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The Mechanical Behavior of Silane-Modified Nano-Al_2O_3/ Basalt Fiber/ Polymer Composite Materials

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Abstract

In this research, the impact of modified alumina nanoparticles on tensile and flexural properties of basalt fibers-epoxy composites was experimentally examined. First, composite without alumina nanoparticles (neat sample) and composites with different loading of alumina nanoparticles (0.5, 1, 3, and 5 wt.%) were manufactured. For the purpose of having a better interaction of alumina nanoparticles with the matrix, the surface of alumina nanoparticles was modified by the silane coupling agent (3-methoxysilyl-propylamine (3-APMS)). Fourier transform infrared (FT-IR) spectrum validated the reaction mechanism between the silane compound and alumina nanoparticles. 3-APMS/alumina nanoparticles were added to the epoxy resin through mechanical and ultrasonic methods. The resulting mixes were then used to make alumina nanoparticles/woven basalt fibers-epoxy composites utilizing the hand-layup methods. The mechanical properties of the composites were studied under tensile and three-point bending tests. Also, field emission scanning electron microscope (FESEM) was employed to investigate the dispersion of alumina nanoparticles in the matrix and the fracture surfaces of specimens. The results showed that the dispersion of 1 wt.% modified nano-alumina, flexural strength, tensile strength, flexural modulus, and tensile modulus enhanced by 15.3, 14, 19.1, and 15.8 %, respectively, compared with a neat sample. The microscopic investigations verified that adding the 3-APMS/alumina nanoparticles to the matrix of basalt fibers-epoxy composites enhances the basalt fibers-matrix interface, resulting in improved mechanical properties.

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Keywords: Multiscale composites, Nano-alumina particles, Basalt fibers, Salinization, Mechanical properties

1. Introduction

Polymer composites incorporating micro-fibers and nanofiller particles have recently received a lot of attention. In this case, the material is called a multiscale composite [1]. Fiber-reinforced polymeric composites possess several distinct advantages over conventional materials like metals, ceramics, and polymers, including high strength-to-weight ratio and stiffness, superior corrosion resistance, and the ability to provide both mechanical and functional properties [2–4]. Consequently, these composites are used in high-performance applications such as automotive, marine, and construction owing to their brilliant properties [3,5].

Basalt fiber is a natural fiber that is derived from volcanic rock basalt. It is environmentally friendly and has high strength, stiffness, thermal stability, outstanding fiber-resin adhesion, and excellent chemical resistance compared to glass fibers [2,3,5]. Similarly, Epoxy resin is a thermosetting polymer that has found extensive applications as coatings, adhesives, electronic materials, and matrices for fiber-reinforced composites owing to their cost-effectiveness, exceptional chemical resistance, and mechanical properties, ranging from extreme flexibility to high strength and hardness, high adhesion strength, good heat resistance, and high electrical resistance [6]. Nonetheless, cured epoxy resins possess inherent brittleness due to their high crosslinking density, which limits their suitability for various engineering applications. To overcome this constraint, epoxy resins are frequently modified using liquid elastomers, thermoplastics, and inorganic particles. Functional nano-size particles added to the matrix can improve microstructure of composites and reduce the probability of matrix failure. Functional nano-size particles such as TiO_2, Al_2O_3, SiC, $CaCO_3$, $Mg(OH)_2$ and ZnO added to the matrix can improve properties such as flexural and tensile strength, also enhance interfacial interactions and intrinsic properties of the matrix [7,8]. Researches have been demonstrated that surface modification can enhance mechanical properties and wear resistance by improving interfacial adhesion between nanofillers and the polymer matrix. Silanization of nanofillers appears to be a viable approach for surface modification [9].

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In recent years, researchers have extensively investigated the effect of incorporating various nano-size particles into epoxy matrices with reinforcement on mechanical properties. For instance, Ary Subagia et al. [3] reported significant enhancements in both tensile and flexural strength and modulus of tourmaline-basalt fibers/epoxy composite laminates with the addition of 1 wt.% tourmaline nanoparticles. Also, Joshua et al. [10] indicated that the addition of 3 wt.% Al_2O_3 nanoparticles led to an enhancement in the impact strength of Kevlar/basalt fibers-epoxy composite. Similarly, Khosravi et al. [1] investigated the impact of adding Na-montmorillonite (Na-Mt) nano clay treated with 3-glycidoxypropyl trimethoxysilane (3-GPTS) on the mechanical properties of unidirectional basalt fibers/epoxy composite laminates. Their results showed that the addition of 5 wt.% 3-GPTS/Mt resulted in considerable increases in flexural, tensile, and compressive strengths. In addition, the scanning electron microscope (SEM) images showed better adhesion between the basalt fibers and the matrix in Mt-enhanced epoxy specimens. He et al. [11] showed that nano-Al_2O_3 particles enhance the compatibility, hardness, and strength of the fluororubber matrix, improving its resistance to external forces. They reduce friction and wear at both room and high temperatures by strengthening interfacial bonding and increasing thermal conductivity. Lee et al. [4] discovered that incorporating silane-treated basalt fibers into the epoxy-anhydride resin is critical and advantageous in the production of high-performance composites. The study revealed that silane coupling agents significantly influence both the thermal properties of the composites and the enhancement of the interfacial adhesion. Also, Azizi et al. [12] studied how incorporating silane-modified nano zirconia impacted the tensile and flexural properties of basalt fibers/epoxy composites. Their experimental findings demonstrated that when 3 wt.% nano zirconia was dispersed, there were significant enhancements in flexural and tensile properties. Furthermore Liu et al. [13] investigated the mechanical and electrical properties of Kaolin-KH550 modified basalt fibers-reinforced epoxy composite. They showed that the composites were modified by 1.5 wt.% Kaolin showed brilliant electrical insulation and mechanical performance. Sagar et al. [9] incorporated nano ZnO_2 into jute fiber/ epoxy composites, which resulted in a 43% increase in compressive strength and a significant improvement in wear resistance and friction coefficient (80% and 48% reduction, respectively) with the addition of 3 wt.% ZnO_2. SEM analysis confirmed improved interfacial bonding due to APTMS-silane modification of ZnO_2.

