



Article

Machine Learning Approach for Mechanical Property Prediction of a Bio-Epoxy and Glass Fiber Composite Reinforced with Titanium Dioxide Nanoparticles

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Abstract

Glass fiber reinforced polymers (GFRPs) have drawn significant attention given their lightweight, mechanical resistance and tunable properties through constituent selection. Due to environmental concerns, research efforts have focused on incorporating sustainable materials, such as bio-epoxy resins, to reduce the ecological impact of GFRPs. This study characterizes a GFRP containing a bio-epoxy resin matrix, various loadings of titanium dioxide (TiO₂) nanoparticles, and a stabilized arrangement of glass fiber. The unreinforced composite exhibited a tensile strength and modulus of 214 MPa, and 13 GPa, respectively, and a flexural strength and modulus of 375 MPa and 14.5 GPa, respectively. The addition of TiO₂ produced an improvement in mechanical response for all the composites. The formulation with 1 wt.% TiO₂ showed the best tensile response with an improvement of 13% and 14% for its tensile strength, and modulus, respectively; meanwhile, the composites with 2 wt.% TiO₂ attained an improvement of 19% and 40% for the flexural strength and modulus, respectively. Scanning electron microscopy (SEM) revealed significant changes in the fracture mechanism of the composites, while energy-dispersive spectroscopy (EDS) confirmed an even nanoparticle distribution. Additionally, machine learning (ML) models were developed to predict the mechanical response as a function of the TiO₂ content.

Keywords: glass fiber reinforced polymer (GFRP); bio-epoxy resin; titanium dioxide nanoparticles; mechanical performance; machine learning modeling



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1. Introduction

In recent decades, polymer matrix composites have progressively replaced traditional materials in a wide range of applications due to the possibility of tailoring their mechanical resistance, thermal conductivity, wear resistance, electrical properties, among other

properties through a careful selection of the composite constituents [1–3]. For instance, the addition of aluminum nitride particles to a polymer matrix has shown to significantly increase the composite thermal conductivity when compared to the neat polymer [4,5]. Similarly, the addition of graphene oxide to epoxy resins has demonstrated a substantial improvement in the composite mechanical resistance [2,6]. In the same manner, the incorporation of glass fiber to different polymers has proven an outstanding efficiency in enhancing mechanical properties, corrosion resistance and thermal stability of unreinforced polymers [7–9].

Thermoset polymers are widely used in the production of composite materials due to their superior mechanical resistance, durability, chemical resistance, and high glass transition temperature when compared to other polymer families [10–12]. Among thermoset polymers, epoxy resins have attracted considerable attention because of their excellent adhesion, good mechanical performance, chemical resistance, and superb electrical insulation properties [13,14]. Despite these advantages, epoxy resins exhibit inherently brittle behavior and relatively low fracture toughness [15,16]. Nevertheless, their processing versatility allows them to be used in the fabrication of particle or fiber reinforced composites with enhanced properties [17–19]. In recent years, increasing sustainability concerns have driven the development of bio-based epoxy (i.e., bio-epoxy) resins that are synthesized with reduced petroleum-derived content. Consequently, more environmentally friendly alternatives to conventional epoxy resins have been formulated by partially substituting petroleum-based components with materials originating from renewable sources [20–23].

Glass fiber reinforced polymers (GFRPs) are manufactured using different techniques by embedding glass fiber with different orientations and arrangements into thermoset polymer matrices, typically epoxy resins, to enhance mechanical strength, wear and corrosion resistance [24–26]. In this context, GFRPs can achieve strength to weight ratios comparable to those of steel or aluminum [7,26,27]. Despite these advantages, GFRPs remain vulnerable to matrix-dominated failure mechanisms, such as interlaminar shear and microcracking, which may compromise their long-term structural performance [28,29]. To address these limitations, the incorporation of particle reinforcements has emerged as an effective approach to further increase the mechanical performance and durability of GFRPs by acting as barriers for microcrack propagation and hindering interlaminar shear [30–33]. For example, Alsaadi et al. [30] investigated the addition of silicon carbide (SiC) particles to a 16-layer glass fiber reinforced epoxy resin laminate fabricated via hot compression molding. The inclusion of 5 wt.% SiC produced an average improvement of approximately 30% in shear strength compared to the unfilled composite. Similarly, Gao et al. [31] incorporated aminosilane-functionalized graphene oxide particles into epoxy-based GFRPs using an ultrasound-assisted impregnation method. The addition of 0.3 g of the reinforcement by square meter of the E-glass fiber improved tensile, flexural, and interlaminar shear strengths by 19%, 22%, and 28, respectively, compared to the unfilled material. Likewise, Megahed et al. [32] developed woven epoxy/E-glass composites reinforced with titanium dioxide (TiO₂) nanoparticles through vacuum infusion. The study concluded that the addition of 0.5 wt.% TiO₂ increased tensile strength, tensile modulus, flexural strength, and flexural modulus by 9%, 50%, 4%, and 49%, respectively.

Among the various particle reinforcements employed in GFRPs, titanium dioxide (TiO₂) has emerged as a promising filler candidate due to its chemical stability [34], cost-effectiveness [35], potential for uniform dispersion [34,36], and high surface area [34,36]. Additionally, TiO₂ nanoparticles have been reported to enhance mechanical performance of composites without affecting their glass transition temperature [32,37], while also improving wear and corrosion resistance [32,38,39]. Previous studies have reported that including appropriate loadings of TiO₂ can increase interlaminar shear strength of laminated com-

posites by toughening the interfacial regions, thereby delaying crack propagation and suppressing premature delamination [32,40–42].

In recent years, machine learning (ML) algorithms have been used to predict the properties of composite materials [2,43–45]. For instance, Navas-Pinto et al. [2] employed Gaussian process regression to predict the tensile strength (TS) of bio-epoxy/graphene oxide (GO) composites and identified an optimal filler loading between 0.22 and 0.25 wt.% of GO. Similarly, Barrionuevo and Ramos-Grez [43] applied ML algorithms to validate the mechanical properties of 3D-printed PLA specimens with the addition of 30 wt.% of beech wood fibers, achieving a root mean square error of 3.09 which validated the applicability of the approach. In the same way, Liu et al. [44] applied Gaussian support vector machine (G-SVM) models to estimate the tensile modulus and strength of graphene/aluminum nanocomposites obtaining an R-square value of approximately 0.945. Correspondingly, Kim and Oh [45] utilized artificial neural networks (ANNs) to predict the UTS of basalt fiber-reinforced polymers (BFRPs) and GFRPs, reporting string agreement between experimental and predicted values.

Despite the growing number of studies on TiO₂ nanoparticles reinforcement in glass fiber reinforced epoxy resins, studies addressing the influence of varying nanoparticle loadings in bio-epoxy-based GFRPs remain limited. Therefore, the present study investigates the incorporation of TiO₂ nanoparticles into a bio-epoxy/glass fiber composite fabricated via vacuum-assisted hand lay-up process, with the objective of enhancing its overall mechanical performance. Furthermore, machine learning models were developed to predict the mechanical properties of the composite which were corroborated through an experimental characterization process.

2. Materials and Methods

2.1. Raw Materials

Super SAP CPM/CPL, a 33% bio-based epoxy resin was acquired from Entropy Resins Inc. (San Antonio, TX, USA). TiO₂ nanoparticles, with an average particle size between 15 and 25 nm, were purchased from Jiangsu XFNANO (Nanjing, China). BGF Aerialite 1522, a 4-oz E-glass cloth (glass fiber) with a plain weave, was procured from BGF Industries (Danville, VA, USA).

