

Tensile and flexural properties of basalt fibers/nano-alumina powder-reinforced multi-scale composites

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Abstract

Basalt fiber-reinforced epoxy (BFRE) composites without nano-alumina (NA) (Al_2O_3) and the corresponding multi-scale-nano-composites multi-scale composites containing NA (0.5, 1, 3 and 5 wt.%) were fabricated. Tensile and flexural tests were conducted to study the effect of NA addition on the composites. Surface modification of the NA was carried out using 3-(trimethoxysilyl)propylamine (as a silane coupling agent) to optimize the interaction of NA with epoxy. Subsequently, an infrared spectroscopy test was performed to confirm this surface modification had generated 3- aminopropyl functional groups on the NA surface. Different weight percentages of surface-modified NA were dispersed in the epoxy resin by mechanical and ultrasonic mixing methods. The resulting mixtures were then used as the matrices for multi-scale BFRE composites. Scanning electron microscopy (SEM) was used to investigate the fracture morphology of the composites after tensile and flexural tests. The greatest tensile and flexural property improvements compared to the control sample (BFRE composites without nano-materials) were obtained by adding 1 wt.% of NA. Upon the incorporation of 1 % NA into the BFRE, there was an improvement in tensile stiffness, increasing from 3.17 to 3.67 GPa. Additionally, the flexural modulus experienced enhancement, rising from 180 to 208 GPa. Improvements were achieved in the tensile and flexural moduli as well as in the tensile and flexural strengths. Microscopic studies also showed the interfacial adhesive enhancements between basalt fibers and the epoxy matrices due to the NA addition.

Keywords: Multi-scale-nano-composites, Basalt fibers, Nano-alumina, Tensile/bending, 3-(trimethoxysilyl)propylamine

1. Introduction

The composite materials use is rapidly growing in various applications thanks to their superior specific tensile stiffness and strength [1], excellent fatigue and impact [2,3], creep [4] and corrosion resistance, leading to a better tailorability compared with conventional materials [5,6]. Nonetheless, global warming is a key environmental concern which is caused by synthetic materials use. For instance, global warming causes glaciers to melt. Recently, 28 previously-undiscovered viruses have been unearthed which were trapped in glacial ice from 15,000 years ago [7]. In fact, there are deadly diseases concealed in ice, ready to wake up and challenge modern life, each may be as harmful as Coronavirus (Covid-19) or even worse [8,9]. Hence, introducing the natural fiber reinforcements has been inspired by prevalent synthetic fibers such as carbon, glass, and aramid [10–13]. As an example, basalt fibers which are made from volcanic igneous rock have shown promising applications compared with glass and carbon fibers. Basalt fibers have greater extensional stiffness and strength, higher heat resistance [14,15] and noticeably lower human being skin irritation than those for glass fibers [16,17]. Also, their impact tolerance and chemical resistance are superior to those of carbon fibers while enjoying lower cost. Despite most synthetic fibers, basalt fibers are non-toxic at elevated temperatures which make them excellent for cement and thermal barrier structures [18]. The basalt fiber market is estimated to reach ~\$392 million by 2025 as a well-established item in the composite industry.

Eslami-Farsani et al. [19] and Daghigh [20] conducted a comparative study on unidirectional basalt fiber-reinforced epoxy (BFRE) composites and unidirectional glass fibers reinforced epoxy (GFRE) composites for their creep behavior at elevated temperatures. Also, the effect of the micro-glass powder (MGP) addition into the epoxy resin on BFRE's creep behavior was studied. Khalili et al. [21] and Daghigh [20] investigated the tensile and flexural

behaviors of the same BFRE composites, MGP-modified BFRE composites, and GFRE composites considered in Ref. [19] and Ref. [20]. The MGP presence enhanced tensile/flexural moduli and flexural strength though the ultimate tensile strength remained almost unchanged. Khalili et al. [21] and Daghigh [20] also developed novel basalt fibers metal laminate (BFML) composites (comprised of BFRE/aluminum, BFRE/steel and BFRE/aluminum/steel layers) which showed greater toughness compared with analogues aluminum and BFRE specimens. Eslami-Farsani et al. [22] and Daghigh [20] studied BFRE and MGP-modified BFRE composites subjected to flatwise and edgewise Charpy impacts at room and elevated ($T = 75$ °C) temperatures. The same Charpy impact tests were conducted on BFML and GFRE specimens at room temperature. The MGP presence improved the energy absorption capability of BFRE composites; this capability is greater at elevated temperatures. Daghigh et al. [23] and Daghigh [20] studied the creep behavior of BFML composites at elevated temperatures. BFRE layers inclusion enhanced the creep resistance of aluminum-based BFML composites. Lamea et al. [24] performed axial compression buckling tests on open-hole BFRE and plain BFRE composite specimens using experimental and ABAQUS finite element methods. Also, plain GFRE composite specimens were studied for their buckling response and compared with that of BFRE ones. BFRE was shown to be more resistant than GFRE composite under the buckling loads. In general, BFRE composites showed superior properties compared with GFRE composites under tensile, flexural, impact, axial creep, and buckling loads [19,21–24]. Seghini et al. [25] studied the interfacial adhesion in BFRE composites. They conducted a comparative study on the effect of plasma-polymerized basalt fibers with a tetravinylsilane film on the mechanical and morphological properties of the composite. Atomic force microscopy was used to measure the surface roughness of untreated and treated basalt fibers.

The incorporation of nano-particles into fiber composites presents a range of advantages and disadvantages that influence their mechanical properties [26,27,28,29]. On the positive side, the addition of nano-sized particles can lead to improvements in tensile strength, modulus of elasticity, and impact resistance, contributing to an enhanced overall mechanical performance. This enhancement is particularly beneficial in applications where a high strength-to-weight ratio is crucial, such as in aerospace or automotive components. Furthermore, nano-particles can improve toughness, durability, and even thermal and electrical conductivity, allowing for tailored material properties to meet specific application requirements [30–32]. However, challenges arise in achieving a uniform dispersion of nano-particles, potential processing difficulties, and increased material costs [30–32]. Issues related to health and environmental concerns, along with a lack of standardized testing methods, also warrant careful consideration. Despite the promising advancements, the successful integration of nano-particles into fiber composites requires a balanced evaluation of the associated benefits and drawbacks.

Although the composite industry's capability for producing various composites is trusted from the market, difficulties in nano-composite fabrication have led to more necessity for research in nano-composites [26,27,33]. Generally, nano-particles are used to reinforce composites for improving elastic moduli [34,30] and thermal resistance [31], and impact improvement [30,32]. The viscosity of liquid resin increases substantially when nano-particles, which enjoy enormous specific surface areas, are added [30]. Nano-fillers, including halloysite nano-clay (HNC), montmorillonite clay (MC), nano-alumina (Al_2O_3) (NA), silica (SiO_2), and silicon carbide (SiC), have found extensive applications in enhancing the mechanical and thermal properties of composites, each leveraging its unique characteristics [28,29,35,36]. For example, due to the NA's incompatibility with epoxy [10], it is imperative to modify the NA's

surface to enhance its compatibility. Nevertheless, there are seldom studies on mechanical behaviors of NA-reinforced BFRE composites [37,38].

In addressing the compatibility challenges observed with NA and BFRE composites, this study focuses on exploring the impact of incorporating surface-modified NA powders (SMNAPs) on the tensile and flexural properties of BFRE composites.

2. Experimental procedure

2.1. Materials

2.1.1 Epoxy resin

KER 828 epoxy resin with polyamine hardener (Charm, South Korea) was used as the matrix. KER 828 epoxy resin has medium viscosity with no diluent used. It has high wetting and filling properties which is suitable for high mechanical loads and chemical resistance. Table 1 shows some properties of the KER 828 epoxy resin. The resin-to- hardener weight ratio is 100:10.

Table 1. Epoxy resin.

