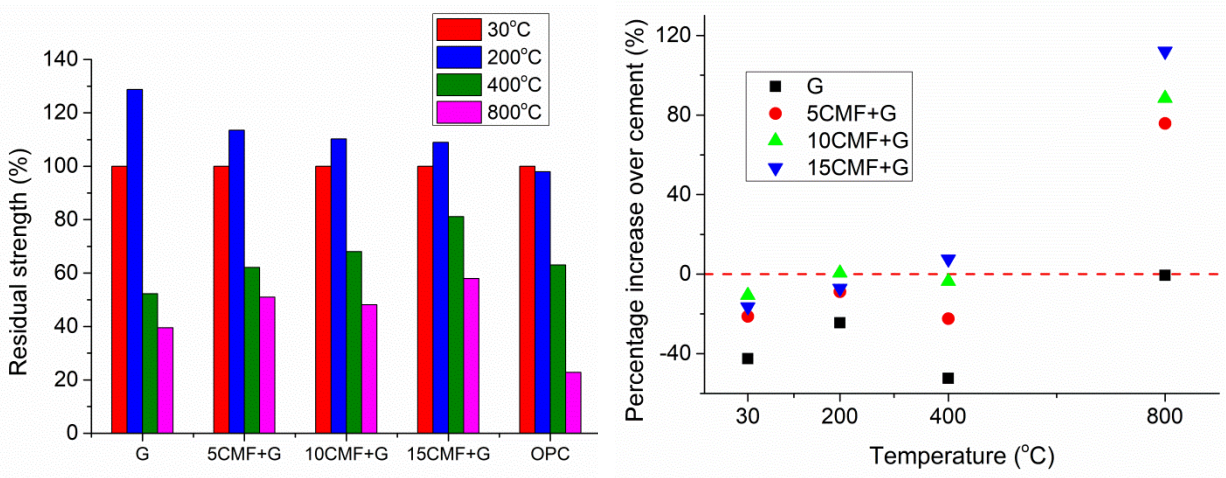
(a) Without exposure (b) After exposure to 200 ºC

(c) After exposure to 400 ºC (d) After exposure to 800 ºC

Figure 14. Stress-strain curve for CMF/geopolymer composites

Table 6. Compression strength of carbon microfiber/geopolymer composites

Temperature (ºC)	G	5 CMF+G	10 CMF+G	15 CMF+G	OPC
	Compressive strength (MPa)	Compressive strength (MPa)	Compressive strength (MPa)	Compressive strength (MPa)	Compressive strength (MPa)
30	28.43±2.5	38.97±4.1	44.22±4.7	41.33±4.3	49.5
200	36.61±3.2	44.23±4.3	48.77±4.8	45.04±4.6	48.5
400	14.85±2.1	24.21±2.7	30.08±3.3	33.55±3.8	31.2
800	11.23±2.2	19.86±2.3	21.29±2.8	23.96±3.1	11.3



(a) Residual strength (b) Percentage increase

Figure 15. Compression strength comparison of CMF/geopolymer composites with OPC

6 Evaluation of results and new findings

The presented thesis studied the role of basalt and carbon microfibers on improvement in elevated temperature properties of metakaoline based geopolymers. The 30 min dry pulverization of basalt fibrous wastes and carbiso powder was carried out in high energy ball milling to obtain respective basalt and carbon microfibers. Further, the geopolymer composites were prepared by addition of 5, 10 and 15 wt % of carbon/basalt microfibers and later exposed to the elevated temperatures of 200, 400, and 800 °C. The performance of basalt and carbon microfibers was evaluated based on measurements of physical properties, micro structural analysis and compression strength of geopolymer composites. Both geopolymer composites showed higher hardness, higher bulk density and compact structure than neat geopolymers over all range of temperature exposures. This was related to presence of inorganic contents in both microfibers, which produced additional calcium silicate or calcium alumino-silicate and sodium alumino-silicate hydrates. Nevertheless, more compact structure of geopolymers was found after addition of CMF due to effective pore filling characteristics and higher thermal resistance than BMF. On the other hand, the development of wider micro-cracks, higher bright crystals content and the relatively large voids were observed in case of BMF/geopolymer composites. Therefore, the surface-cracking and internal damage of the geopolymer structure was reported to cause the reduction in the strength of geopolymers. The compression strength deteriorated significantly in case of BMF/geopolymer composites than CMF/geopolymer composites at 400 and 800 °C, which was attributed to thermal incompatibility (i.e. differential thermal expansion between geopolymer and basalt micro fibers), pore pressure effects (i.e. movement of free water and hydroxyls) and possible phase transition in geopolymers at elevated temperature. The less deterioration for CMF/geopolymer composites indicated the thermal resistance characteristics of geopolymers after the addition of carbon micro fibers, which further decreased the thermal stresses and restricted the swelling of unreacted geopolymer phases. Towards the end, the performance of geopolymer composites was compared with previously reported studies on elevated temperature properties of OPC binders. The geopolymers filled by BMF and CMF showed higher compression strength values than the previously reported results on neat OPC when exposed to 800 °C. The 5, 10 and 15 wt% BMF filled geopolymers showed 22 %, 42 %, and 34 % increase over OPC respectively, whereas 5, 10 and 15 wt% CMF filled geopolymers showed 76 %, 88 % and 112 % increase over OPC respectively.