2.2. Composite Fabrication

The glass fiber reinforced bio-epoxy (GFRBE) resin with TiO₂ nanoparticles was manufactured through a vacuum assisted hand layup method using a stabilized arrangement of the fibers, as shown in Figure 1.

Prior to the composite fabrication process, as illustrated in Figure 2, different loadings of the particle reinforcement were added to the bio-epoxy resin at the proportions shown in Table 1, which were selected after performing a literature review [32,46–48]. The respective amount of the filler was added to the CPM bio-epoxy and stirred at a constant speed of 750 RPM for a duration of 25 min to achieve a uniform particle dispersion [30,49,50]. Then, the CPL hardener was added to the mixture and mixed thoroughly for 10 min, followed by vacuum degassing [2].

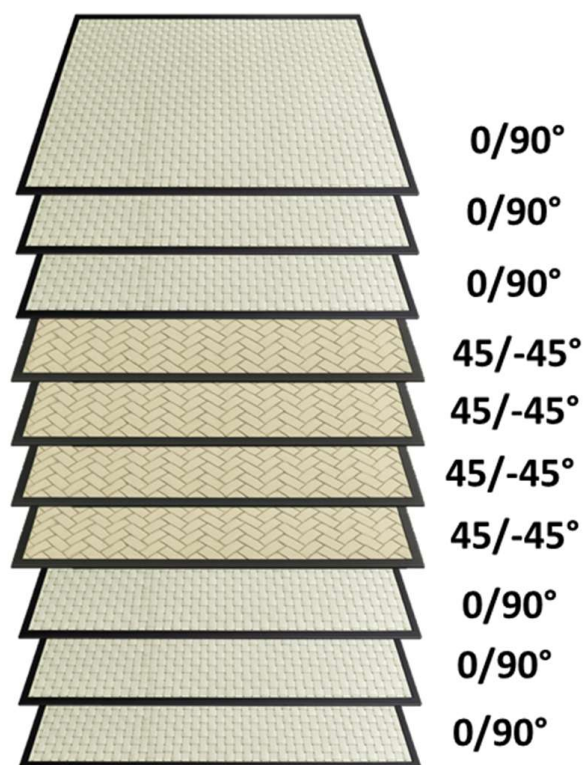


Figure 1. Orientation of the different layers in the composite.

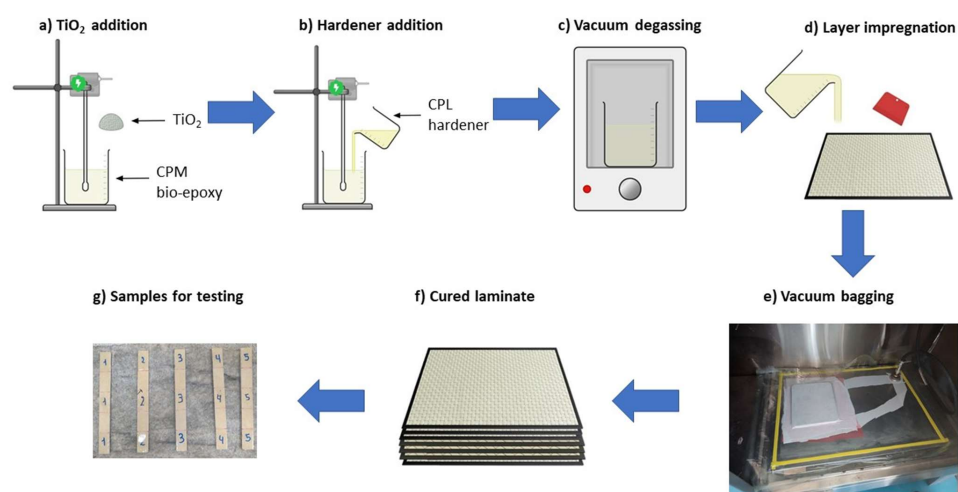


Figure 2. Composite fabrication schematic diagram.

Table 1. Weight percentage of each of the components for the reinforced matrix preparation.

Formulation	Titanium Dioxide (wt.%)	CPM Bio-Epoxy (wt.%)	CPL Hardener (wt.%)
1	0.0	70.40	29.60
2	0.5	70.05	29.45
3	1.0	69.70	29.30
4	2.0	69.00	29.00

The E-glass cloth was cut into 300 mm × 400 mm rectangular sheets considering the different orientations used for the stabilized arrangement [51]. Subsequently, a sealant tape was placed in a rectangular shape onto an aluminum plate. Then, the area inside of

the sealant tape was covered with a release agent to allow the laminate removal after the curing process. Afterwards, every layer, as outlined in Figure 1, was impregnated with the different reinforced bio-epoxy formulations using a spatula and stacked on top of the plate. Later, a peel ply film was placed over the laminate. Next, a perforated releasing film was placed on top, followed by a breather and bleeder fabric and the vacuum bagging film. Once the vacuum sealing of the bagging system was confirmed, the laminate was subjected to a 0.3 MPa pressure, used to uniformly compress the composite laminate. Finally, the sealed vacuum bag containing the laminate was placed in an oven at a temperature of 82 °C during 40 min to allow the curing process of the bio-epoxy resin matrix [52,53]. A schematic vacuum bagging configuration is shown in Figure 3. Once the composites were adequately cured, the vacuum bagging was removed and the composite was cut according to ASTM standards D3039-17R25 [54] and D7264-21 [55] for tensile and flexural testing, respectively, and further described in Table 2.

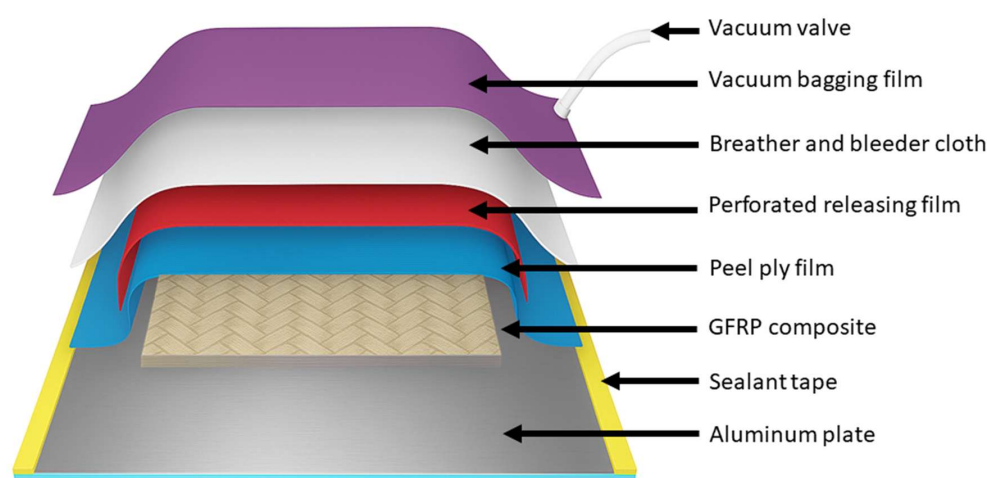


Figure 3. Vacuum bagging schematic configuration.

Table 2. Specimen dimensions for composite characterization.