Mateiral status	Color	Acidic or base property	Density in room temprature	Viscosity in room temprature
Liquid	Pale yellow	7	1160 Kg/m ³	12-14 Pa.s

2.1.2. Basalt fibers

Fabric Basalt fibers (Basaltex, Belgium) were used to reinforce the matrix. The following technical properties were extracted from the datasheet:

Name: 1500/35/7101; Type of fiber texture: Satin; Thickness: 0.3 mm; Surface density: 3 g/cm².

2.1.3. Nano-alumina powders

The nano-alumina powders (NAPs) used in fabricating the multi- scale composites (Neutrino, Iran) were gamma-Al₂O₃. The crystalline gamma-NA was used rather than alpha-NA. The reason is a higher specific surface of the alpha sample. Alpha-NA has higher thermal properties and heat resistance; however, gamma-NA has a higher specific surface area and better interaction with epoxy resin leading to an increase in the composites' tensile and flexural properties. Properties of the NAPs are tabulated in Table 2.

Table 2. Properties of nano-alumina powers extracted from the company datasheet.

Chemical formula	Color	form	Crystal form	Average size (nm)	Density (gg/cc)
gamma-Al ₂ O ₃	white	spherical	Gamma	10-20	3.65

2.1.4. Silane coupling agent

3-(Trimethoxysilyl)-propylamine (Merck, Germany, 818,550) was used as a silane coupling agent for nano-particle surface modification. The epoxy resin's epoxide groups were utilized to engage in a chemical reaction with the predominant 3-aminopropyl functional groups of this organic silane, facilitating enhanced adhesion of alumina to the matrix. Furthermore, upon cooling the NA, the epoxy resin exhibited improved wetting characteristics on its surface. This coupling agent has the following properties:

The molecular formula: C₆H₁₇NO₃Si; Molecular weight: 179.29 g/mol; Acidic media (g/l, H₂O): 9<; Flash point: 90 °C.

2.2. Nano-alumina surface modification

Nano-alumina (NA) (1.0 g) was poured into 100 ml of the solution containing distilled water (5 ml) and ethanol (95 ml). Next, it was dispersed by sonication (ultrasonic device-

FAPAN, Iran) at 80 W for 15 min. Subsequently, the coupling agent, (3-(trimethoxysilyl)-propylamine), NA was added to the mixture and this new mixture was refluxed for 8 h at 70 °C while stirring at 100 rpm by a magnetic stirrer. The solution pH was held at 3–4 by adding 37 % hydrochloric acid. After refluxing, the resultant mixture was centrifuged twice at 4000 rpm for 15 min to remove the SMNAPs. Prior to each centrifugation, the mixture was rinsed to remove unreacted silane compounds and, then dried in the oven (60 °C, 1 atm) for 12 h.

The addition of nano-sized powders to composites can significantly impact their mechanical behavior. The mechanical properties of these composites can be influenced by various factors, and one key parameter is the mixing and stirring time and rotation velocity during the fabrication process. In particular, this parameter can influence the dispersion of nano-particles, interfacial bonding, uniformity of structure, void elimination, and consistency in performance.

2.3. Specimen preparation

Epoxy-nano-alumina mixtures were prepared. Different ratios (0, 0.5, 1, 3 and 5 wt.%) of SMNAPs were added to the epoxy resin (without hardener). These mixtures were stirred using a mechanical mixer (SD11- D, Finitech, South Korea) for 15 min at 2000 rpm to distribute the SMNAPs in the epoxy resin. To reduce existing agglomeration and improve SMNAPs dispersion, the mixture was subjected to sonication at 24 kHz and 120 W for 60 min. After cooling to room temperature, the mixture was stirred again for 15 min at 2000 rpm. The hardener was then added to the mixtures at a ratio of 10:100 and the mixtures were stirred to generate the required materials for composite fabrication.

The addition of nano-sized powders to composites can significantly impact their mechanical behavior [26,30,32]. The mechanical properties of these composites can be influenced by various factors, and one key parameter is the mixing and stirring *time and*

rotational velocity during the fabrication process. We adopted a predetermined and fixed mixing and stirring time and rotation velocity, guided by considerations related to laboratory equipment capabilities and benchmark values reported in relevant literature. BFRE 4-layer composites with 50 wt.% of basalt fibers were fabricated (dimensions $15 \times 30 \text{ cm}^2$) using each mixture as the matrix and the hand lay-up technique. BFRE composites were cured at room temperature for 7 days and then cut to ASTM standard dimensions required for tensile and flexural tests. Tensile specimens were tabbed with GFRE composites (dimensions of $50 \times 25 \text{ mm}^2$) on both sides of BFRE composites to prevent them from slipping and reduce stress concentration. The nominal dimensions of ASTM standard specimen are listed as:

- Tensile specimens: length, 250 mm; width, 25 mm.
- Flexural specimens: length, 150 mm; width, 25 mm.

2.4. Mechanical tests, infrared spectroscopy tests, and testing apparatus

2.4.1. Tensile and 3-point flexural tests

The tensile and flexural tests were performed (Hounsfield 25KS) according to ASTM D790 and ASTM D3039, respectively. The support span for the flexural test was set to be 112.5 mm. Tensile and 3-point flexural tests were performed with crosshead speeds of 2 mm/min to reduce the load rate effects. Figs. 1a and 1b show the BFRE composite specimens during tensile and 3-point flexural tests, respectively.

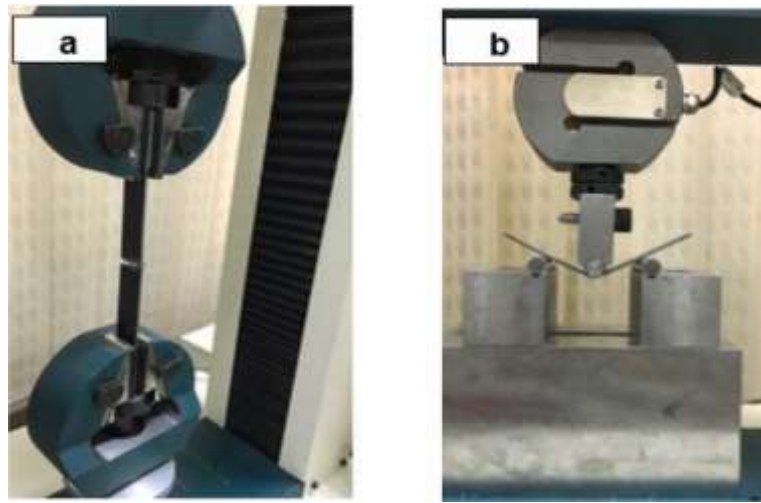


Fig. 1. BFRE composite specimens during (a) tensile and (b) 3-point flexural tests.

2.4.2. FT-IR and microscopy analysis

Fourier-transform infrared spectroscopy (FTIR) is a method employed to capture the infrared absorption or emission spectrum of materials. Unlike dispersive spectrometers, FTIR spectrometers can gather detailed spectral data across a broad range of wavelengths simultaneously, offering a substantial advantage in terms of resolution and coverage. FT-IR (PerkinElmer) in the wave range of $4000\text{--}400\text{ cm}^{-1}$ and sensitivity of 4 cm^{-1} was used to investigate the reactions between NA and silane agent to ensure the effect of silane factor on NA's surface and NA's surface treatment. Also, after performing the tensile and 3- point flexural tests, field emission scanning electron microscopy (FESEM) (Model Mira3-XMU) was used to study the fracture morphology. The fracture surfaces were sputter-coated with a thin gold layer to enhance the conductivity and image quality.

3. Results and discussion

3.1. FT-IR analysis

The results of FT-IR spectroscopy for surface-unmodified nano- alumina (SUNAPs) and SMNAPs are given in Fig. 2. By comparing the results of SUNAPs and SMNAPs spectroscopy,

it was observed that the absorption peak in the 3748 cm^{-1} wave number indicates the presence of -OH band on NA's surface. The 2936 cm^{-1} wave number also indicates the tensile vibration of -CH₂ on the NA's surface. The 1157 cm^{-1} wave number represents the extensional vibration of C-N on the NA's surface which confirms the successful interaction of the silane coupling agent with NA's surface.