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Khosravi et al. [14] examined the impact of various contents of functionalized multi-walled carbon nanotubes (MWCNTs) on the mechanical responses of unidirectional basalt fibers/epoxy composites. They demonstrated improvements in tensile strength, flexural strength, and moduli of unidirectional basalt fibers/epoxy composites with the incorporation of 0.4 wt.% functionalized MWCNTs. Furthermore, Aghamohammadi et al. [15] examined the influence of MWCNTs on the flexural and high-velocity impact behavior of basalt fibers/epoxy/aluminum laminate composite. The results showed that adding 0.5 wt.% MWCNTs had a great impact on the adhesion and integrity of the composite. Consequently, the results demonstrated that the inclusion of 0.5 wt.% MWCNTs led to a remarkable enhancement in the flexural strength and modulus. Moreover, Sukur et al. [16] reinforced the epoxy matrix with varying concentrations of graphene nanoplates (GNPs) and manufactured basalt fibers/epoxy composites. The resulting composites showed significant improvements in tensile strength and modulus. Likewise, the basalt fibers/epoxy composites reinforced with graphene nanoplates demonstrated an increase in wear resistance. Similarly, Azimopour-Shishevan et al. [17] examined the low-velocity impact behavior of basalt fibers reinforced epoxy composites with different amounts of graphene nanoplates. The results demonstrated that incorporating graphene in the composites matrix enhanced low-velocity impact behavior by boosting the contact force and decreasing the energy absorbed in the rebounding state. According to the tests, the composite with 0.5 wt.% GNPs exhibited the best impact performance, withstanding an impact force of 15.08 kN compared to 13.78 kN and 11.208 kN for composites with 0.1 wt.% and no graphene, respectively Additionally, Sánchez et al. [18] showed when an amino-functionalized CNT is used to reinforce the carbon fibers/epoxy composite, it creates a more robust interface that enhances stress transfer and facilitates the involvement of more efficient strengthening mechanisms.

Among the nanofillers, alumina is one of the important ceramic materials that has outstanding mechanical properties such as high compressive strength and stiffness. Alumina can also be used in composites that work at relatively high temperatures due to its high hardness at high temperatures and non-flammability. According to the existing literature, no research has been conducted on the integration of alumina nanoparticles for reinforcing purposes in basalt fibers reinforced polymer matrix multiscale composites. Therefore, the current study's goal is to investigate the influence of varied weight ratios of silane-modified nano-alumina particles on the mechanical properties of basalt fibers-epoxy multiscale composites during tensile and flexural testing. In essence, the microscopic investigations confirm the role of silane-modified nano-alumina particles in enhancing the interface between basalt fibers and the epoxy matrix, thereby enhancing the overall mechanical properties of the composites.

2. Materials and methods

2.1. Materials

In the manufacturing process of the composite specimens, the epoxy resin of KER 828, along with a polyamine hardener (KUMHO P&B CHEMICALS, INC., Korea), was utilized as the matrix. Table 1 shows some of the resin's properties. The resin-hardener weight ratio was 100:10, as specified by the manufacturer. In addition, woven basalt fibers (Basaltex Co., Belgium) were used as the reinforcement, and their technical specifications are presented in Table 2. The γ-Al_2O_3 nanoparticles (Neutrino, Iran) were used to manufacture multiscale composites. The choice of γ-Al_2O_3 nanoparticles over α-Al_2O_3 ones was based on their higher specific surface area. While α-Al_2O_3 nanoparticles exhibit superior thermal properties and heat resistance, γ-Al_2O_3 nanoparticles were selected to enhance the mechanical properties of the composite. This decision was driven by their higher specific surface area and superior interaction with epoxy resin, resulting in a more significant improvement in the composite's flexural and tensile properties. The properties of alumina nanoparticles are given in Table 3.

Table 1. Properties of epoxy resin of KER 828 [19]

Physical State	Color	pH	Density at 25˚C	Dynamic Viscosity at 25˚C
Liquid	Pale Yellow	Circa 7	1160 Kg/m^3	12-14 Pa.s

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Table 2. Technical specifications of basalt fibers [20]

Fabric Name	Wave type	Thickness (mm)	Surface Density (g/m^2)	Sizing Type
7101/35/1500	Satins	0.3	3	Silane

Table 3. Specifications of nano-alumina particles [21]

Chemical Formula	Color	Shape	Crystal Phase	Average Particle Size (nm)	Density (g/cm^3)
γ-Al_2O_3	White	Spherical	Gamma (γ)	10-20	3.65

The 3-Trimethoxysilyl (silane) propylamine (3-APMS, Merck Co., Germany) was considered to modify the surface of nano-alumina particles to have a better interaction with the epoxy resin and to strengthen the matrix. Table 4 shows the specifications of the 3-APMS surface modification agent.