Characterization Test	ASTM Standard	Specimen Dimensions
Tensile test	D3039-17R25 [54]	Length: 250 mm Width: 25 mm Thickness: 1.5 mm
Flexural test	D7264-21 [55]	Length: 125 mm Width: 12.5 mm Thickness: 1.5 mm Support span to depth ratio: 40 to 1

2.3. Composite Characterization

Tensile tests were performed on rectangular samples for each of the reinforcement loadings (0.0, 0.5, 1.0, and 2.0 wt.%) according to D3039-17R25 ASTM standard [54] with an Instron 600 LX universal testing machine equipped with a video extensometer (Norwood, MA, USA). For the purpose, a constant head displacement of 2 mm/min and an initial gauge length of 90 mm were used.

Three-point flexural tests were performed on rectangular samples for each of the reinforcement loadings (0.0, 0.5, 1.0, and 2.0 wt.%) according to D7264-21 ASTM standard [54] with an Instron 600 LX universal testing machine (Norwood, MA, USA) using a

fixed crosshead displacement of 1 mm/min. The flexural stress and strain were calculated according to the mentioned standard using Equations (1) and (2), respectively:

$$\sigma = \frac{3PL}{2bh^2} \quad (1)$$

$$\varepsilon = \frac{6\delta h}{L^2} \quad (2)$$

where σ is the flexural stress at the outer surface in the load span region (MPa), P is the applied force (N), L is the support span (mm), b is the width of the beam, h is the thickness of the beam, ε is the flexural strain at the outer surface at mid-span (mm/mm), and δ is the mid-span deflection (mm).

SEM was used for the fractured surface morphology examination on both tensile and flexural specimens with a Tescan Mira 3 (Brno, Czech Republic) operated with an acceleration voltage of 5 kV. For that purpose, the fractured specimens were cut at a distance of approximately 5 mm from the fracture and mounted in a specimen holder. Prior the SEM observation, the specimens received a gold coating layer by means of Quorum Q150 R gold evaporator (Laughton, United Kingdom) to improve their conductivity and avoid a charging effect on the samples [23,56].

In addition, to corroborate a homogeneous particle distribution of the reinforcement within the bio-epoxy resin matrix, energy-dispersive spectroscopy (EDS) was used to obtain elemental mapping along the samples [57–59]. The SEM microscope was equipped with a BrukerXFlash 6-30 EDS detector (Billerica, MA, USA) with an energy resolution of 123 eV at Mn-K α radiation and operated at an acceleration voltage of 25 kV. Ten measurements were performed at different positions and average values were reported.

Similarly, Fourier-transform infrared (FT-IR) spectroscopy was used to assess the interaction of the reinforcement within the bio-epoxy resin matrix [57] by means of a PerkinElmer Frontier spectrometer (Waltham, MA, USA). In this sense, the different spectra were individually acquired along the range from 500 to 4000 cm^{−1} using a resolution of 4 cm^{−1}.

2.4. Machine Learning Prediction

The experimental dataset included 49 tensile strength measurements for a bio-epoxy/glass fiber composite reinforced with varying amounts of TiO₂ nanoparticles, along with 45 measurements of flexural strength. The raw data were sourced from a GitHub v3.15.12 repository and initially examined using descriptive statistics and scatter plots to verify a normal distribution, confirm the absence of missing values, and identify a clear nonlinear relationship between TiO₂ content and both tensile and flexural strength. In order to perform the machine learning modeling, the TiO₂ loading for each of the composites was used as an input variable, while the tensile or flexural strengths were employed as the target output. To avoid any biased assessment in the performance of the developed models, the research data were divided into two groups, training and test, which included 80% and 20% of the data by means of a fixed random seed, respectively. Subsequently, the input feature was standardized via Z-score normalization, with the scaling parameters learned only from the training data to avoid information leakage [60].

A broad model screening phase was performed using LazyPredict 0.2.16, which included the training process for over 40 regression algorithms with the default hyperparameters. This allowed rapid identification of the most suitable model families for the dataset. Several characteristic performance metrics, such as R², adjusted R², root mean square error (RMSE), and computation time, were employed to find the robust algorithm for mechanical property prediction of the developed composite. The top performers in

the screening phase were then selected for detailed evaluation. For the prediction of the properties obtained from the flexural test, the same procedure was followed.

Four regressors, (1) random forest (2000 trees), (2) XGBoost (1000 estimators), (3) Multi-layer perceptron (4-neuron hidden layer), and (4) Gaussian process regressor using an RBF kernel with constant scaling, were trained. Afterwards, every model was evaluated using mean absolute error (MAE), mean square error (MSE), and R^2 for training and test datasets. Parity plots were developed to compare the experimental and predicted values of the tensile and flexural strength in order to assess the accuracy obtained from each model.

A 5-fold cross-validation process was also developed as a supplementary level of validation to compare how the developed models functioned when evaluated with the same data subset. In addition, Leave-One-Out Cross-Validation (LOOCV) was also employed, as it provides a nearly unbiased performance estimate, ideal for small datasets. For this purpose, the predicted values for every model were generated and displayed in overlaid parity plots, which allowed an easy and direct comparison between the different model families and provided a clear assessment of prediction stability and agreement under identical conditions. All data processing, model training, and visualization tasks were performed in Python 3.12.12 using scikit-learn libraries within a Google Colab environment.

3. Results and Discussion

3.1. Tensile Tests

Tensile stress vs. strain curves for the GFRBE composites with different TiO_2 loadings are presented in Figure 4. The main tensile properties (tensile strength, modulus of elasticity, and elongation percent at break) are summarized in Table 3, while Figure 5 illustrates the variation in tensile strength and modulus as a function of TiO_2 content. For the unreinforced GFRBE, the tensile strength, modulus of elasticity, and elongation at break were 214.43 ± 7.35 MPa, 13.11 ± 0.36 GPa, and $1.99 \pm 0.10\%$, respectively. The incorporation of TiO_2 nanoparticles enhanced the tensile performance of the composites. It can be observed that the tensile strength improved by 9.0, 12.8, and 8.6% with the addition of 0.5, 1.0, and 2.0 wt.% of the reinforcement, respectively. Accordingly, tensile modulus improved by 12, 13.6, and 5.3% for the same TiO_2 loadings. The more effective improvement at low TiO_2 loadings can be attributed to an enhanced stress transfer mechanism caused by a uniform nanoparticle dispersion [2], improved interfacial interaction between the bio-epoxy matrix and glass fibers [41], and interfacial strengthening induced by the nanoparticle addition [61]. However, although the composite including 2.0 wt.% TiO_2 still exhibited an improved mechanical resistance compared to the unreinforced GFRBE, this improvement was less pronounced than the observed at lower reinforcement contents. This behavior may be ascribed to a possible nanoparticle agglomeration at higher contents, which can reduce the homogeneity of the bio-epoxy matrix and promote localized stress concentrations [62–64]. Previous studies have reported that increasing TiO_2 nanoparticle loadings, enhanced the possibility of agglomeration due to greater van der Waals forces which might compromise the mechanical performance of the manufactured composites [46–48].