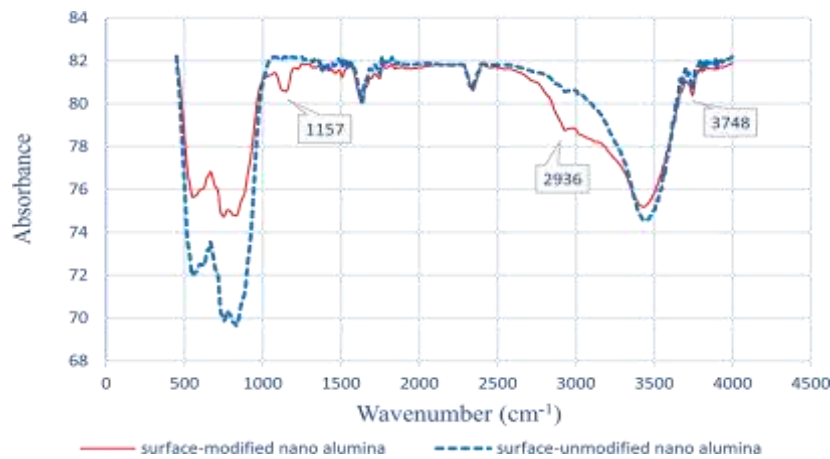


Fig. 2. FTIR spectra of surface-unmodified nano-alumina and surface-modified nano-alumina.

3.2. Tensile and flexural test analysis

Tensile stress versus strain diagrams show that the behavior of the composites is reasonably elastic. The tensile properties of BFRE specimens with no SMNAP are compared with those BFRE containing 0.5, 1, 3 and 5 wt.%. Addition of 0.5 wt.% SMNAP to the BFRE composites significantly increased the tensile modulus by 25 % and slightly enhanced tensile strength by 9 %. The maximum tensile strength was obtained by adding 1 wt.% SMNAP to BFRE which led to a tensile strength increase of 14 %. The addition of further NA (3 or 5 wt.%) led to a decrease in tensile properties compared to 1 wt.% SMNAP due to SMNAP particle agglomeration [26,30–32], e.g. 5 wt.% NA addition to BFRE decreased the tensile fracture strain and tensile strength by 12 % and 10 %, respectively compared with those for plain BFRE; tensile modulus remained unchanged (Fig. 3 and Table 3).

Owing to the significantly elevated modulus of the reinforcing SMNAP in comparison to that of the epoxy resin, the anticipated rise in both tensile and flexural moduli within the resultant nano-composite is predictable. The reduction in tensile and flexural moduli in exchange for further SMNAP addition (3 and 5 wt.%) is probably due to the SMNAP agglomerations in the matrix, which has reduced SMNAP effectiveness in material stiffness.

Generally, NA addition to polymer composites improves thermal and mechanical properties in contrast with the bulk polymer [31]. The current study shows that SMNAP addition into BFRE composites has a substantial effect on the tensile and flexural properties of BFRE composites. Possessing high surface areas, SMNAP can limit motions of polymer segmental at their solid surfaces leading to increase the entanglement between fibers and the matrix [1,31,37]. Moreover, SMNAP addition to the matrix reduces the stress concentration of the basalt fibers subjected to loads leading to rupture stress enhancement. Nonetheless, further addition of SMNAP to the matrix has an adverse effect on tensile and flexural properties. It is likely due to nano-particle agglomerations which behave as a hole/defect provider creating stress concentration in a pre-damaged material [22,30,31,37].

Comparing the effect of SMNAP addition on tensile and flexural properties shows that the SMNAP addition to BFRE has a more positive effect on tensile modulus and strength to those of flexural ones. This is attributed to the different state of stress in tensile and flexural tests. The in-plane tensile load is applied on the entire specimen length, whereas only three points are subjected to flexural test loads [24]. It causes the flexural tests to provide compressive stress above the neutral axis and tensile stress under the neutral axis. Since the SMNAP addition has more positive effects on matrix reinforcement rather than fiber reinforcement, the flexural properties of BFRE get more benefits than the tensile properties of BFRE [37]. Abdi et al. [38] reported a similar study to investigate the effect of surface-modified CaCO_3 particle (SMCN)

addition on BFRE composites. SMCN addition enhanced flexural properties more than tensile properties. Also, Khalili et al. [21] studied the effect of micro-glass powder (MGP) addition on BFRE composites subjected to tensile and flexural loads. MGP addition could improve flexural strength and failure strain more than those for tensile. It shows both micro- and nano-scale powders have capabilities for improving the stiffness of the materials though agglomeration is more a matter of concern for nano-particles rather than micro-particles [19,22,30].

Addition of 1 wt.% SMNAP to BFRE enhanced the flexural modulus from 8.6 GPa to 10.9 GPa (15 % increase), and flexural strength from 180.38 to 208 MPa (19 % increase), respectively. Consistent with expectations, the incorporation of 5 wt.% SMNAP into the BFRE composite led to a reduction in both flexural modulus and strength by 9.5 % and 14 %, respectively (Figs. 3 and 4, Table 3).

Table 3. Summary of tensile and 3-point flexural properties for basalt fiber/nano-alumina powder-reinforced multi-scale composites.

Nano-alumina (%)	Tensile modulus (MPa)	Tensile strength (MPa)	Tensile failure strain (mm/mm)	Flexural modulus (GPa)	Flexural strength (MPa)	Flexural failure strain (mm/mm)
0	3.17	134.33	0.042	8.9	180.38	0.029
0.5	3.97	146.42	0.036	9.04	181	0.026
1	3.67	153.22	0.041	10.6	207.98	0.024
3	3.63	148.18	0.04	8.16	189.85	0.031
5	3.22	120.14	0.037	8.05	155.14	0.025