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8 List of papers published by the author

8.1 Publications in International Journals:

1. **P. Behera**, V. Baheti, J. Militky, and S. Naeem, "Microstructure and mechanical properties of carbon microfiber reinforced geopolymers at elevated temperatures," *Constr. Build. Mater.*, vol. 160, pp. 733–743, Jan. 2018. ***[Impact factor*=4.685*]***
2. **P. Behera**, V. Baheti, J. Militky, and P. Louda, "Elevated temperature properties of basalt microfibril filled geopolymer composites," *Constr. Build. Mater.*, vol. 163, pp. 850–860, Feb. 2018. ***[Impact factor*=4.685*]***
3. M. Salman Naeem, V. Baheti, J. Militky, V. Tunakova and **P. Behera** "Impact of carbonization temperature on activated carbon web for EMI shielding and ohmic heating," *Vlakna a Text.*, vol. 25, no. 3, pp. 57–62, 2018. ***[Impact factor*=0.3*]***
4. **P. Behera**, V. Baheti, J.Militky, P. Louda, S. Naeem, "Role of carbon microfibers on elevated temperature properties of geopolymers" *Vlakna a Text.*, vol. 25, no. 4, pp. 3–7, 2018. ***[Impact factor*=0.3*]***
5. S. Faheem, V. Baheti, **P. Behera**, and S. Naeem, "Development of flame retardant high loft polyester nonwovens," *J. Text. Inst.*, vol. 108, no. 8, pp. 1357–1364, Aug. 2017. ***[Impact factor*=1.063*]***
6. S. Naeem, V. Baheti, J. Militky, J. Wiener, **P. Behera**, and A. Ashraf, "Sorption properties of iron impregnated activated carbon web for removal of methylene blue from aqueous media," *Fibers Polym.*, vol. 17, no. 8, pp. 1245–1255, Aug. 2016. ***[Impact factor*=1.439*]***

Future publications:

1. **P. Behera**, H. Savastano, "Microstructure and mechanical properties of basalt waste fibre reinforced cementicious composite through slurry dewatering method".
2. **P. Behera**, H. Savastano, G. Carmello "Mechanical, physical and microstructural properties of basalt fiber reinforced hybrid composites at different curing conditions through extrusion process".
3. **P. Behera**, H. Savastano, G. Carmello "Evaluation of mechanical, physical and microstructure performance of extruded cementitious matrices reinforced with micro and macro basalt fibrous wastes".
4. **P. Behera**, A. Koch, T. Gries, J. Militky "Feasibility analysis - using recycled carbon short fibres for FRC".

8.2 International Conference Papers

1. **P. Behera**, V. Baheti, A. Koch, J. Wiener, J. Militky, and T. Ltd, Feasibility analysis - using recycled carbon short fibres for trc, 9th International Conference on Nanomaterials - Research & Application, 2018.
2. **P. Behera**, V. Baheti, D. Karthik, J. Militky, "Mechanical performance of basalt and carbon microfibril filled geopolymer composites at elevated temperature" 44th Textile research symposium, 2016.
3. S. Naeem, S. Javed, V. Baheti, J. Militky, Z. Ahmed, **P. Behera**, Effect of temperature, heating rate and holding time on the properties of Carbon web made from Acrylic waste, Strutex conference, December 2016.
4. **P. Behera**, V. Baheti, J. Militky, P. Louda, mechanical performance of basalt microfibril filled geopolymer composites at elevated temperature, the 25th annual international conference on composites/nano engineering (icce-25), 2017.
5. **P. Behera**, V. Baheti, J. Militky, P. Louda, microstructure and mechanical properties of carbon microfiber reinforced geopolymers at elevated temperatures, the 25th annual international conference on composites/nano engineering (icce-25), 2017.