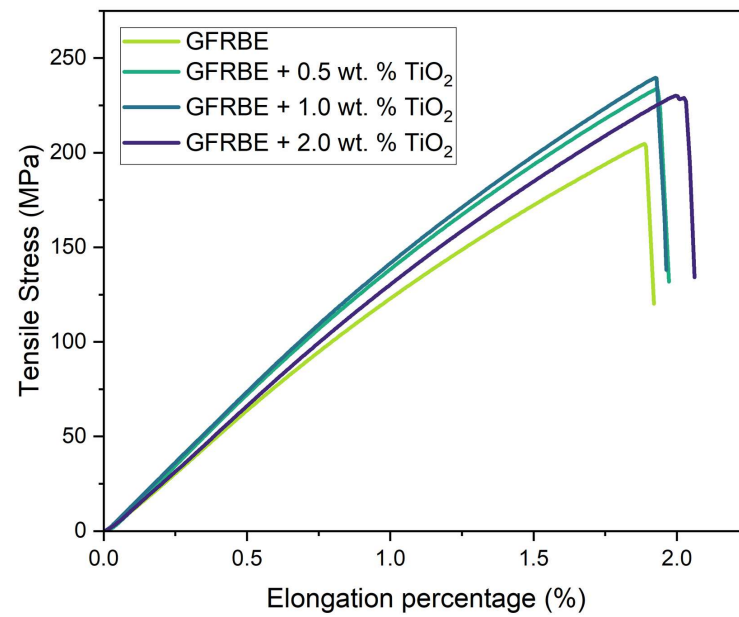


Figure 4. Tensile stress vs. elongation percentage for the GFRBE composites containing different TiO₂ loadings.

Table 3. Tensile properties of the GFRBE composites at different reinforcement loadings.

Composite Formulation	Tensile Strength (MPa)	Percent of Increment (%)	Tensile Modulus (GPa)	Percent of Increment (%)	Elongation Percent at Break (%)	Percent of Increment (%)
GFRBE	214.43 ± 7.35	--	13.11 ± 0.36	--	1.99 ± 0.10	--
GFRBE + 0.5 wt.% TiO ₂	233.65 ± 2.22	8.96	14.67 ± 0.19	11.90	2.00 ± 0.06	0.50
GFRBE + 1.0 wt.% TiO ₂	241.80 ± 3.72	12.76	14.89 ± 0.13	13.58	2.01 ± 0.03	1.01
GFRBE + 2.0 wt.% TiO ₂	232.93 ± 6.60	8.63	13.80 ± 0.29	5.26	2.05 ± 0.07	3.02

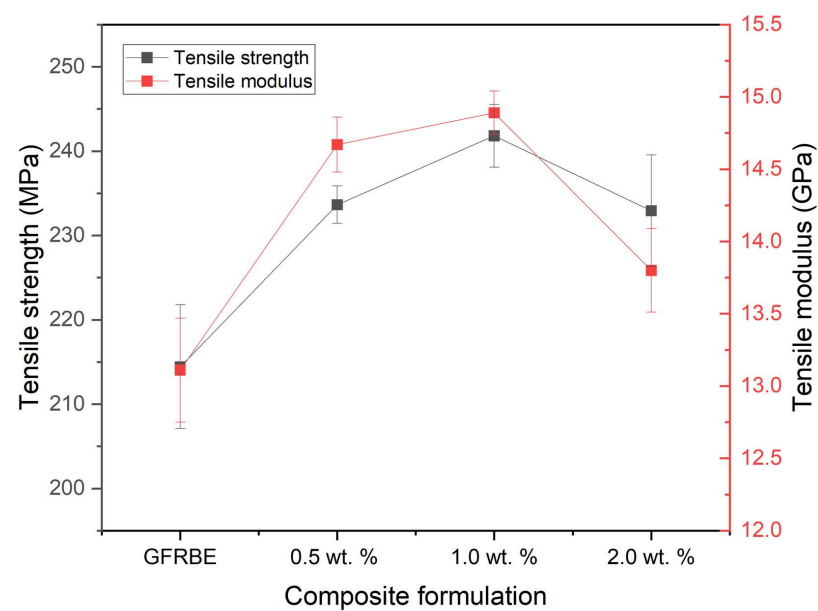


Figure 5. Tensile strength and modulus variation for GFRBE composites.

3.2. Flexural Tests

Flexural stress vs. deflection percentage curves for the GFRBE composites with various TiO_2 reinforcement loadings are portrayed in Figure 6. Additionally, Table 4 depicts the flexural strength, and flexural modulus for the composites developed in the present study. Moreover, Figure 7 visually presents how both mechanical properties changed with increasing additions of the nanoparticle reinforcement. As a baseline, the flexural properties of the GFRBE were determined to be 374.97 ± 5.40 MPa, and 14.54 ± 0.17 GPa for the flexural strength and flexural modulus, respectively. After the addition of different TiO_2 nanoparticle loadings, a distinguishable flexural strength reinforcement effect can be observed for composites containing 0.5, 1.0, and 2.0 wt.% of the nanoparticle reinforcement due to improvements of 11.5, 17.5, and 19.3%, respectively. Correspondingly, the flexural modulus increased by 17.6, 23.3, and 39.6% for the same filler loadings. This composite flexural property behavior produced can be attributed to the high surface area of the TiO_2 nanoparticles which produced an increased interface, allowing an effective energy absorption process during the composite deformation [65]. In addition, the strengthening mechanism caused by the addition of the TiO_2 reinforcement can be ascribed to a favorable interlocking effect between the glass fiber, the bio-epoxy resin matrix and the reinforcement [65,66]. Similarly, it has been previously observed that the addition of nanoparticle reinforcement prevented slip mechanisms between the fiber and the matrix which delayed crack propagation in the composites [67].

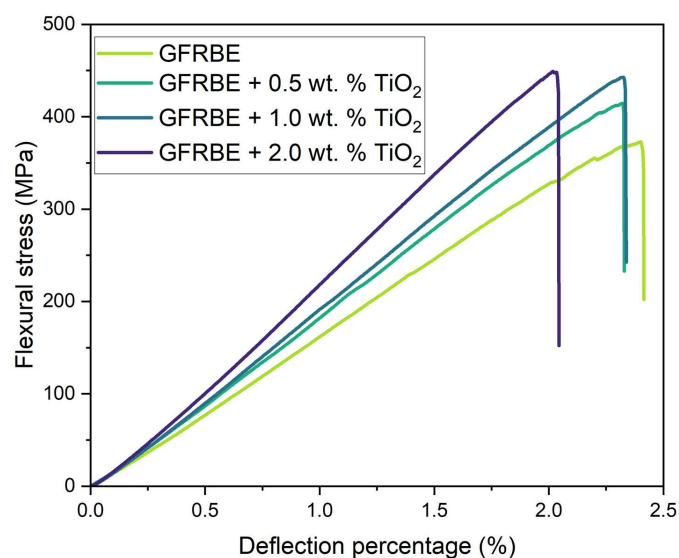


Figure 6. Flexural stress vs. deflection percentage for the GFRBE composites containing different TiO_2 loadings.

Table 4. Flexural properties of the GFRBE composites at different reinforcement loadings.