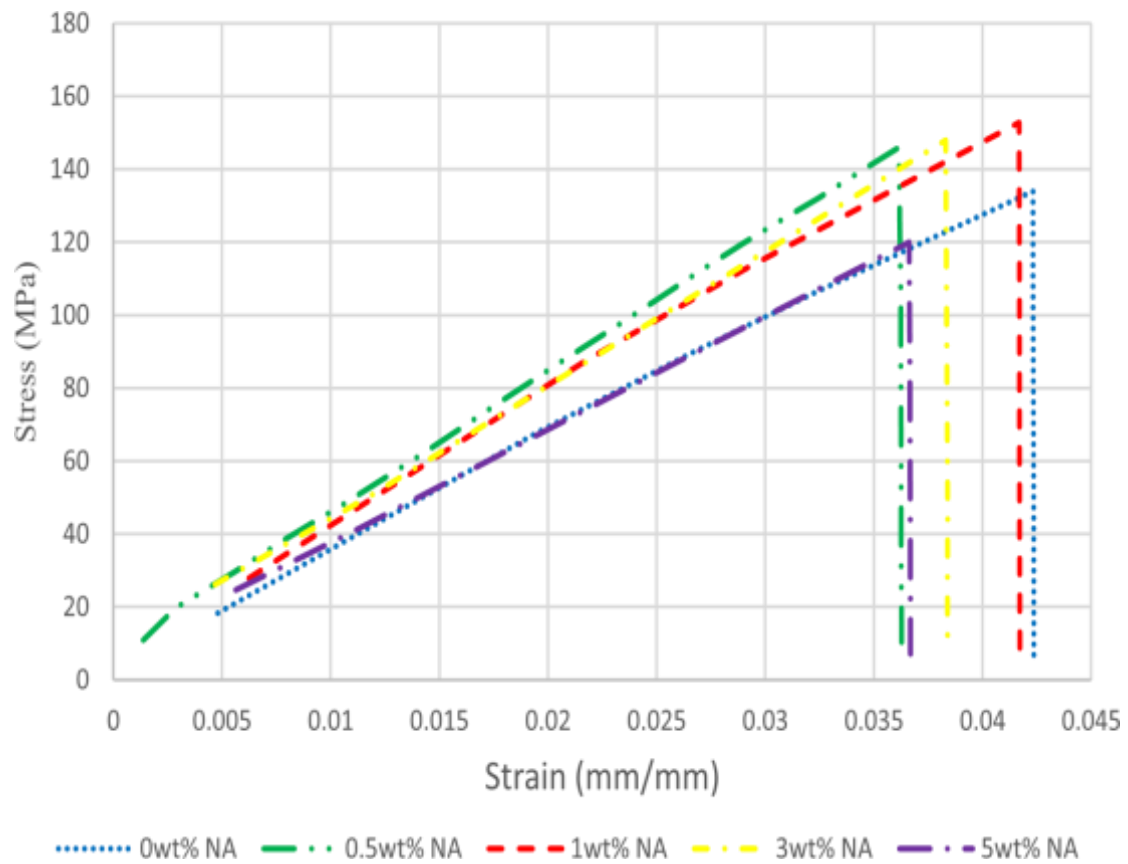


Fig. 3. Effect of SMNAP content on tensile behavior of BFRE composites containing 0, 0.5, 1, 3, and 5 wt.% of SMNAP.

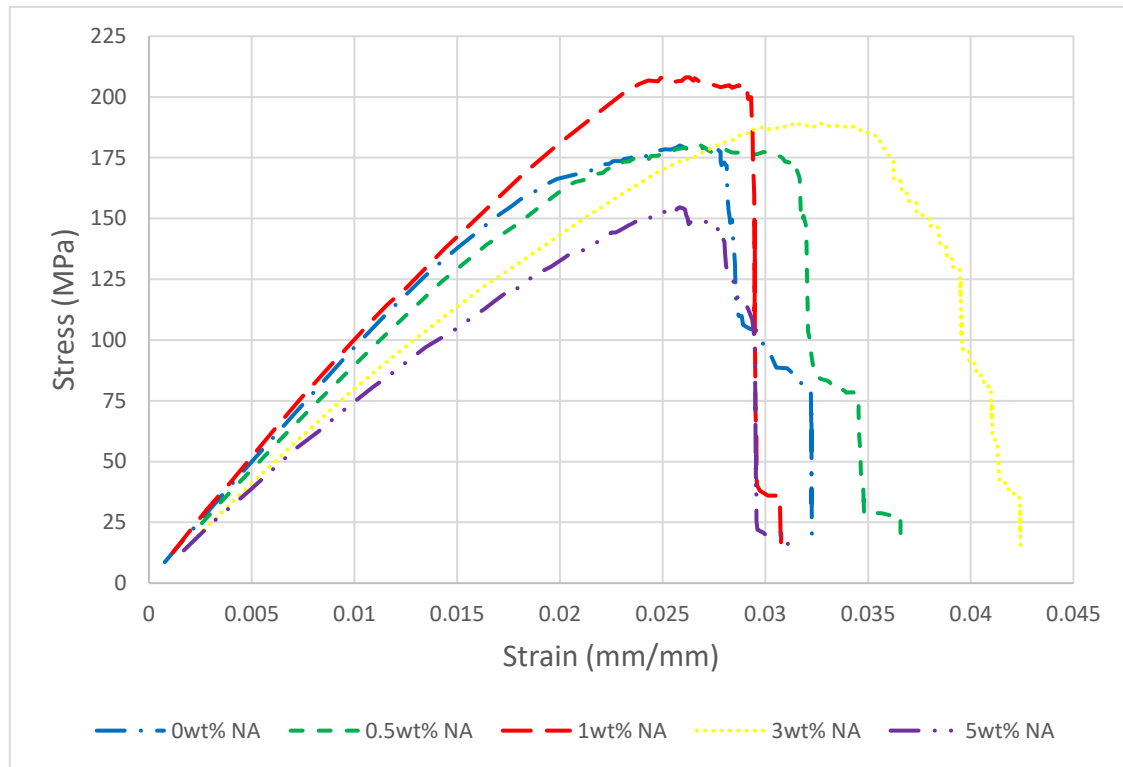


Fig. 4. Effect of SMNAP content on three-point flexural behavior of BFRE composites containing 0, 0.5, 1, 3, and 5 wt.% of SMNAP.

The strength of 148.18 MPa in the sample containing 3 wt.% of SMNAP reached 120.14 MPa which shows a decrease of 23.3 %. By adding 3 and 5 wt.% SMNAP, the tensile modulus was reduced compared to the 1 wt.% SMNAP sample. The tensile modulus of 3.67 GPa in the 1 wt.% SMNAP sample reached 3.63 GPa and 3.22 GPa for the 3 and 5 wt.% SMNAP samples, respectively.

Generally, nano-sized particles have a higher surface area compared to their larger counterparts. This increased surface area allows for better interaction and bonding between the nano-particles and the polymer matrix, resulting in improved load transfer between the matrix and the reinforcing fibers. It is critical for load-bearing applications as it helps in distributing applied loads more effectively by reducing the risk of localized stress concentrations that could lead to failure [26,30–32]. Moreover, the fiber-matrix interface plays a pivotal role in

determining the overall mechanical performance of composite materials. Nano-powders can enhance the adhesion between the fibers and the epoxy matrix. The high surface area of nano-particles provides more sites for interaction with the matrix, leading to improved wetting of the fibers by the resin. This improved wetting results in better adhesion, which, in turn, enhances the load transfer between the fibers and the matrix. Reducing the interfacial debonding is another benefit of nano-powders' incorporation. Nano-particles can act as bridging agents across the fiber-matrix interface that prevent crack initiation and propagation and improve the overall interfacial strength [26,30–32,39].

The flexural response of BFRE is generally anticipated to exhibit elasticity akin to its tensile behavior. However, variations in the flexural behavior are contingent upon several factors, including the characteristics inherent to BFRE stemming from basalt fiber attributes, epoxy resin properties, interlaminar/intralaminar characteristics, and the fiber/matrix interplay. Consequently, the flexural behavior may manifest some degrees of nonlinearity (See Fig. 4).

This nonlinearity can be attributed to the differential stresses experienced by the layers above and below the neutral surface during flexural loading. The upper layers undergo compression, while the layers beneath the neutral surface experience tension. As the loading process advances, damage initiates and propagates, causing the bottom layers to fail prematurely in comparison to those closer to the neutral surface [20, 40]. Consequently, the slope of the flexural diagram for BFRE diminishes, reflecting a reduction in bending rigidity.

This deviation from linear behavior is reminiscent of plastic behavior, yet it fundamentally diverges from the conventional definition of plasticity. The introduction of SMNAPs into the BFRE composite exacerbates the reduction in bending rigidity upon damage initiation and propagation. Comparable trends in the flexural behavior of BFRE composites have been documented in Refs. [20–22,40], where BFRE composites were fortified with MGPs.

3.4. Fracture morphology

FESEM was used to observe the fracture surfaces after the tensile and flexural testing. Failure in polymer composites is a result of damage accumulation including several energy absorption phenomena including crack initiation and or growth [1,22]. Several parameters (and their interactions) such as fiber volume fraction, fiber/matrix interfacial properties, uncertainty in fiber strengths, filler/powder properties, and matrix strengths control this damage formation and progression [1]. Figs. 5a and 5b show microscopic images of a BFRE tensile specimen with no SMNAP content. The fiber surfaces are smooth and clear.