8.3 Book chapters

1. V. Baheti, **P. Behera**, J. Militký, D. Karthik, Fly Ash Based Geopolymer Concrete Materials, Recent Developments in Fibrous Material Science, Pages 403-412. ISBN: 978-80-87269-45-9.
2. V. Baheti, **P. Behera**, D. Karthik, J. Militký, Properties of basalt fibres suitable in concrete composites, Advances in fibrous material science, Pages 95-118. ISBN: 978-80-87269-48-0.

8.4 Citations

Article: P. Behera, V. Baheti, J. Militky, and P. Louda, "Elevated temperature properties of basalt microfibril filled geopolymer composites," *Constr. Build. Mater.*, vol. 163, pp. 850–860, Feb. 2018.

Cited in

1. A. A. Arslan *et al.*, "Influence of wetting-drying curing system on the performance of fiber reinforced metakaolin-based geopolymer composites," *Constr. Build. Mater.*, vol. 225, pp. 909–926, Nov. 2019.
2. M. Abdulkareem, J. Havukainen, and M. Horttanainen, "How environmentally sustainable are fibre reinforced alkali-activated concretes?," *J. Clean. Prod.*, vol. 236, p. 117601, Nov. 2019.
3. D. Shi, Y. Yao, J. Ye, and W. Zhang, "Effects of seawater on mechanical properties, mineralogy and microstructure of calcium silicate slag-based alkali-activated materials," *Constr. Build. Mater.*, vol. 212, pp. 569–577, Jul. 2019.
4. Š. Hýsek *et al.*, "Fire-Resistant Sandwich-Structured Composite Material Based on Alternative Materials and Its Physical and Mechanical Properties," *Materials (Basel).*, vol. 12, no. 9, p. 1432, May 2019.

5. M. M. Szczypinski *et al.*, “Evaluation of mechanical properties of composite geopolymer blocks reinforced with basalt fibres,” *Manuf. Technol.*, 2018.
6. P. Behera, V. Baheti, J. Militky, and S. Naeem, “Microstructure and mechanical properties of carbon microfiber reinforced geopolymers at elevated temperatures,” *Constr. Build. Mater.*, vol. 160, pp. 733–743, Jan. 2018.

Article: P. Behera, V. Baheti, J. Militky, and S. Naeem, “Microstructure and mechanical properties of carbon microfiber reinforced geopolymers at elevated temperatures,” *Constr. Build. Mater.*, vol. 160, pp. 733–743, Jan. 2018.

Cited in

1. S. Saha, N. Shaik, and C. Rajasekaran, “Volume Change Characteristics of Eco-Friendly Mortar Mixes Produced with Geopolymeric Binder and Recycled Fine Aggregate,” *J. Test. Eval.*, vol. 48, no. 1, p. 20180316, Jan. 2020.
2. Y. Rifaai, A. Yahia, A. Mostafa, S. Aggoun, and E.-H. Kadri, “Rheology of fly ash-based geopolymer: Effect of NaOH concentration,” *Constr. Build. Mater.*, vol. 223, pp. 583–594, Oct. 2019.
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4. K. Sotiriadis, S. G. Guzii, P. Mácová, A. Viani, K. Dvořák, and M. Drdácký, “Thermal Behavior of an Intumescent Alkaline Aluminosilicate Composite Material for Fire Protection of Structural Elements,” *J. Mater. Civ. Eng.*, vol. 31, no. 6, p. 04019058, Jun. 2019.
5. A. Saccani, S. Manzi, I. Lancellotti, and L. Lipparini, “Composites obtained by recycling carbon fibre/epoxy composite wastes in building materials,” *Constr. Build. Mater.*, vol. 204, pp. 296–302, Apr. 2019.
6. Z. Tang, W. Li, Y. Hu, J. L. Zhou, and V. W. Y. Tam, “Review on designs and properties of multifunctional alkali-activated materials (AAMs),” *Constr. Build. Mater.*, vol. 200, pp. 474–489, Mar. 2019.
7. M. T. Tran, X. H. Vu, and E. Ferrier, “Experimental and analytical analysis of the effect of fibre treatment on the thermomechanical behaviour of continuous carbon textile subjected to simultaneous elevated temperature and uniaxial tensile loadings,” *Constr. Build. Mater.*, vol. 183, pp. 32–45, Sep. 2018.
8. A. Saludung, Y. Ogawa, and K. Kawai, “Microstructure and mechanical properties of FA/GGBS-based geopolymer,” *MATEC Web Conf.*, vol. 195, p. 01013, Aug. 2018.
9. J. Novotna, V. Baheti, B. Tomkova, J. Militky, and J. Novak, “Development of Multilayered Nanocomposites for Applications in Personal Protection,” *Fibers Polym.*, vol. 19, no. 6, pp. 1288–1294, Jun. 2018.
10. P. Behera, V. Baheti, J. Militky, and P. Louda, “Elevated temperature properties of basalt microfibril filled geopolymer composites,” *Constr. Build. Mater.*, vol. 163, pp. 850–860, Feb. 2018.
11. N.-E.-H. Fardjaoui, B. Wicklein, P. Aranda, I. Sobrados, F. Z. El Berrichi, and E. Ruiz-Hitzky, “Modulation of Inorganic Matrices for Functional Nanoarchitectures Fabrication: The Simultaneous Effect of Moisture and Temperature in the Preparation of Metakaolin Based Geopolymers,” *Bull. Chem. Soc. Jpn.*, vol. 91, no. 7, pp. 1158–1167, Jul. 2018.