Composite Formulation	Flexural Modulus (GPa)	Percent of Increment (%)	Flexural Strength (MPa)	Percent of Increment (%)
GFRBE	14.54 ± 0.17	--	374.97 ± 5.40	--
GFRBE + 0.5 wt.% TiO_2	17.10 ± 0.10	17.61	418.24 ± 4.80	11.54
GFRBE + 1.0 wt.% TiO_2	17.93 ± 0.05	23.31	440.54 ± 2.80	17.49
GFRBE + 2.0 wt.% TiO_2	20.30 ± 0.12	39.62	447.32 ± 3.69	19.29

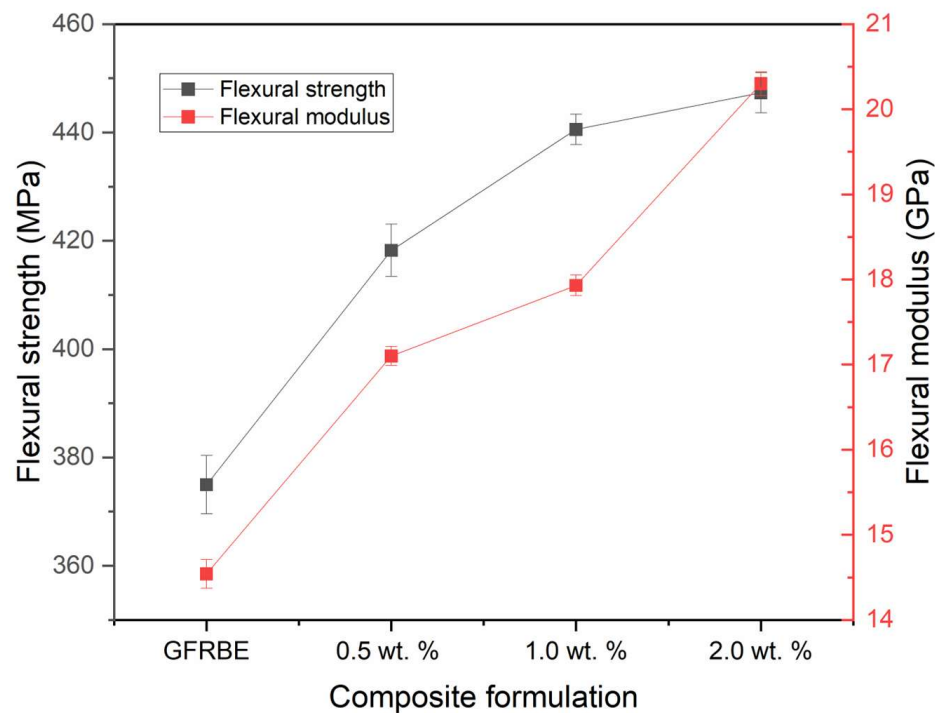


Figure 7. Flexural strength and modulus variation for GFRBE composites.

3.3. SEM of Fractured Specimens

Figure 8 depicts SEM micrographs ($1300\times$ magnification) of tensile fractured surfaces for the GFRBE at increasing TiO_2 loadings. For the unreinforced GFRBE (Figure 8a), the fractured surface appears relatively smooth, indicating a near brittle fracture, which eases fiber debonding [68]. With the incorporation of TiO_2 nanoparticle (Figure 8b–d), different fractured zones show an increased surface roughness, which is indicative of crack pinning mechanisms that enhance energy dissipation while improving the mechanical performance of the composite [2,32,47,68]. Different failure features are observed in the SEM micrographs, including fiber breakage, fiber delamination, and fiber pullout, which reveals an adequate load transfer from the bio epoxy resin matrix to the glass fiber [69–71]. Similar observations were reported by Abebe et al. [72], who concluded that nanoparticle reinforcement delayed the polymer matrix cracking and reduced premature fiber debonding. However, other studies have observed that higher reinforcement concentrations can lead to nanoparticle clustering or agglomeration which can result in uneven stress distribution resulting in a reduced reinforcing effect of the nanoparticles [69,73]. Figure 9 illustrates the SEM micrographs ($1300\times$ magnification) for the flexural fractured specimens with increasing nanoparticle reinforcement loadings. Comparable to the tensile fractured specimens, flexural fractured specimens depict the same modified surface pattern when the nanoparticle reinforcements were added which act as pinning sites to suppress rapid crack propagation when a nanoparticle is reached. In addition, the fractured fibers' orientations slightly differ from the tensile specimens as the applied load configurations are different, as observed in Figure 9. This effect might be attributed to the compressive and tensile stresses that are applied during a flexural test and the stabilized arrangement used for the composite fabrication [73–75].

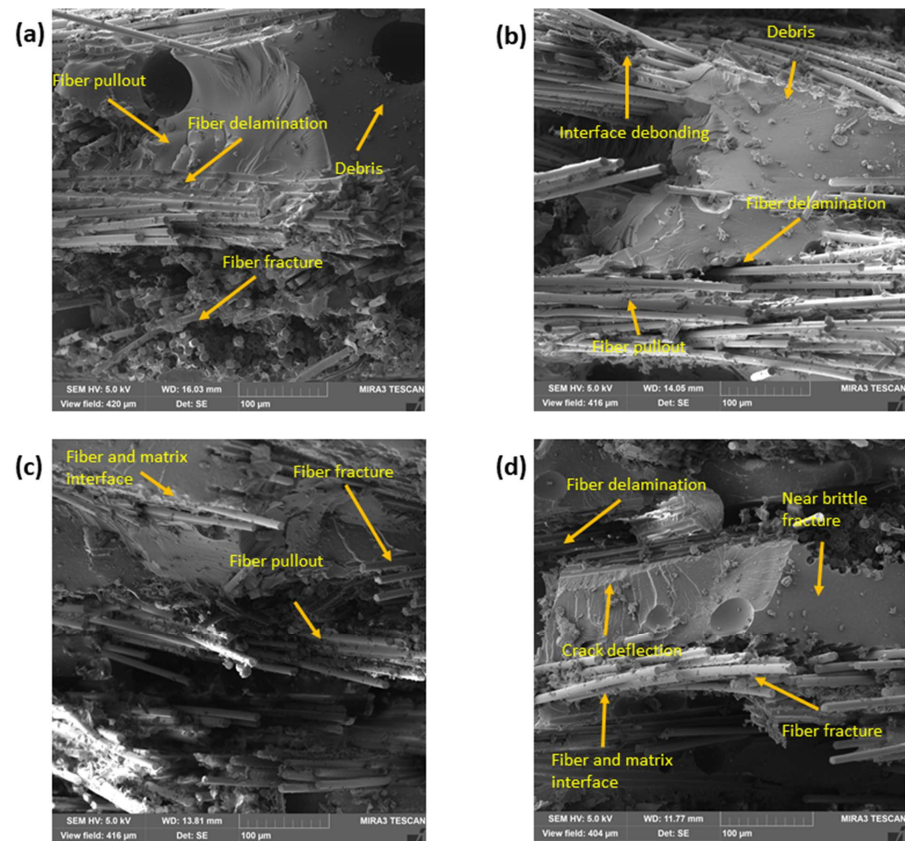


Figure 8. SEM micrographs of tensile fractured specimens of (a) GFRBE, (b) GFRBE including 0.5 wt.% of TiO_2 , (c) GFRBE including 1.0 wt.% of TiO_2 , and (d) GFRBE including 2.0 wt.% of TiO_2 .