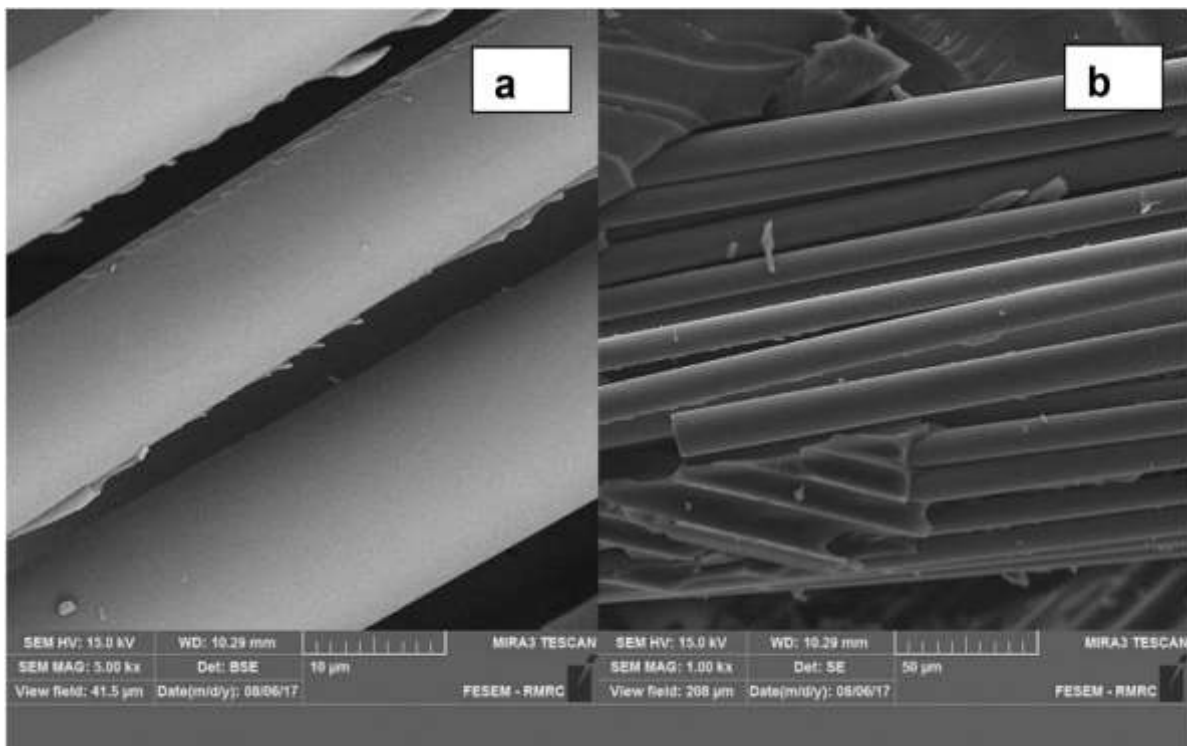


Fig. 5. BFRE composites with no SMNAP content after tensile tests; clean and smooth surface of basalt fibers, (a) Magnification of 5 kx, (b) Magnification of 1 kx.

Figs. 6a and 6b show a BFRE specimen after a tensile test with a magnification of 500 x and 200 x, respectively. The visible failure criterion is debonding or fiber pull-out which is

associated with weak adhesion between the fibers and the matrix. Fiber pull-out significantly absorbs energy during the loading process. Also, there are some surface cracks on the matrix which indicates damage progression [41].

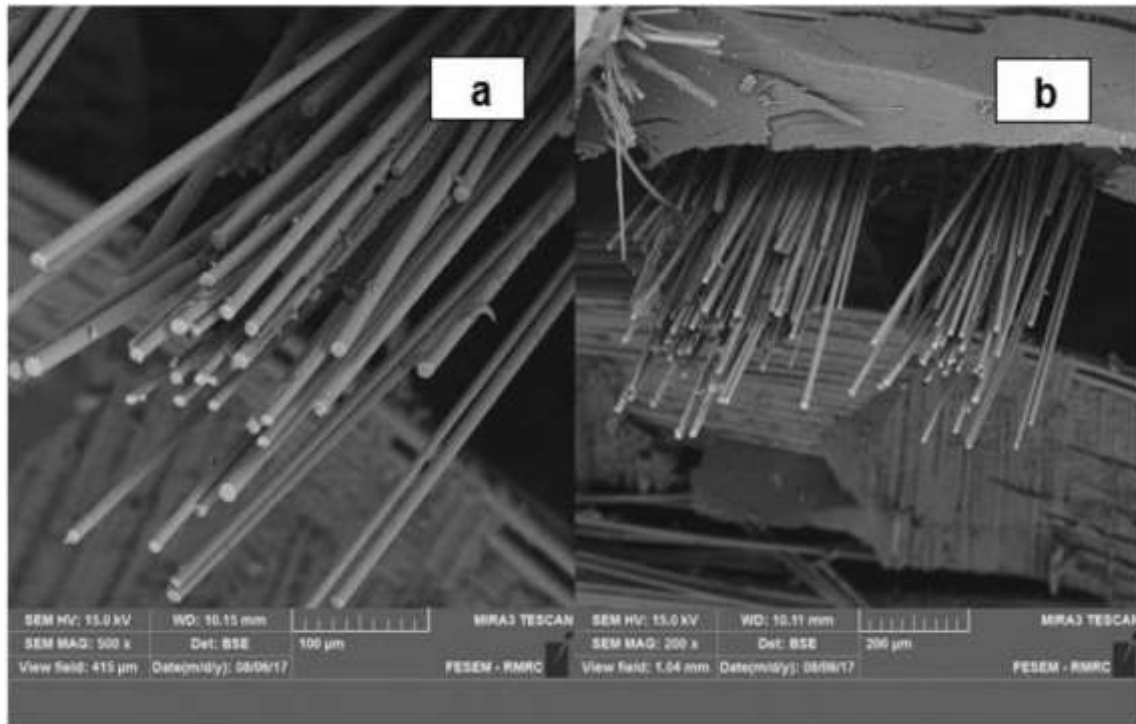


Fig. 6. BFRE composites with no SMNAP content after tensile tests; fiber pull-out as one of the main failure criteria, (a) Magnification of 500 x, (b) Magnification of 200 x.

Fig. 7 depicts the BFRE composites with various contents of SMNAP (0.5, 1, 3 and 5 wt.%). Comparing SEM images of fractured BFRE composites reveals the dominant failure mechanism attributed to matrix cracking in BFRE composites containing SMNAP. In contrast, BFRE composites without SMNAP primarily experience failure due to fiber debonding.