Table 4. The specifications of the 3-APMS surface modification agent [22]

Formula	Molar (g/mol)	pH (g/L in H_2O)	Flash Point ($^{\circ}C$)
$C_6H_{17}NO_2Si$	179.29	9 <	90

2.2. Surface modification of nano-alumina particles

First, 1 g of nano-alumina particles were combined in 100 ml solution, including 95% ethanol and 5% water, with an ultrasonic homogenizer (Fapan Co., Iran) at 80 W for 15 min. Second, the surface modifier with a weight ratio equal to nano-alumina particles was added to the mixture, and the new mixture was subjected to reflux for 8 hours at 70°C. The rotational speed of the solution using a magnetic stirrer was 100 rpm, and the pH of the solution was adjusted between 3-4 by adding 37% hydrochloric acid. After the reflux operations, the final solution was centrifuged (at 4000 rpm) twice for 15 minutes to separate the surface-modified nano-alumina particles. Before each centrifugation, this solution was washed with ethanol to separate unreacted silane compounds with nano-alumina particles, and then it was dried for 12 hours in an oven at 60°C.

2.3. Specimen fabrication and preparation

In order to fabricate the multiscale composite, the epoxy/ nano-alumina mixture was first prepared. For this purpose, surface-modified nano-alumina particles with four different weight ratios of 0.5, 1, 3, and 5 were initially distributed in the epoxy resin without hardener and using a high-speed mechanical stirrer (SDS-11D, Finetech Co., South Korea) at 2000 rpm for 15 minutes. Following that, an ultrasonic wave of 24 kHz and 120 W was applied for 60 minutes to break the existing agglomerates and adequately distribute the nano-alumina particles. Then, after cooling, the resulting mixture was again stirred by a high-speed mixer for 15 minutes at a speed of 2000 rpm. Next, hardener (with a ratio of 10:100 to epoxy) was added to the mixture and remixed. The resulting mixture was used as the matrix for fabricating basalt fibers/alumina nanoparticles reinforced epoxy multiscale composites via the hand-layup technique. In fabricating the samples, 4 layers of basalt fibers were used with an equal weight ratio of resin to fibers (50:50). To compare with the basalt fibers/alumina nanoparticles reinforced epoxy multiscale composites, a neat basalt fibers/epoxy composite was also made using the same technique. In order to final cure and achieve maximum strength in the samples, mechanical tests were performed after seven days of construction.

2.4. Mechanical tests

To perform mechanical tests and study the tensile and flexural behavior of samples, a universal testing machine (Hounsfield, H25K) was used. Flexural and tensile tests were conducted according to ASTM D790 [23] and ASTM D3039 [24], respectively.

In the three-point bending test, samples with dimensions of 150×20 mm^2 were placed on the fixtures so that about 25% of their length was outside the support to prevent them from sliding during the test. Then, all the samples were loaded from the middle with a strain rate proportional to the dimensions of the samples. Flexural properties were analyzed based on the force-displacement graph that the machine drew.

In the tensile test, the samples were cut with dimensions of 250×25 mm^2 and then sanded to fill the possible voids in the composite samples. In order to strengthen the place of contact of the samples with the jaw of the machine and prevent them from sliding, glass fibers/epoxy composite tabs with dimensions of 25×50 mm^2 were used on both sides of the samples. The cross-head speed was 2 mm/min during loading conditions, and then stress-strain graphs were drawn by the machine to analyze the tensile properties of the composite samples.

2.5. Microstructure characterization

To investigate the reactions between the silane agent and nano-alumina particles to ensure the effectiveness of the silane agent on the surface of nano-alumina particles and surface modification of it, FT-IR analysis (PerkinElmer) was performed. The specimens were scanned at a wavelength range of 400-4000 cm^{-1}, with a resolution of 4 cm^{-1}. Also, after performing mechanical tests, the FESEM test (Mira3-XMU) was utilized to investigate fracture surfaces and identify fracture mechanisms.

3. Results and discussion

3.1. FT-IR spectroscopy

FT-IR spectroscopy results of unmodified nano-alumina particles and silane-modified ones are represented in Figure 1. Comparing these two diagrams, observed that the absorption peaks at the wavelength of 3748 cm^{-1} show the presence of the -OH group on the surface of nano-alumina particles. Also, the wave number of 2936 cm^{-1} indicates the stretching vibration of 2CH- on the surface of nano-alumina particles. The wave number 1157 cm^{-1} represents the tensile vibration of C-N on the nano-alumina surface, which confirms the silane coupling agent's successful interaction on the nano-alumina surface [26, 27].

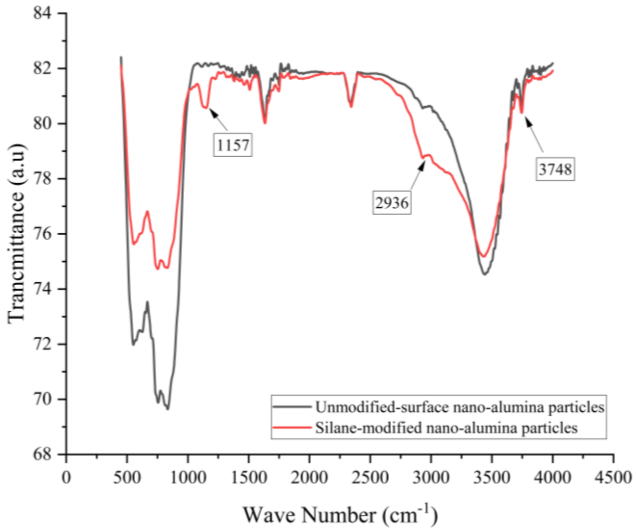

Figure 1. FT-IR spectra of silane-modified and unmodified nano-alumina particles.