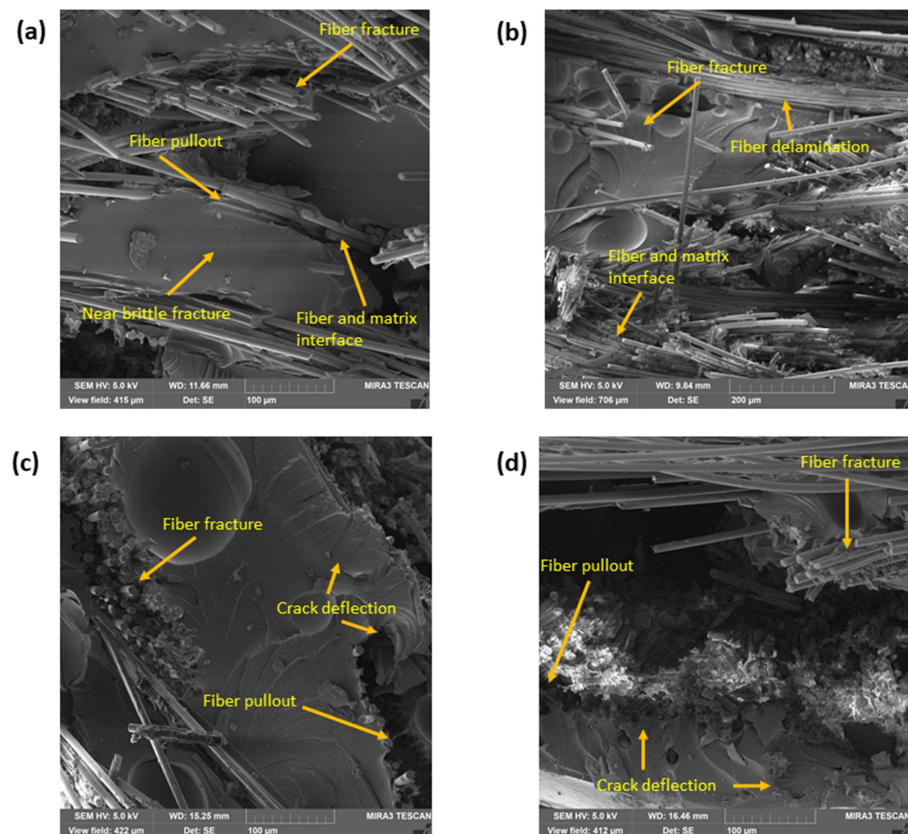


Figure 9. SEM micrographs of flexural fractured specimens of (a) GFRBE, (b) GFRBE including 0.5 wt.% of TiO_2 , (c) GFRBE including 1.0 wt.% of TiO_2 , and (d) GFRBE including 2.0 wt.% of TiO_2 .

3.4. Nanoparticle Distribution in the Matrix

Figure 10 depicts the elemental mapping images and EDS plots for the GFRBE composites with various TiO_2 nanoparticle loadings, while Table 5 presents the elemental compositions by weight percentage within the polymer matrix. The elemental distribution of the neat bio-epoxy resin, shown in the left column, exhibited a homogeneous distribution of carbon and oxygen, which are the main elemental components of the bio-epoxy resin matrix, and the absence of TiO_2 particles. In contrast, the columns corresponding to the composites containing 0.5, 1.0, and 2.0 wt.% of the reinforcement showed a consistent distribution of the TiO_2 nanoparticles, corroborated through their respective titanium mapping. These observations suggest high effectiveness in the process used to disperse the reinforcement within the polymer matrix prior to the vacuum-assisted lamination process.

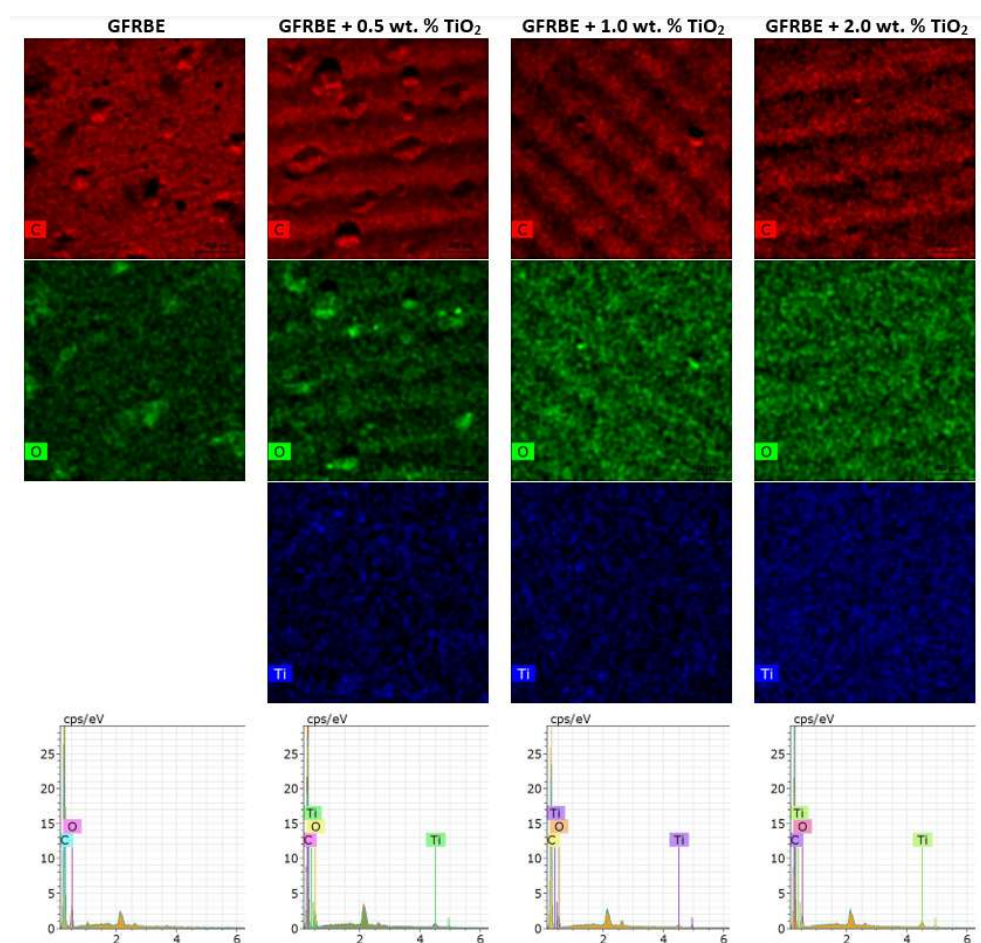


Figure 10. Elemental mapping images and EDS plots for GFRBE at different nanoparticle formulations.

Table 5. Elemental distribution for the different matrix formulations.

Composite Formulation	C (wt.%)	O (wt.%)	Ti (wt.%)
GFRBE	80.40	19.60	—
GFRBE + 0.5 wt.% TiO_2	79.70	19.90	0.40
GFRBE + 1.0 wt.% TiO_2	78.90	20.30	0.80
GFRBE + 2.0 wt.% TiO_2	80.30	18.10	1.60

3.5. FT-IR Spectroscopy

Cured bio-epoxy resin spectrum, illustrated in Figure 11.a, exhibits a broad absorption band at circa 3360 cm^{-1} which is indicative of the presence of hydroxyl group stretching ($-\text{OH}$) [6,76]. In addition, two characteristic peaks of C-H stretching from epoxy groups

can be observed between 3000 and 2800 cm^{-1} [77]. Another absorption band can be distinguished at around 1730 cm^{-1} which can be attributed to carbonyl and carboxyl group vibration [78]. A group of peaks can be found between 1650 and 1500 cm^{-1} which are attributed to C=C bond vibration present in aromatic groups in epoxy resin [79]. The absorption band found at around 1230 cm^{-1} and 935 cm^{-1} are indicative of the symmetric vibration of epoxy ring and the C-O-R bond of ether [6,79]. A relatively high peak can be observed at around 820 cm^{-1} which is assigned to the vibration of oxirane rings [80,81]. After the addition of the TiO_2 nanoparticle reinforcement, a slight attenuation can be observed in the broad band around 3360 cm^{-1} due to the presence of O-H vibration from moisture usually found in TiO_2 [69,82]. In the same way, the peak found at around 1024 cm^{-1} is modified due to the OH out-of-plane vibration. Finally, a minor peak can be observed for the FT-IR spectra of the bio-epoxy resin after the addition of the TiO_2 reinforcement (Figure 11b–d) at a wavenumber of around 660 cm^{-1} which is indicative of the vibration of O-Ti-O bond [69,82].