Curriculum Vitae

PERSONAL INFORMATION Promoda Kumar Behera M.Sc.,

17 Listopadu 584/2, 46015 Liberec, Czech Republic

+420 775112734

promodabehera@gmail.com

Sex: Male

Date of birth: 06/06/1989

Nationality: Indian

Vision & Skills

An enthusiastic, adaptive and fast-learning individual with a broad and acute interest in the *different characterizations (analytical, physical and mechanical properties) of particle/fiber-reinforced cementitious matrix composites*. I particularly enjoy collaborating with scientists of different disciplines to develop robust skills and solve new challenges.

Education

October 2014-Until now — Doctoral Student

Department of Material Engineering

Technical University of Liberec, Czech Republic
Topic: Influence Of High Temperatures On Properties Of Geopolymers Filled By Inorganic Fibrous Particles (Thesis submitted).

May 2013 — Master of Science

Department of Chemistry

National Institute of Technology, Rourkela, Odisha, India
Inorganic Chemistry, Physical chemistry, Organic chemistry etc.
Topic: Synthesis Of Chalcogen Bridged Metal Clusters Containing Acetylide Ligands

May 2010 — Bachelor of science

Department of Chemistry

Utkal university, Odisha, India
Physical, Inorganic, Organic and Industrial chemistry

Training

Jul. 2016-Sept. 2016

Internship for PhD

Three months internship under supervision **Prof. Holmer Savastano Junior** in University of Sao Paulo, Brazil.

- Mechanical, Physical and microstructural properties of cellulosic pulp fiber reinforced hybrid cementitious composites with addition of different volume fraction of basalt fiber
- Nanofibrillated cellulose for reinforcing cement matrix
- Optimized adherence for vegetable fiber in cement based materials
- Routes for dimensional and mechanical stability of non conventional fiber cements using vegetable fibers
- Accessing strain hardening behavior of fibrous composites

July. 2017-Sept. 2017

Internship for PhD

Three months internship under supervision **Prof. Thomas Gries** in ITA RWTH Aachen University, Germany.

Feasibility analysis of recycled carbon fibre reinforced concrete.

Dec. 2017-Jan. 2018

Internship for PhD

Two months internship under supervision **Prof. Kypros Pilakoutas** in Sheffield University, UK.

Mitigating plastic shrinkage cracking of concrete using recycled tyre polymer fibers

Two months Research experience in **Prof. John L Provis** Laboratory with PDRAs Dr Maria Criado.

Professional Development

Technical Skills And Competences

Image J, Origin Lab, Mini TAB, MATLAB, Q C Expert, MS Project, MS Windows, SAP, MS Office, MS Outlook

X-Ray Diffraction Analysis (XRD), Thermo gravimetric Analysis (TGA), Mass Spectrometry Analysis, Differential Scanning Calorimetry Analysis (DSC), Differential Thermal Analysis (DTA), Scanning Electron Microscopy, Energy Dispersive X-Ray Analysis (EDX), Fourier-transform infrared spectroscopy (FTIR) and Nuclear magnetic resonance spectroscopy (NMR).