3.2. Mechanical tests

The stress-strain curves of the flexural test of the specimens containing various weight percent of nano-alumina particles are depicted in Figure 2. According to Figure 2, it can be seen that the samples have non-uniform elastic-plastic behavior, which indicates the heterogeneous structure change in the samples. The flexural strength (181 MPa) of sample with 0.5 wt.% nano-alumina particles was slightly higher than that of the neat composite (180.38), showing an increase of approximately 0.34%. In other words, adding 0.5 wt.% nano-alumina particles to the basalt fibers/epoxy composite did not significantly affect flexural properties. Among all specimens, the highest flexural strength of 207.98 MPa was obtained for the sample with 1 wt.% nano-alumina particles, which shows a rise of 15.3%, compared to neat sample. Adding 3 wt.% nano-alumina particles has caused a reduction in flexural strength compared to the sample with 1 wt.% nano-alumina particles. The flexural strength in a sample containing 3 wt.% nano-alumina particles has reached 189.85 MPa from 207.98 MPa in a sample containing 1 wt.% nano-alumina particles, showing an 8.7% decrease. Further, by adding 5 wt.% nano-alumina particles, the flexural strength has continued to decrease. The flexural strength in the sample containing 3 wt.% nano-alumina particles has reached 155.14 MPa, representing a decrease of 18.3%.

The flexural modulus of samples containing 0.5 wt.% and 1 wt.% nano-alumina particles was respectively enhanced to 9.04 and 10.60 GPa compared to the neat sample (8.9 GPa), indicating a decrease in flexibility. By adding 3 and 5 wt.% nano-alumina particles, the flexural modulus has decreased compared to the sample with 1 wt.% of nano-alumina particles. The flexural modulus in the sample of 1 wt.% nano-alumina particles has reached 8.16 and 8.05 GPa for samples containing 3 and 5 wt.% nano-alumina, respectively.

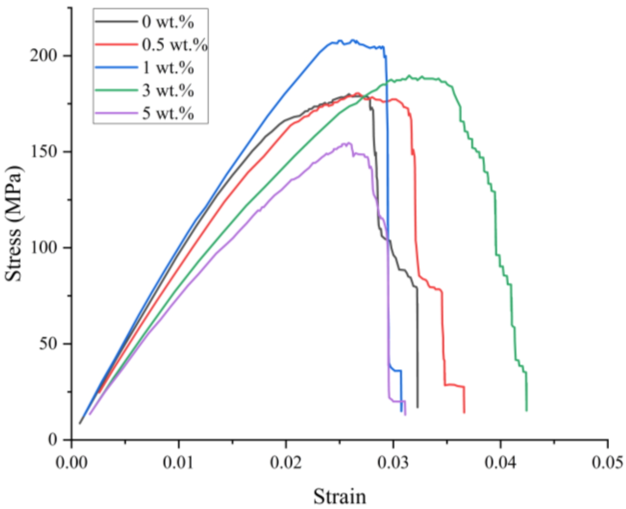

Figure 2. The stress-strain curves of the flexural test of the basalt fibers/nano-alumina reinforced epoxy composites containing different weight percent of nano-alumina particles.

Figure 3 represents the stress-strain curve of the tensile test of the specimens containing different weight percent of nano-alumina particles. Based on Figure 3, the behavior of the samples is fully elastic, and the samples are brittlely broken without plastic deformation. The tensile strength of the specimen containing 0.5 wt.% nano-alumina particles was enhanced to 146.42 MPa compared to the sample without nano-alumina particles (134.33 MPa), indicating an increase of 9% in tensile strength. The highest tensile strength has been obtained when 1 wt.% of nano-alumina particles were added. In this sample, the strength has reached to 153.22 MPa, which shows an improvement of 14%. Adding 3 wt.% of nano-alumina particles has decreased the tensile strength compared to the sample with 1 wt.% of nano-alumina particles.

The tensile modulus increased from 3.17 GPa in the sample without nano-alumina to 3.97 GPa in the 0.5 wt.% sample, and then it decreased to 3.67 GPa with the addition of 1 wt.% of nano-alumina particles. The tensile modulus in the sample of 1 wt.% of nano-alumina

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particles has reached 3.63 and 3.22 GPa for samples with 3 and 5 wt.% of nano-alumina particles, respectively.

The summary of the results (strength and modulus) of the tensile and flexural tests on basalt fibers/epoxy composite samples having various weight percentages of nano-alumina particles are given in Table 5 and in the form of diagrams in Figure 4.

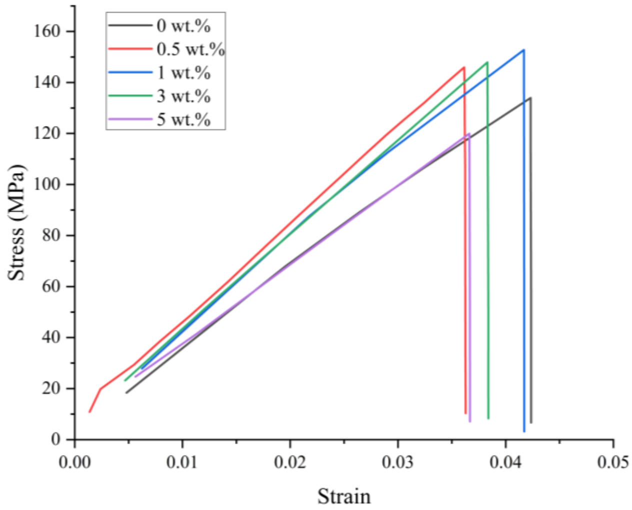


Figure 3. The stress-strain curves of the tensile test of the basalt fibers/alumina reinforced epoxy composites containing different weight percent of nano-alumina particles.