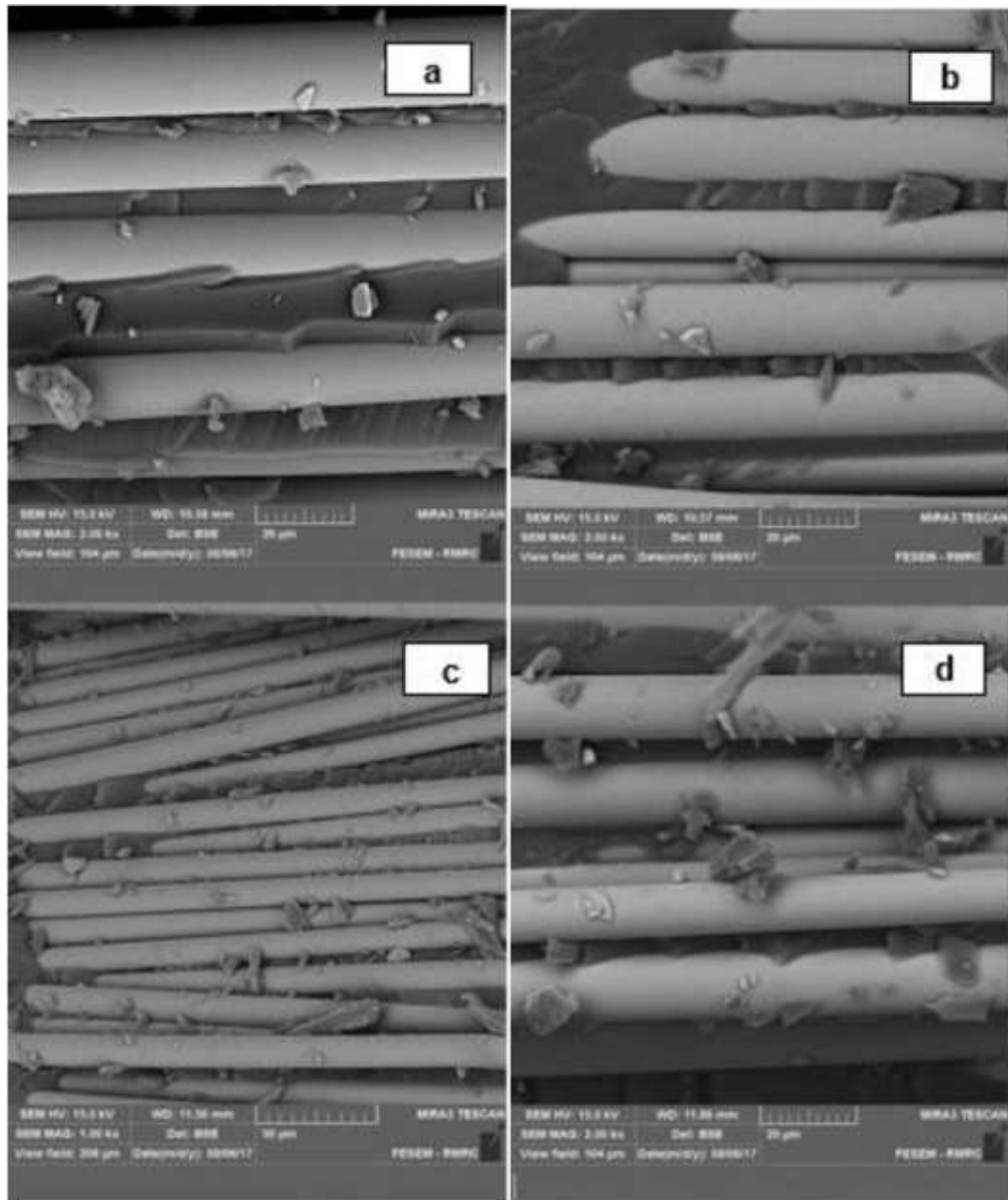


Fig. 7. Fiber pull-out as one of the main failure criteria in BFRE composites with various contents of SMNAP, (a) 0.5 wt.%, (b) 1 wt.%, (c) 3 wt.%, (d) 5 wt.%.

The presence of SMNAP on the basalt fiber in the BFRE composite containing 1 wt.% SMNAP is shown in Fig. 8. This picture shows a good adhesion between the SMNAP and basalt fiber due to the surface modification conducted on SMNAP particles. Surface modification of SMNAP particles causes SMNAP particles to contribute to energy dissipation during the loading procedure. Consequently, basalt fibers break at higher levels of energy and

stress. Fig. 9 illustrates the homogeneous distribution of SMNAPs upon the basalt fiber substrate. This homogenous distribution is pivotal for optimizing the interfacial interactions within the composite material.

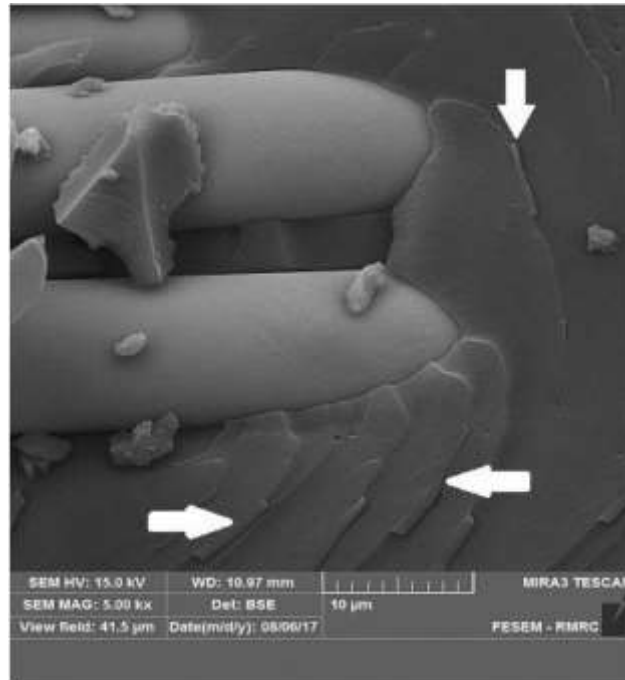


Fig. 8. BFRE composites with 1 wt.% SMNAP.

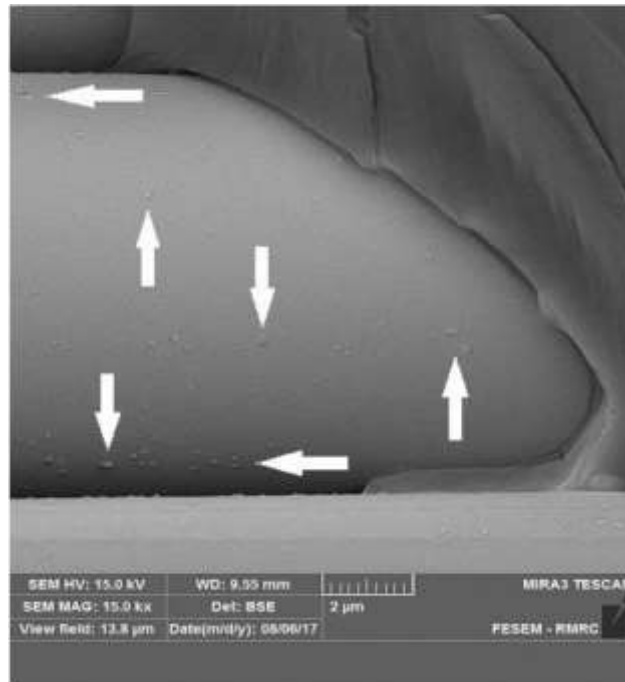


Fig. 9. BFRE composites with 1 wt.% SMNAP, illustrating the presence of SMNAP particles adhering to the fibers.

The formation of agglomerated SMNAP particles is shown in regions specified with white color arrows for BFRE composites with 3 wt.% and 5 wt.% SMNAP (Figs. 10a and 10b). These are agglomerated SMNAP particles in BFRE composites with 3 wt.% SMNAP and 5 wt.% SMNAP, respectively. Agglomerations act as stress concentration providers and facilitate the crack propagation leading to a major reduction in the strength of materials. To avoid agglomeration, the power and speed of the ultrasonic operation should be increased [41,42].