Social Skills And Competences

Initiative, Communication, Innovation, Teamwork, Leadership, Problem solving, Task Orientation, Resource Management, Integrity, Perseverance

Personal Interests

Scientific Novelties, Playing Cricket, Exploring new places

Brief description of the current expertise, research and scientific activities

Doctoral Studies:	Full-time student at the Faculty of Textile Engineering Department of Material Engineering Specialization: Textile Technics and Material Engineering
List of Exams Passed:	1) Heat and Mass Transfer in Porous Media 2) Structure and Properties of Textile Fibers 3) Theoretical Textile Metrology 4) Mathematical Statistics and Data Analysis 5) Experimental Technique of Textile
State Doctoral Examination:	Passed
Research Projects:	Student grant scheme project

Record of the state doctoral exam

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ZÁPIS O VYKONÁNÍ STÁTNÍ DOKTORSKÉ ZKOUŠKY (SDZ)

Jméno a příjmení doktoranda:	**Promoda Kumar Behera**
Datum narození:	**6. 6. 1989**
Doktorský studijní program:	**Textilní inženýrství**
Studijní obor:	**Textile Technics and Materials Engineering**
Forma:	**prezenční**
Termín konání SDZ:	**18. 6. 2018**

prospěl **neprospěl**

doc. Ing. Lukáš Čapek, Ph.D.

Ing. Blanka Tomková, Ph.D.

prof. Ing. Zdeněk Bittnar, DrSc.

prof. Dr. Ing. Petr Lenfeld

doc. Ing. Michal Petrů, Ph.D.

doc. Ing. Antonín Potěšil, CSc.

Ing. Pavla Těšinová, Ph.D.

V Liberci dne 18. 6. 2018

O průběhu SDZ je veden protokol.

Recommendation of the supervisor

TECHNICKÁ UNIVERZITA V LIBERCI
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Supervisor's opinion on PhD thesis of Mr. Promoda Kumar Behera, M.Sc.

Thesis title: Influence Of High Temperatures on Properties of Geopolymers Filled by Inorganic Fibrous Particles

Mr. Promoda Kumar Behera, M.Sc., has worked for his PhD thesis under my supervision since 2014. The PhD thesis titled "Influence of High Temperatures on Properties of Geopolymers Filled by Inorganic Fibrous Particles" is quite comprehensive and fulfills the objectives outlined in his thesis. The thesis systematically investigated the effects of incorporation of inexpensive inorganic microfibers (basalt microfibrils and carbon microfibers) on the structure and thermal evolution of geopolymers synthesized from metakaoline. He developed expertise in preparation of particles by ball milling, preparation of geopolymer composites, characterization of their microstructure by SEM, EDS, Image analysis, XRD, TGA and testing of mechanical properties by measurement of compression strength, hardness, density, etc. The candidate has done all his work quite systematically, on required scientific level, with specific objectives. Discussion of the results is comprehensive and logical with citations of previous work where necessary. The quality of figures and tables is good and understandable. The language level of the thesis is good and meets the PhD standard. His publication activities are in excellent level. He has published 6 papers in international journals with impact factor. A few more are under review and expected to be published soon. He has presented more than 5 papers individually or jointly at international conferences, 2 chapters are published in reputed books. These are strong indicators for his thesis as a comprehensive work of independent research.

I therefore recommend the thesis for defense.

prof. Ing. Jiří Militký, CSc.

Supervisor

Opponent's reviews

TECHNICAL UNIVERSITY OF LIBEREC
Faculty of Textile Engineering

Assessment of PhD Thesis

Aspirant:	**Promoda Kumar Behera, M.Sc.**
Thesis title:	**Influence of High Temperatures on Properties of Geopolymers Filled by Inorganic Fibrous Particles**
Specialization:	**Textile Technics and Materials Engineering**
Supervisor:	**prof. Ing. Jiří Militký, CSc.**
Reviewer:	**doc. Ing. Antonín Potěšil, CSc.**

Topicality of the thesis

Comment:

The topic of the presented dissertation is undoubtedly up-to-date and it respects current innovative trends towards the development, manufacturing and use of alternative environmental materials which could be applied in industrial applications. The goal is to decrease the devastation of non-renewable natural resources.

excellent[1]	above standard	X	standard	substandard	weak

[1] Mark selected with a cross

Meet the objectives of the thesis

Comment:

The present work clearly describes and clarifies approaches to the preparation and testing of metakaolin-based geopolymers filled with basalt and recycled carbon particles.