Table 5. The summary of the results of the tensile and flexural tests on basalt fibers/epoxy composite samples avenging various weight percentages of nano-alumina particles

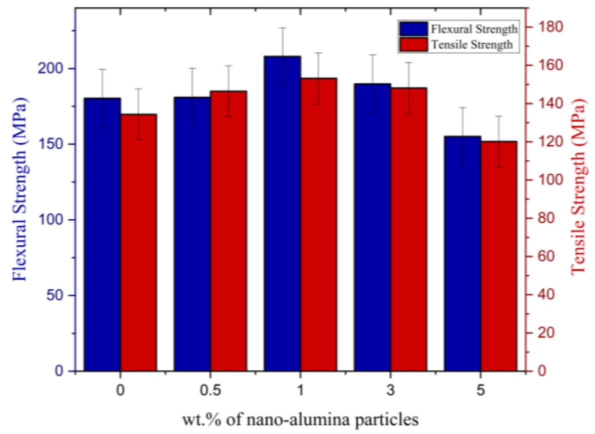
Nano-Alumina (wt.%)	Flexural Strength (MPa)	Flexural Module (GPa)	Fracture Strain	Tensile Strength (MPa)	Tensile Module (GPa)	Tensile Strain
0	180.38	8.9	0.029	134.33	3.17	0.042
0.5	181	9.04	0.026	146.42	3.97	0.036
1	207.98	10.60	0.024	153.22	3.67	0.041
3	189.85	8.16	0.031	148.18	3.63	0.040
5	155.14	8.05	0.025	120.14	3.22	0.037

Figure 4 illustrates the impact of incorporating nano-alumina on both flexural and tensile strength, as well as the flexural and tensile modulus of the samples. According to Figure 4a, it can be observed that the flexural and tensile strength of the samples initially increased and then decreased with the increase of nano-alumina particles in the matrix. The highest amount of strength corresponds to the specimen containing 1 wt.% nano-alumina particles. According to Table 5, this sample has increased by 15.3% and 14%, respectively, for flexural and tensile strength compared to the sample without nano-alumina particles. The increase in composite strength is attributed to the addition of nano-alumina particles, which results in the amelioration of the interface between the matrix and the basalt fibers. In this case, the load is transferred from the matrix to the fibers well, and this leads to an increase in the strength of the final composite. In other words, the presence and addition of nano-alumina cause fibers and matrix to be entangled and locked by creating a bridge between them. When the basalt fibers/epoxy composites containing nano-alumina particles are subjected to mechanical tests, most of the applied load is concentrated on the fibers and the fibers are considered as the main load bearing component. The reinforcement of the matrix with surface-modified nano-alumina particles lead to a reduction in stress concentration both on the fibers and at the fibers-matrix interface. Consequently, the required stress for fiber rupture and interface separation decreases, resulting in an overall increase in the strength of the sample. But adding 3 and 5 wt.% nano-alumina particles lead to decreased 8.7% and 25.4%, respectively, in flexural strength compared to the sample with 1 wt.% of nano-alumina particles. The tensile strength for these samples has also decreased 3.3% and 21.6%, respectively. This lower level of strength can be attributed to the reduction of the adhesion of fibers and the matrix due to the formation of a discontinuous network in the matrix as well as the formation of defects and agglomerates of nano-alumina particles in weight percentages higher than 1% [27].

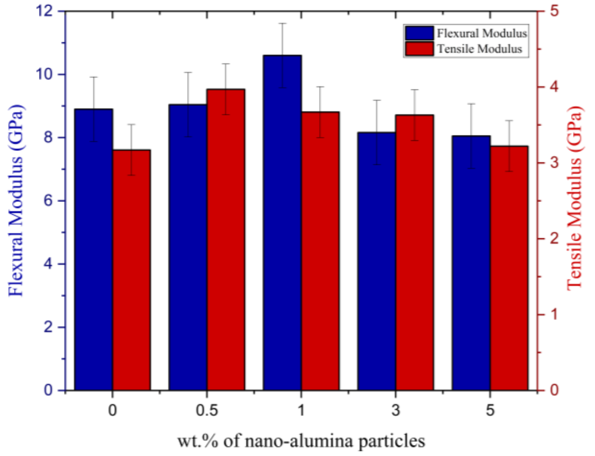
The flexural strength has reached from 180.38 MPa in the sample without nano-alumina particles to 207.98 MPa in the specimen with 1 wt.%, and the tensile strength has reached from 134.33 MPa in the sample without nano-alumina particles to 153.22 MPa in the specimen with 1 wt.% nano-alumina particles. The difference in the rise of flexural strength compared to tensile strength in samples without and 1 wt.% nano-alumina is 27.6 MPa and 18.89 MPa, respectively. It can be seen that the effect of adding nano-alumina particles in a specific weight percentage on the flexural strength of the sample is more significant than its effect on the tensile strength of the same sample. This is because, during the three-point bending test, the sample above the neutral axis experiences compressive forces, while the sample below it experiences tensile forces [28]. As a result, the matrix's efficiency is more excellent than when the sample is subjected to uniaxial tension. Therefore, by strengthening the matrix with nano-alumina, a more remarkable improvement is achieved in the bending strength of the sample compared to the tensile strength [27].

According to figure 4b, the flexural and tensile modulus of the sample containing 0.5 wt.% of nano-alumina particles has raised by 1.6% and 25.2%, respectively, compared to the sample without nano-alumina particles. For adding 1 wt.% of nano-alumina particles, the flexural and tensile modulus has improved by 19.1% and 15.8%, respectively, compared to the composite without nano-alumina particles.