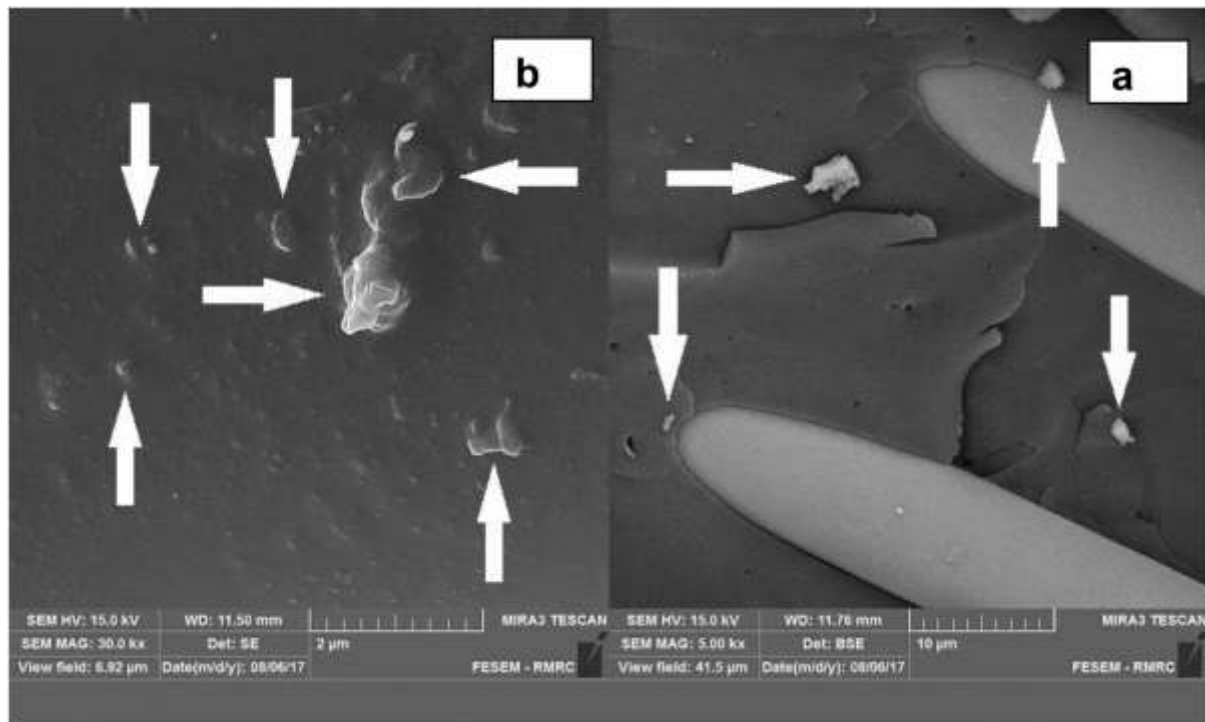


Fig. 10. Agglomerated SMNAP particles in BFRE composites with; (a) 3 wt.% SMNAP, b) 5 wt.% SMNAP.

4. Conclusion

The effect of nano-alumina (NA) (Al_2O_3) on the tensile and flexural behavior of basalt fiber-reinforced epoxy (BFRE) composites was investigated. The multi-scale BFRE composites containing surface- modified NA powders (SMNAPs) (0.5, 1, 3 and 5 wt.%) were fabricated and tested. The results were compared with BFRE composites without SMNAP. NA surface modification was conducted using 3-(trimethoxysilyl)propylamine (as a silane coupling agent) to enhance the NA interaction with epoxy. An infrared spectroscopy test was performed to confirm the generation of 3-aminopropoxyl functional groups on the NA's surface. The relative improvement in tensile and flexural properties of BFRE composites roughly increased with increasing surface- modified NA powders (SMNAPs) up to 3 wt.%; clearly, SMNAPs use resulted in better fiber/matrix interfacial adhesion and correspondingly greater stiffness and mechanical energy dissipation. Hence, SMNAP may be employed to improve

BFRE tensile and flexural properties for applications where both stiffness and energy dissipation are critical. Nevertheless, adding 5wt% SMNAP to BFRE composites exhibited a deleterious effect on mechanical properties by lowering tensile stiffness and strength as well as flexural stiffness and strength. This observation implies that the SMNAP agglomeration results in stress concentration, leading to reduced stiffness and diminished energy dissipation in BFRE composites. Lastly, scanning electron microscopy was employed to investigate the fracture morphologies of tensile and bending specimens. Failure mechanisms of multi-scale SMNAP-reinforced BFRE composites included matrix crazing/cracking, fiber pull-outs, fiber fracture, micro-void nucleation, and fiber tensile fracture. SMNAP addition, by the time it is *not* severely agglomerated, improves fiber matrix adhesion of BFRE composites. It makes sense since the covalent bonds are formed among SMNAP, basalt fiber, and matrix surfaces. In the future, this work may be developed to examine the basalt fiber/epoxy ratio that is required for optimized mechanical properties for a given fiber loading. Moreover, SMNAP/fiber surface area ratio, fiber/matrix ratios, NA modification method and other factors are important in the mechanical composite bulk properties.

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CRediT authorship contribution statement

R. Eslami Farsani: Writing – review & editing, Visualization, Supervision, Project administration, Methodology, Investigation, Conceptualization. **Vahid Daghigh:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal

analysis, Conceptualization. **Kasra Derakhshani:** Investigation, Formal analysis, Data curation, Methodology, Visualization.

Declaration of competing interest

The author has no relevant financial or non-financial interests to disclose regarding this research, authorship, or publication.

Data availability

Data will be made available on request.

References

- [1] V. Daghigh, T.E. Lacy, C. Pittman, H. Daghigh, Influence of maleated polypropylene coupling agent on mechanical and thermal behavior of latania fiber- reinforced PP/EPDM composites, *Polym. Compos.* 39 (2018) E1751–E1759.
- [2] R. Petrucci, E. Nisini, D. Puglia, F. Sarasini, M. Rallini, C. Santulli, G. Minak, J. M. Kenny, Tensile and fatigue characterization of textile cotton waste/ polypropylene laminates, *Compos. B* 81 (2015) 84–90.
- [3] F. Sarasini, J. Tirillo, L. Ferrante, C. Sergi, F. Sbardella, P. Russo, G. Simeoli, D. Mellier, A. Calzolari, Effect of temperature and fiber type on impact behavior of thermoplastic fiber metal laminates, *Compos. Struct.* 223 (2019) 110961.
- [4] J. Raghavan, M. Meshii, Creep of polymer composites, *Compos. Sci. Technol.* 57 (12) (1998) 1673–1688.
- [5] S.M.R. Khalili, R. Eslami Farsani, V. Daghigh, Aging influence on charpy impact behavior of basalt fiber reinforced epoxy composites, *Int. J. Adv. Manuf. Technol.* 6 (223) (2013) 81–85.
- [6] F. Dominici, D. Puglia, F. Luzi, F. Sarasini, M. Rallini, L. Torre, Novel class of cost effective and high performance composites based on terephthalate salts reinforced polyether ether ketone, *Polymers* 11 (12) (2019) 2097. . (Basel).
- [7] J. Leman, Jan 23, 2020 Welp, scientists found 28 new virus groups in a melting glacier <https://www.popularmechanics.com/science/health/a30643717/virusesfoundmelting-glacier/>.
- [8] <https://ontd-political.livejournal.com/11819113.html>.

- [9] “How climate change plunders the planet”,https://www.edf.org/climate/how-climatechangeplundersplanet?gclid=Cj0KCQjw3qzzBRDnARIsAECmrypajFkTw8zppAwnYArUNB9XrL0ROrzi5g3kzEXPEcX1Lie2Qs4UigAaAneYEALw_wcB.
- [10] D.A. Derusova, V.P. Vavilov, S. Sfarra, F. Sarasini, N.V. Druzhinin, Applying ultrasonic resonance vibrometry for the evaluation of impact damage in natural/ synthetic fibre reinforced composites, *Polym. Test.* 68 (2018) 70–76.
- [11] S. Behnia, V. Daghigh, K. Nikbin, A.B. Fereidoon, J. Ghorbani, The influence of stacking sequence and notch angle on Charpy impact behavior of hybrid composites, *Mech. Compos. Mater.* 52 (4) (2016) 489–496.
- [12] F. Sarasini, J. Tirillo, L. Lampani, E. Barbero, S. Sanchez-Saez, T. Valente, P. Gaudenzi, C. Scarponi, Impact behavior of sandwich structures made of flax/ epoxy face sheets and agglomerated cork, *J. Nat. Fibers* 17 (2) (2020) 168–188. [13] D.U. Bing, C.H.E.N. Liming, L.I.U. Houchang, H.E. Qinghao, Q.I.N. Weiming, L.
- [13] I. Weiguo, Resistance welding of glass fiber reinforced thermoplastic composite: experimental investigation and process parameter optimization, *Chin. J. Aeronaut.* 33 (12) (2020) 3469–3478.
- [14] L.C. Hao, W.D. Yu, Evaluation of thermal protective performance of basalt fibre nonwoven fabrics, *J. Therm. Anal. Calorim.* 100 (2) (2010) 551–555.
- [15] C. Suresh Kumar, V. Arumugam, H.N. Dhakal, R. John, Effect of temperature and hybridisation on the low velocity impact behavior of hemp-basalt/epoxy composites, *Compos. Struct.* 125 (2015) 407–416.
- [16] World health organization international agency for research on cancer (IARC), Monographs on the evaluation of carcinogenic risks to humans, man-made vitreous fibers, summary of data reported and evaluation 81 (2002) IARC2002ISBN-13978- 92-832-1281-2ISBN-13978-92-832-1581-3. <https://publications.iarc.fr/Book-And-Report-Series/Iarc-Monographs-On-The-Identification-Of-Carcinogenic-Hazards-To-Humans/Man-made-Vitreous-Fibres-2002>.
- [17] E. McConnell, O. Kamstrup, D.R. Musselman, T.W. Hesterberg, J. Chevalier, W. C. Miiller, P. Thevenaz, Chronic inhalation study of size-separated rock and slag wool insulation fibers in Fischer 344/N rats, *Inhal. Toxicol.* 6 (1994) 571–614. No.6.
- [18] A. Anandamurthy, V. Guna, M. Ilangoan, A review of fibrous reinforcements of concrete, *J Reinf. Plast. Compos.* 36 (7) (2017) 519–552.
- [19] R.E Farsani, S.M.R. Khalili, V. Daghigh, R. Fazaeli, Creep behavior of basalt and glass fiber reinforced epoxy composites, *J. Mech. Res. Appl.* 3 (1) (2011) 29–36.
- [20] V. Daghigh, “Mechanical behavior of basalt fiber-reinforced and basalt fiber metal laminate composites under tensile, bending, Charpy impact and creep loads”, master’s thesis in Mechanical Engineering, IAU, Tehran. 2010. | Request PDF (researchgate.net) https://www.researchgate.net/publication/335173233_Mechanical_behavior_of_basalt_fib