The author is aware of the limits and limitations of their industrial use.

After studying the work as a whole, it can be stated that the defined goals of the work and PhD students intentions were fulfilled.

excellent	above standard	X	standard	X	substandard	weak

Methods and solutions

Comment:

Standard test methods, procedures and measuring devices (SEM, EDS, Image analysis, XRD, TGA, temperature and mechanical test, etc.) were used to characterize the properties of students created geopolymer structures.

excellent	above standard	standard	X	substandard	weak

Results of the Thesis - specific benefits of the student
Comments: The main contribution of this work is the comparison of selected physical and especially mechanical properties of several geopolymers filled with basalt (VEBA Industries) and recycled carbon particles (Easy composites, UK). Valuable information for the use of made geopolymers is especially data about material failure of geopolymer structures - destruction in compression. However, it is worth noting that the work is devoid of the deeper theoretical mechanics foundations of composite materials, which geopolymers undoubtedly are. Reported results of experimental findings do not always contain otherwise usual statistical processing.

excellent	above standard		standard		substandard		weak
		X					

Significance for practice and for the development of the scientific branch
Comments: The work presented is a good starting point for further research and development activities in the field of use of these geopolymers types in various industrial applications. That being said corresponds with student current publishing activities. I recommend the follow-up work be oriented deeper into the theoretical areas of geocomposite materials physical properties both regard to following processing technologies in the production and their industrial applications in a specific sense.

excellent	above standard		standard		substandard		weak
		X					

Formal layout of the Thesis and its language level
Comments: The work has a logical division, the text is understandable, the Czech version of the abstract text has minor editing errors, but does not spoil the overall impression of the work.

excellent	above standard	standard		substandard		weak
			X			

Comments and questions
1. What methods were used to determine the volume fractions of particulate fillers and matrix (metakaolin) in geopolymer samples? Please state the relationship between the weight and the volume part of the filler and geopolymers matrix structure. 2. In terms of continuum mechanics, geopolymer materials should generally be considered as composite structures exhibiting anisotropic properties, so-called geocomposites. What approaches are used to describe the relationship between stress and strain, which makes it possible to use modern simulation tools (CAD)?

Final evaluation of the Thesis
Based on the preview above I recommend the thesis submitted for defence in front of the scientific committee for the defence of the doctoral thesis.

I recommend after a successful defence of the dissertation grant Ph.D.[2]	*yes*	~~*no*~~

[2] Delete where applicable

Place and Date: In Liberec 20.12.2019

Signature: Antonín Potěšil

ČESKÉ VYSOKÉ UČENÍ TECHNICKÉ V PRAZE
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Posudek disertační práce

Uchazeč Promoda Kumar Behera

Název disertační práce Influence of High Temperatures on Properties of Geopolymers Filled by Inorganic Fibrous Particles

Studijní obor Textile Engineering

Školitel Prof. Ing. Jiří Militký, CSc.

Oponent Prof. Ing. Michal Šejnoha, Ph.D., DSc.

e-mail sejnom@fsv.cvut.cz

Aktuálnost tématu disertační práce

komentář: Scientific relevance of the submitted work

The present thesis is focused on the evaluation of the effect of high temperature on metakaolin based geopolymers modified by inorganic fibrous particles. Given its potential application as a replacement of OPC based mortar thus reducing the negative environmental impact by both the reduction of CO2 emission and exploitation of waste material, the alcali activated cementitious materials have enjoyed a considerable attention in the last decades. However, the use of these binders in large scale engineering applications is still an open question and much research is still needed. The presented study is another promising fragment in this endeavor and the achieved results certainly deserve attention.

☐ vynikající	☒ nadprůměrný	☐ průměrný	☐ podprůměrný	☐ slabý

Splnění cílů disertační práce

komentář: Goals of the work and their achievements

The research objectives are stated in Chapter 2. Concentrating on metakaolin as one particular source of aluminosilicate for geopolymer preparation I find reasonable, since it made the thesis compact and allowed for achieving all the goals stated. This is evident from Chapter 5 summarizing the principal outcomes of a very broad and diverse experimental program. Their choice is also supported by a detailed state of the art section and literature survey presented in Chapter 3, which shows a good orientation of the candidate in this field.