In more significant amounts of nano-alumina (3 and 5 wt.%), the flexural modulus and tensile modulus showed a downward trend compared to the 1 wt.% nano-alumina particles sample. The presence of nano-alumina particles in the matrix limits the movement of the polymer chain, which, as a result, increases the stiffness of the material [27]. Also, owing to the very high modulus of nano-alumina compared to epoxy, the modulus (flexural and tensile) of the resulting nanocomposite can be expected to increase. The decrease in flexural and tensile modulus for higher weight percent of nano-alumina (3 and 5 wt.%) is probably caused by the agglomeration of nano-alumina particles in the matrix, which reduces their effect on the hardness of the material [29]. Also, since the binding force between atoms (nano-alumina and ground) has decreased due to agglomeration, the tensile and bending modulus in 3 and 5 wt.% has taken a downward trend. In the samples containing 3 and 5 wt.% nano-alumina particles, the flexural modulus is 8.16 and 8.05 GPa, respectively, which has decreased by 8.3% and 9.5% from the neat sample, respectively. The tensile modulus was also 3.63 and 3.22 GPa in 3 and 5 wt.% nano-alumina particles, which has increased by only 14.5% and 1.6%, respectively, compared to the neat sample. These results indicate a decrease in the rate of modulus increase for a higher weight percentage of nano-alumina particles. The reason for the strong downward trend of the flexural modulus in 3 and 5 wt.% nano-alumina particles is probably due to the more significant influence of the matrix in the flexural test (as mentioned); that is, the agglomeration of nano-alumina particles and the subsequent lack of suitable interatomic force in the matrix has reduced the flexural modulus. On the other hand, the tensile modulus has mostly stayed the same for higher weight percentages due to more dependence on fibers than the matrix [14].



(a)



(b)

Figure 4. Effect of adding different amounts of nano-alumina particles in the epoxy matrix on a) tensile and flexural strength, b) tensile and flexural modulus of composite samples.

3.3. Study of the fracture surfaces

To investigate the fracture surfaces of the specimens and to identify the mechanisms of nano-alumina particles effect on the mechanical behavior of basalt fibers/epoxy composites, microscopic studies were conducted using FESEM (Figures 5-12). The fracture surface of the neat composite specimen (without the addition of nano-alumina particles), shown in Figure 5, shows basalt fibers that have a clean and smooth surface. In Figure 6, the basalt fibers debonding from the epoxy matrix in samples without nano-alumina particles and containing 1 wt.% nano-alumina particles can be seen, which occurred due to the weak bond between the epoxy matrix and the basalt fibers [18].

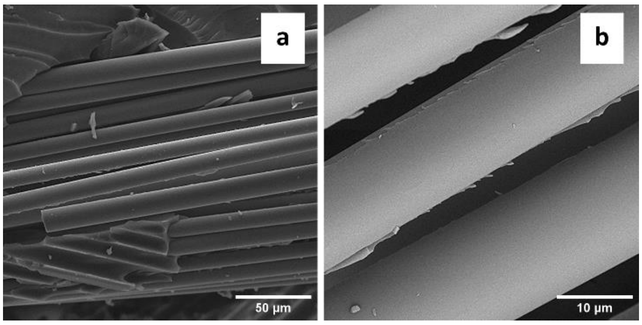


Figure 5. FESEM images of the basalt fibers surface of the basalt fibers/epoxy composite sample without nano-alumina particles, a) smooth and clean basalt fibers surface, b) enlarged image.

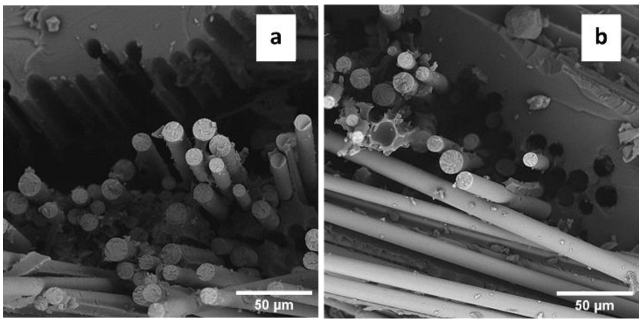


Figure 6. Basalt fibers debonding from epoxy matrix, a) sample without nano-alumina particles, b) sample containing 1 wt.% nano-alumina particles.

Figure 7 shows the presence of the epoxy matrix phase on the basalt fibers in the samples of 0.5, 1, 3, and 5 wt.% nano-alumina particles. These images indicate the adhesion of the matrix phase (epoxy/nano-alumina particles) on the basalt fibers.

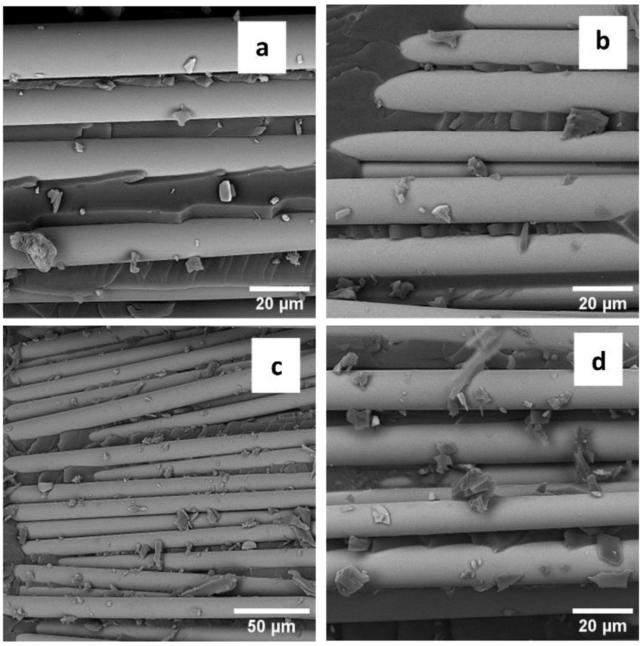


Figure 7. Epoxy matrix phase on basalt fibers in composite samples containing, a) 0.5 wt.%, b) 1 wt.%, c) 3 wt.%, d) 5 wt.% of nano-alumina particles in the matrix.