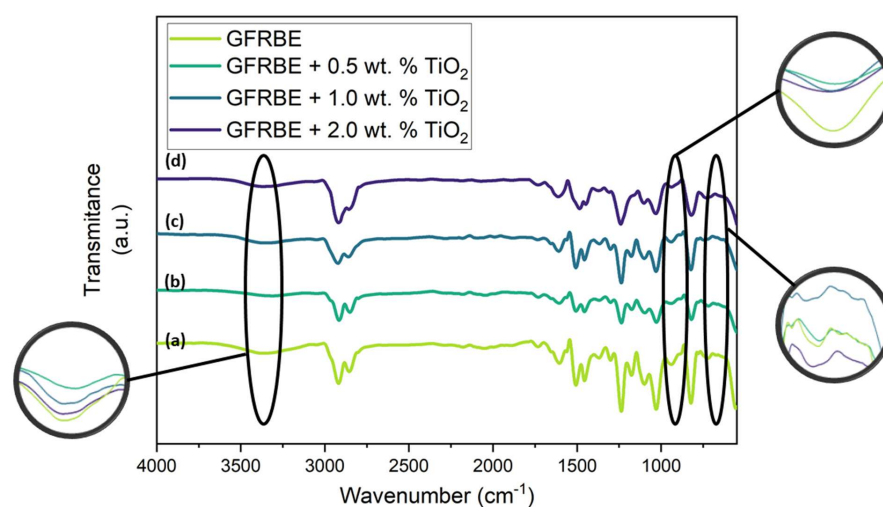


Figure 11. FT-IR spectra of (a) GFRBE, (b) GFRBE including 0.5 wt.% of TiO_2 , (c) GFRBE including 1.0 wt.% of TiO_2 , and (d) GFRBE including 2.0 wt.% of TiO_2 .

3.6. Machine Learning Predictions

An initial model screening was performed using LazyPredict which revealed clear differences in how well the various regression families captured the nonlinear relationship between TiO_2 loading and the ultimate strength measured in tensile and flexural tests. In addition, the initial screening revealed that the models that rely on linear or strongly constrained functional forms failed to successfully follow the material behavior in both tensile and flexural datasets. On the other hand, it was observed that nonlinear approaches, particularly ensemble-based and kernel-based regressors, displayed a consistent ability to explain the obtained data. The analysis of the R^2 values applied to the evaluated algorithms revealed that most of the best performing models relayed on tree-based ensembles, gradient boosting techniques, and Gaussian process regressors.

This trend indicates that the material's mechanical response is dominated by nonlinear effects, which are more effectively captured by flexible, non-parametric learning methods (Figure 12a). The automated comparison also helped identify regressors that were unstable or incompatible with single-feature datasets, such as Orthogonal Matching Pursuit and LightGBM under default configurations, further reinforcing the importance of algorithm selection when modeling low-dimensional materials data.

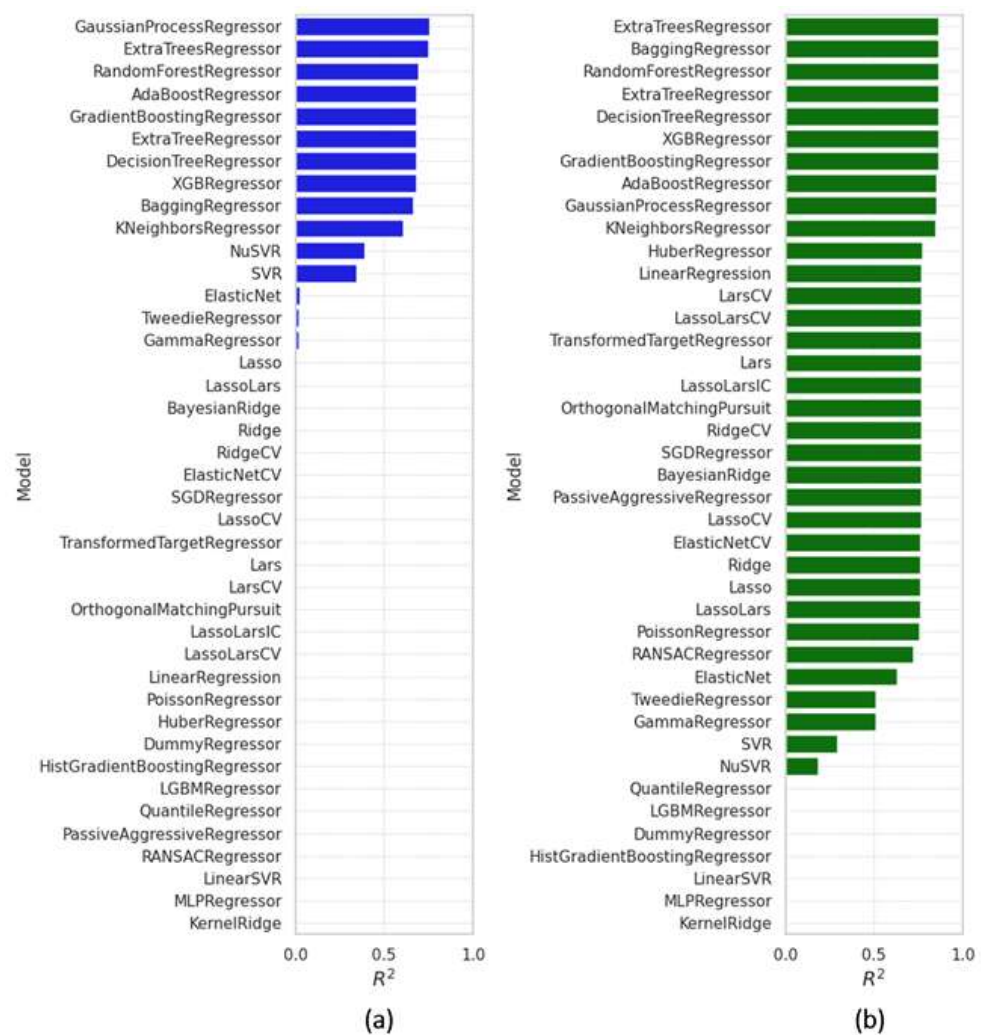


Figure 12. Comparison of the performance of different machine learning models using LazyPredict for ultimate strength prediction in (a) tensile tests and (b) flexural tests.

For the flexural dataset, the initial screening allowed to rank the regression families in terms of their ability to describe the flexural behavior of the developed composites. It could be differentiated that linear and regularized models exhibited a limited predictive performance. Conversely, ensemble approaches, such as ExtraTreesRegressor, BaggingRegressor, RandomForestRegressor, and GradientBoostingRegressor, achieved the highest R^2 values and minimized prediction errors. These results suggest that the flexural strengths of the developed composites are commanded through nonlinear reinforcement mechanisms, such as nanoparticle-driven matrix stiffening and changes in interfacial shear transfer, which are more effectively captured by tree-based and other non-parametric models that adaptively partition the feature space. In addition, Poisson-Regressor, DummyRegressor, and certain spline-based methods obtained a poor prediction ability given their limited suitability for a dataset with a single input and a monotonic response (Figure 12b).