☐ vynikající	☒ nadprůměrný	☐ průměrný	☐ podprůměrný	☐ slabý

Metody a postupy řešení

komentář: Treatment of the topic - methodical and conceptual approach

The methodical and conceptual approach is described in Chapter 4 outlining the experimental program carried out in the course of this thesis. While the program is rather broad, its theoretical description is quite brief particularly for a reader not familiar with the subject. In most cases, the number of specimens is either not mentioned or the selection of a particular number is not explained. For example, from Section 4.10 it is not clear, whether the experiment was run in a stress or displacement controlled regime. One may only guess from the presented results that

the latter approach was adopted. When discussing the results the author quite often refers to the available literature suggesting similar observations. But no direct comparison is provided. So a non-experienced reader may just take these conclusions for granted. For example, the discussion on thermal stresses is rather difficult to accept if the mismatch in coefficients of thermal expansion of geopolymer and fibrous particles is not provided. This I find as the principal thesis shortcoming, which slightly pollutes otherwise interesting research activities.

☐ vynikající	☐ nadprůměrný	☒ průměrný	☐ podprůměrný	☐ slabý

Výsledky disertace - konkrétní přínosy disertanta

komentář: Thesis results - author's specific contribution

The principal outcomes are summarized in Chapter 5. Regardless of the above comments the results show a positive effect of fibrous particles over the neat samples with carbon particles appearing slightly more advantageous in comparison to basalt particles.

☐ vynikající	☒ nadprůměrný	☐ průměrný	☐ podprůměrný	☐ slabý

Význam pro praxi a pro rozvoj vědního oboru

komentář: Extent of new knowledge and contribution to the practice

The thesis certainly shed a light on a number of specific issues concerning thermal behavior of geopolymer based composites. But I am not an expert in this field so I suggest the author to give, during the thesis defense, his own opinion on a potential applicability particularly in conjunction with large scale engineering applications such as prefabricated concrete elements.

☐ vynikající	☐ nadprůměrný	☐ průměrný	☐ podprůměrný	☐ slabý

Formální úprava disertační práce a její jazyková úroveň

komentář: Organization of the work and overall comprehensiveness

The thesis are written in good English with only few grammatical errors. If leaving out too brief description of individual experimental measurements, the thesis are easy to follow and the results are clearly explained. However, in my opinion it might be beneficial to present some of the results of both composites within the same tables or graphs. Also, some of the general conclusions are not always fully supported by the presented results, e.g. the mentioned increase of hardness of CMF based composites with increasing amount of particles over of the whole range of temperature, see Table 8. The results for 800 0C of BMF composites in Table 7 are missing.

☐ vynikající	☒ nadprůměrný	☐ průměrný	☐ podprůměrný	☐ slabý

Připomínky

Comments:
Apart from comments raised already in the review sections "Treatment of the topic" and "Thesis results" the following questions might be addressed in more details:

1. Please explain what the difference between Wi and Wa in Eq. (1) is.
2. Please check Eq. (2). I suppose the temperature should be replaced with BMF(CMF)%.
3. Table 7: Why there is such a huge drop in hardness for G samples from 200 to 400 C?

4. Table 8: Please explain the increase in hardness for 15% CMF composite from 200 to 400 C. Perhaps this is an error.
5. Could you please plot the stress strain curves for both composites on the common graph to support the last sentence on page 56 and the first sentence on page 57?
6. Please explain the drop in weight for BMF composites in Fig. 33(a) not seen for CMF composites.
7. Please provide the coefficients of thermal expansion for individual phases in BMF and CMF based composites. I expect some mismatch which might generate excessive local stresses. In this regard, the last sentence on page 61 is not clear to me. Please explain.

Závěrečné zhodnocení disertace

Final statement:

Based on the submitted review, consisting of an assessment of the scientific relevance, fulfillment of the goals of the work, the quality of treatment of the topic and the extent of new knowledge, it is concluded that this work meets high quality standards.

As it complies with the requirements for a Ph.D. work, I recommend the thesis for further defense and if successful to appoint Mr. P.K. Behera the title

doctor (Ph.D.)

Doporučuji po úspěšné obhajobě disertační práce udělení titulu Ph.D.	***ano*** ☒	***ne*** ☐

Datum: 17.12.2019

Podpis oponenta: .....