As a result, the dominant failure mechanism in the samples can be drawn by comparing their images. In the case of the nanocomposite sample, the fracture mechanism is mainly owing to the delamination of the fiber-matrix interface, where as in the case of neat sample, cracking of the matrix is the dominant fracture mechanism.

Figure 8-a presents the fracture surface of the nanocomposite matrix in the sample containing 1 wt.% nano-alumina particles, which have many shear bands. The reason for this is the presence of nano-alumina particles in the epoxy matrix. The areas marked with arrows are shear bands. When the crack reaches the fibers, the presence of nanoparticles changes the crack path and fracture mode, which causes energy loss. As a result, less fracture energy reaches the fibers and increases the overall fracture energy of the composite [30]. Figure 8-b depicts the existence of modified nano-alumina particles on the basalt fiber surface in the composite sample containing 1 wt.% of nano-alumina particles. The areas marked with arrows are nano-alumina particles on basalt fibers. This figure represents the proper adhesion of nano-alumina particles on the fiber surface, which is due to the surface modification of nano-alumina particles. Surface modification of nano-alumina particles causes the energy that is used to fracture and transfer the load to the fibers to be dissipated by nano-alumina particles, and the fibers fracture at higher stress [30–32].

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https://doi.org/10.1007/s10853-024-10080-1

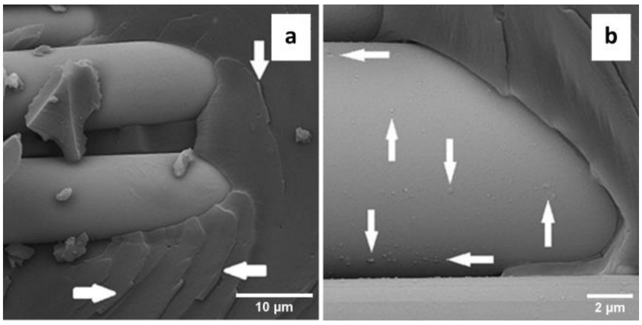

Figure 8. fracture surface of the nanocomposite matrix in the sample containing 1 wt.% nano-alumina particles, a) Shear bands in epoxy matrix, b) Distribution of nano-alumina particles on basalt fibers.

Figure 9 shows the fracture surface of the matrix in nanocomposites containing 3 and 5 wt.% nano-alumina particles. The areas marked with arrows show the presence of some nano-alumina agglomerates in the composite matrix, which is the main reason for the loss of some mechanical properties in the samples containing 3 and 5 wt.% nano-alumina particles. The existence of agglomerates within the matrix creates regions of stress concentration and prompts the formation of cracks. In order to better distribute nano-alumina particles in the matrix, the power and time of ultrasonic operation should be increased. Also, better distribution depends on multiple factors such as the specific properties of the nano-alumina particles, the matrix material, and the desired outcome of the distribution process [30, 31, 33].

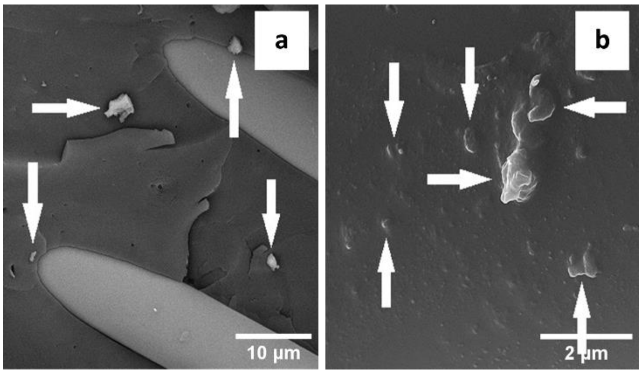

Figure 9. Nano-alumina agglomerates in the matrix, a) sample containing 3 wt.%, b) sample containing 5 wt.% nano-alumina particles.

Figure 10 shows the presence of holes in the matrix, which are another factor for the loss of mechanical properties. The areas marked with arrows are holes in the matrix. To reduce porosity, other methods can be considered for manufacturing composites. In the sample containing 1 wt.% of nano-alumina particles, the crack path has changed due to reaching nano-alumina particles. Figure 11-a shows the change in the crack path owing to the existence of nano-alumina particles in the crack path. Also, according to Figure 11-b, it can be seen that the crack stops due to reaching nano-alumina particles. Areas marked with arrows indicate crack changes and crack stops. Nanoparticles, with their high surface area and unique mechanical properties, can significantly enhance the mechanical performance of fiber-reinforced polymer composites by bridging the gap between the fibers and the matrix. Furthermore, these nanoparticles inhibit crack propagation within the composite, thereby improving its durability and fracture toughness [34].

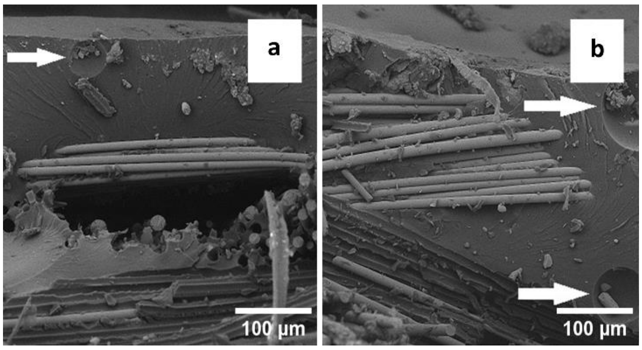

Figure 10. Presence of the hole in the composite matrix containing a) 1 wt.%, b) 5 wt.% nano-alumina particles.