er-reinforced_and_basalt_fiber_metal_laminate_composites_under_tensile_bending_
Charpy_impact_and_creep_loads.

- [21] S.M.R. Khalili, V. Daghigh, R. Eslami Farsani, Mechanical behavior of basalt fiber-reinforced and basalt fiber metal laminate composites under tensile and bending loads, *J. Reinf. Plast. Compos.* 30 (2011) 647–659. No.8.
- [22] R.E Farsani, S.M.R. Khalili, V. Daghigh, Charpy impact response of basalt fiber reinforced epoxy and basalt fiber metal laminate composites: experimental study, *Int J Damage Mech* 23 (2014) 729–744. No.6.
- [23] V. Daghigh, S.M.R. Khalili, R.E Farsani, Creep behavior of basalt fiber-metal laminate composites, *Compos. B.* 91 (2016) 275–282.
- [24] M. Lamea, V. Daghigh, M. Soroush, K. Nikbin, The buckling behavior of vacuum-infused open-hole unidirectional basalt-fiber composites experimental and numerical investigations, *Mech. Compos. Mater.* 55 (2020) 761–774.
- [25] M.C. Seghini, F. Touchard, F. Sarasini, V. Cech, L. Chocinski-Arnault, D. Mellier, J. Tirillo, M.P. Bracciale, M. Zvonek, Engineering the interfacial adhesion in` basalt/epoxy composites by plasma polymerization, *Compos. A* 122 (2019) 67–76.
- [26] H. Daghigh, V. Daghigh, Free vibration of size and temperature-dependent carbon nanotube (CNT)-reinforced composite nanoplates with CNT agglomeration, *Polym. Compos.* 40 (S2) (2018) E1479–E1494.
- [27] Jr. H. Daghigh, V. Daghigh, A. Milani, D. Tannant, T.E. Lacy, J.N. Reddy, Nonlocal bending and buckling of agglomerated CNT-Reinforced composite nanoplates *Compos. B* 183 (2020) 107716.
- [28] D. Saber, M.A. Abd El-baky, M.A. Attia, Advanced fiber metal laminates filled with silicon dioxide nanoparticles with enhanced mechanical properties, *Fiber Polym.* 22 (2021) 2447–2463.
- [29] AA. Melaibari, MA. Attia, MA.A El-baky, Understanding the effect of halloysite nanotubes addition upon the mechanical properties of glass fiber aluminum laminate, *Fibers Polym.* 22 (2021) 1416–1433.
- [30] Jr V. Daghigh, T. Lacy, H. Daghigh, G. Gu, K. Baghaei, M. Horstemeyer, C. Pittman, Machine learning predictions on fracture toughness of multiscale bio-nano- composites, *J. Reinf. Plast. Compos.* (2020).
- [31] V. Daghigh, T.E. Lacy Jr, H. Daghigh, G. Gu, K.T. Baghaei, M.F. Horstemeyer, C. U. Pittman Jr, Heat deflection temperatures of bio-nano-composites using experiments and machine learning predictions, *Mater. Today Commun.* 22 (2020) 100789.
- [32] L. Sun, R.F. Gibson, F. Gordaninejad, Energy absorption capability of nanocomposites: a review, *Compos. Sci. Technol.* 69 (2009) 2392–2409.

- [33] M. Nasihatgozar, V. Daghigh, M. Eskandari, K. Nikbin, A. Simoneau, Buckling analysis of piezoelectric cylindrical composite panels reinforced with carbon nanotubes, *Int. J. Mech. Sci.* 107 (2016) 69–79.
- [34] Jr M. Nasihatgozar, V. Daghigh, T.E. Lacy, H. Daghigh, K. Nikbin, A. Simoneau, Mechanical characterization of novel latania natural fiber reinforced PP/EPDM composites, *Polym. Test.* 56 (2016) 321–328.
- [35] MA.A El-baky, DA. Hegazy, MA. Hassan, Advanced thin-walled composite structures for energy absorption applications, *Appl. Compos. Mater.* 29 (2022) 1195–1233 (Dordr).
- [36] MA.A El-baky, MA. Attia, Experimental study on the improvement of mechanical properties of GLARE using nanofillers, *Polym. Compos.* 41 (10) (2020) 4130–4143, <https://doi.org/10.1002/pc.25699>.
- [37] S.S. Vinay, M.R. Sanjay, S. Siengchin, C.V. Venkatesh, Effect of Al₂O₃ nanofillers in basalt/epoxy composites: mechanical and tribological properties, *Polym. Compos.* 42 (4) (2021) 1727–1740.
- [38] S.S. Vinay SS, M.R. Sanjay, S. Siengchin, C.V. Venkatesh, Basalt fiber reinforced polymer composites filled with nano fillers: a short review, *Materials Today Proc.* 52 (Part 5) (2022) 2460–2466. Pages 2460-2466.
- [39] Mechanical and Thermal Behavior of Multiscale Bi-nano-composites Using Experiments and Machine Learning Predictions, Daghigh, Vahid. PhD Dissertation, <https://scholarsjunction.msstate.edu/td/3045/>.
- [40] V. Daghigh, M. Soroush, K. Nikbin, A. Simoneau, Finite element modeling of tensile and bending on fiber reinforced polymer composites (n.d.), in: *Proceedings of the 3rd International Conference on Composites Characterization, Fabrication and Application (CCFA-3)*, December 18–19, Tehran, Iran, 2012. Iran University of Science and Technology accessed June 6, 2020, https://www.researchgate.net/publication/319044138_Finite_Element_Modeling_of_Tensile_and_Bending_on_Fiber_Reinforced_Polymer_Composites.
- [41] M. Sanchez, M. Campo, A. Jimenez-Suarez, et al., Effect of the carbon nanotube functionalization on flexural properties of multiscale carbon fiber/epoxy composites manufactured by VARIM, *Compos. B Eng.* 45 (2013) 1613–1619.
- [42] H. Khosravi, R. Eslami-Farsani, On the mechanical characterizations of unidirectional basalt fiber/epoxy laminated composites with 3-glycidoxypyltrimethoxysilane functionalized multi-walled carbon nanotubes–enhanced matrix, *J. Reinf. Plast. Compos.* 35 (2016) 421–434.