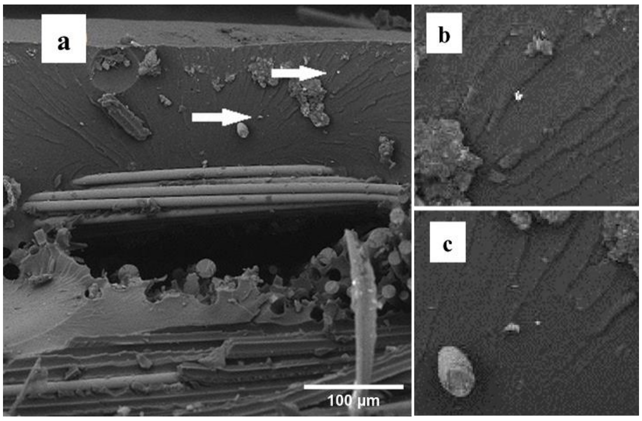

Figure 11. FESEM image of a) change direction and crack stop, b) magnified image of crack change direction, c) magnified image of crack stop.

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Figures 12-a to 12-d shows the distribution and histogram of the size ranges of nano-alumina particles on the composite surface containing 0.5, 1, 3, and 5 wt.% nano-alumina particles, respectively. As shown in the histograms, the average size of the samples containing 0.5, 1, 3, and 5 wt.% nano-alumina particles are 0.12342, 0.12436, 0.1316 and 0.13901 µm, respectively. It can be seen that increasing the weight percentage of nano-alumina particles in the samples has increased the distribution and size of nano-alumina particles on the surface of the samples.

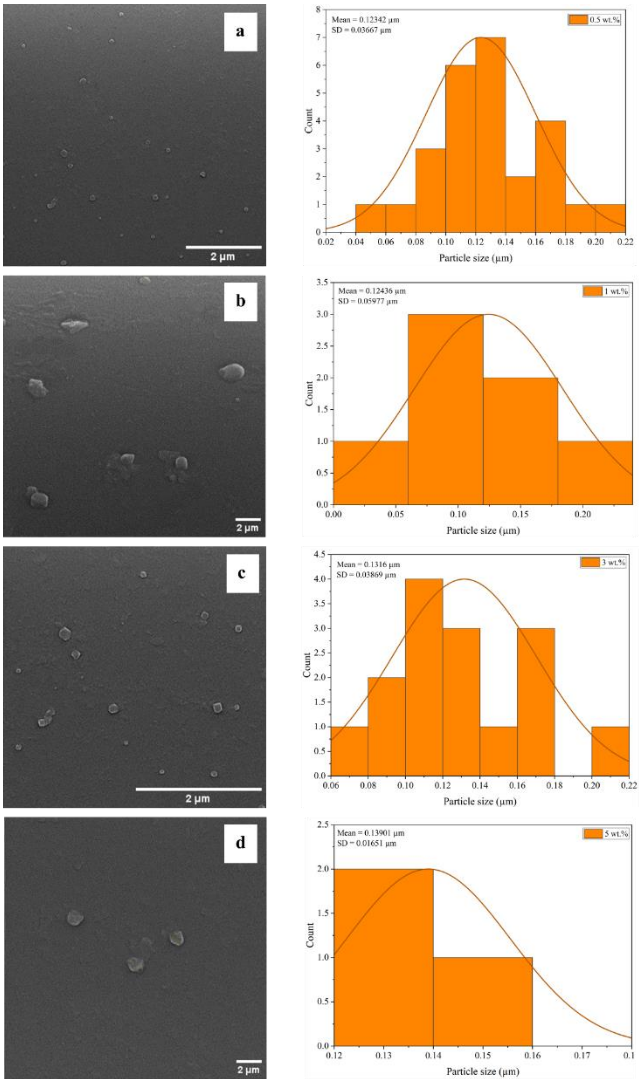

Figure 12. Distribution and size of nano-alumina particles on the surface of the samples containing a) 0.5 wt.%, b) 1 wt.% c) 3 wt.% and d) 5 wt.%.

4. Conclusion

This research examines the influence of adding nano-alumina particles in amounts of 0.5, 1, 3, and 5 wt.% on the mechanical properties of basalt fibers/epoxy composites under tension and bending loads, and with the basalt fibers/epoxy composite without nano-alumina particles (neat sample) compared. In order to enhance the interaction of nano-alumina particles with the epoxy matrix, their surface was first modified by a silane coupling agent (3-methoxysilyl-propylamine). FT-IR spectroscopy confirmed the successful interaction between the silane compound and the carboxyl groups on the nano-alumina particle's surface. Adding surface-modified nano-alumina particles as a secondary reinforcing component improves the mechanical properties of basalt fibers/epoxy composites. Also, microscopic studies of the fracture surface of the samples showed the improvement of the interface between the basalt fibers and the epoxy matrix because of the presence of modified nanoparticles. The mechanical results showed that the highest impact of modified nano-alumina particles on both tensile and flexural strength was obtained in composites containing 1 wt.% of nano-alumina particles. In this sample, the flexural and tensile strength increased by 15% and 13.5%, respectively, compared to the neat sample. In addition, the composite that contained 1 wt.% of modified nano-alumina particles appeared to have the most prominent effect on tensile and flexural modulus. In this particular sample, the flexural and tensile modulus increased by 15% and 19%, respectively, compared to the neat sample. According to the results, the effect of nano-alumina particles in a certain amount on flexural strength was more significant than the effect of the same amount of nano-alumina particles on improving tensile strength.

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Conflict of interest: The authors declare that they have no conflict of interest.

Data and code availability: Not Applicable

Supplementary information: Not Applicable

Ethical approval: Not Applicable

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