Random forest and gradient boosting are often more accurate for predicting mechanical properties because they can naturally capture the nonlinear and interacting mechanisms that control material behavior [83,84]. Properties such as strength or stiffness do not follow simple linear trends, but instead result from the combined effects of microstructure, reinforcement efficiency, defects, and processing variability. The developed process outlined that tree-based models possess a high adaptability to the underlying complexity through avoiding the assumption of predefined equations into different data regions. Moreover, random forest models achieved a strong prediction ability by averaging many indepen-

dent trees, which reduced noise and overfitting, while gradient boosting improved their accuracy by a gradual correction of the precision error and capturing subtle trends. These advantages allowed such methods to become robust with suitable approaches to predict complex responses observed in composite materials.

Following the initial screening, the four selected regressors—random forest, XGBoost, Multi-layer perceptron (MLP), and Gaussian process regression (GPR)—were trained in detail and evaluated using identical training–testing partitions and consistent error metrics. All models successfully learned the monotonic increase in tensile strength of the GFRBE composites with increasing TiO_2 content but differed in their ability to reproduce finer variations within the 200–260 MPa range. The parity plots generated for each regressor illustrated these differences clearly: tree-based ensembles provided robust trend capture, the neural network yielded smoother but less precise predictions, and the Gaussian process model provided tightly clustered predictions along the ideal fit line (Figure 13). Across all regressors, the results confirmed that the one-dimensional feature space still contained sufficient information to generate accurate predictions of mechanical performance when paired with an appropriate nonlinear model.

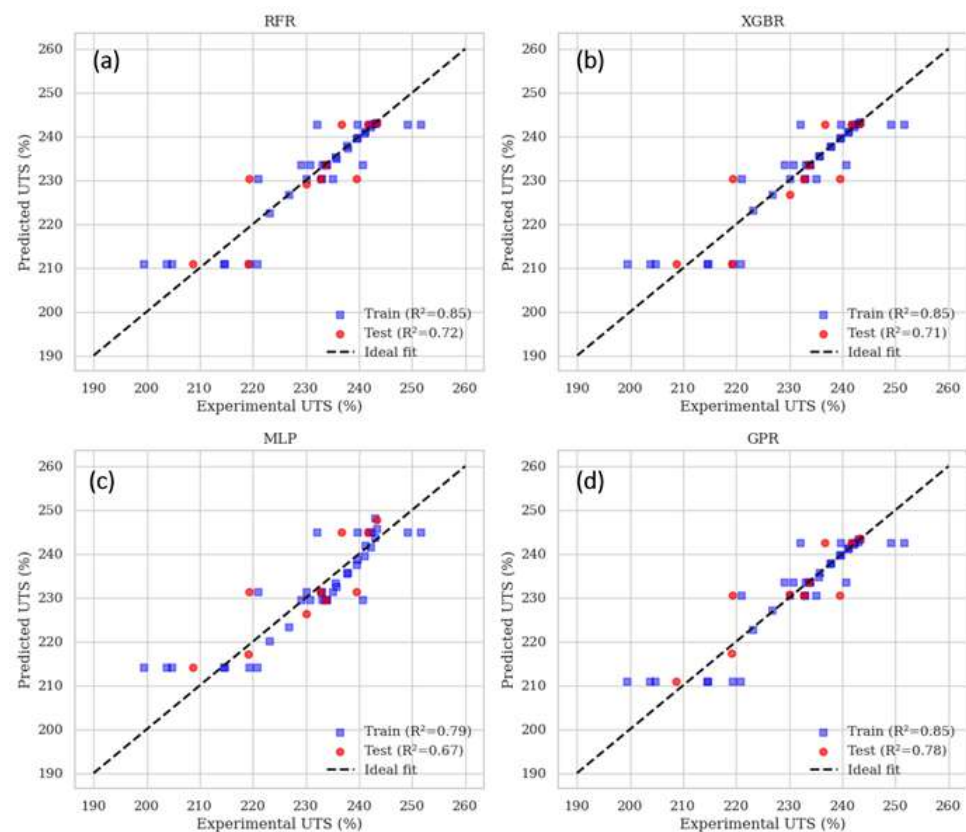


Figure 13. Model performance evaluation for predicting tensile strength of (a) random forest regressor (RFR), (b) Extreme gradient boosting regressor (XGBR), (c) Multi-layer perceptron (MLP), and (d) Gaussian process regressor (GPR).

Table 6 listed the metrics obtained for the training, testing, and cross-validation (CV) procedures. On the one hand, the metrics associated with errors (MAE, MSE) show similar values, which indicates the consistency of the predicted results, while the R^2 values show a significant decrease. LOOCV evaluation shows consistent error magnitudes and stable R^2 behavior comparable to the reported k-fold validation results, confirming that the learned relationships are not artifacts of data partitioning. The algorithm that best fits the data is GPR.

Tree-based ensembles (RFR and XGBoost) obtained adequate R^2 values around 0.71–0.72, while the MLP exhibited a minor accuracy due to the limited size of the dataset. The Gaussian process regressor obtained the best predictive response with an R^2 of 0.78 and the lowest error metrics among all models, which confirmed their suitability low-dimensional, nonlinear mechanical property prediction.

Table 6. Comparative evaluation of the performance of the ML algorithms employed to predict the tensile strength for the training, testing, and cross-validation dataset.

Model	Training			Testing		
	MAE	MSE	R^2	MAE	MSE	R^2
RFR	2.963	22.087	0.847	4.122	32.163	0.723
XGBR	2.963	22.047	0.846	4.387	33.211	0.714
MLP	4.082	29.701	0.793	5.274	37.781	0.674
GPR	3.036	22.080	0.846	3.456	25.808	0.777
	Cross-validation			LOOCV		
	MAE	MSE	R^2	MAE	MSE	R^2
RFR	3.580	29.375	0.482	3.840	31.496	0.773
XGBR	4.027	31.754	0.465	4.119	32.407	0.766
MLP	4.448	36.471	0.284	4.705	37.272	0.731
GPR	3.414	27.635	0.504	3.526	29.519	0.787

The implemented cross-validation process revealed coherence among the three non-linear regressors and a consistent similarity between predicted and experimental tensile strength values. Although each model approach converses, the obtained predictions converged for all the TiO_2 reinforcement loadings, which indicated stability of the learned relationships and accuracy to reproduce the modeling pattern. The GPR models obtained the most consistent and smooth prediction profile, while RFR and XGBR provided more discrete responses given their tree structure.

Regarding the results obtained in the flexural test dataset, RFR and XGBR further confirmed the advantages that ensemble methods provide for this type of problem. Both random forest and XGBoost produced accurate predictions that followed the experimental flexural strength values, with a strong alignment along the prediction plots (Figure 14).

Their acceptable performance indicates their ability to predict abrupt and smooth nonlinearities produced by microstructural changes given the increasing TiO_2 concentration. In contrast, the shallow MLP, configured with a single hidden layer of four neurons, showed higher dispersion and weaker generalization on the test set, consistent with the limited dataset size and the sensitivity of neural networks to small sample regimes.

The cross-validation analysis (Table 7), in which all trained models were evaluated on the same held-out test set, confirmed both the robustness of the ensemble-based approaches and the stability of the learned structure–property relationship. Overall, the results show that tree-based ensemble regressors delivered the most reliable and physically meaningful predictions of flexural strength in TiO_2 -modified composites, as they are able to capture the nonlinear mechanical response introduced by nanoparticle reinforcement.

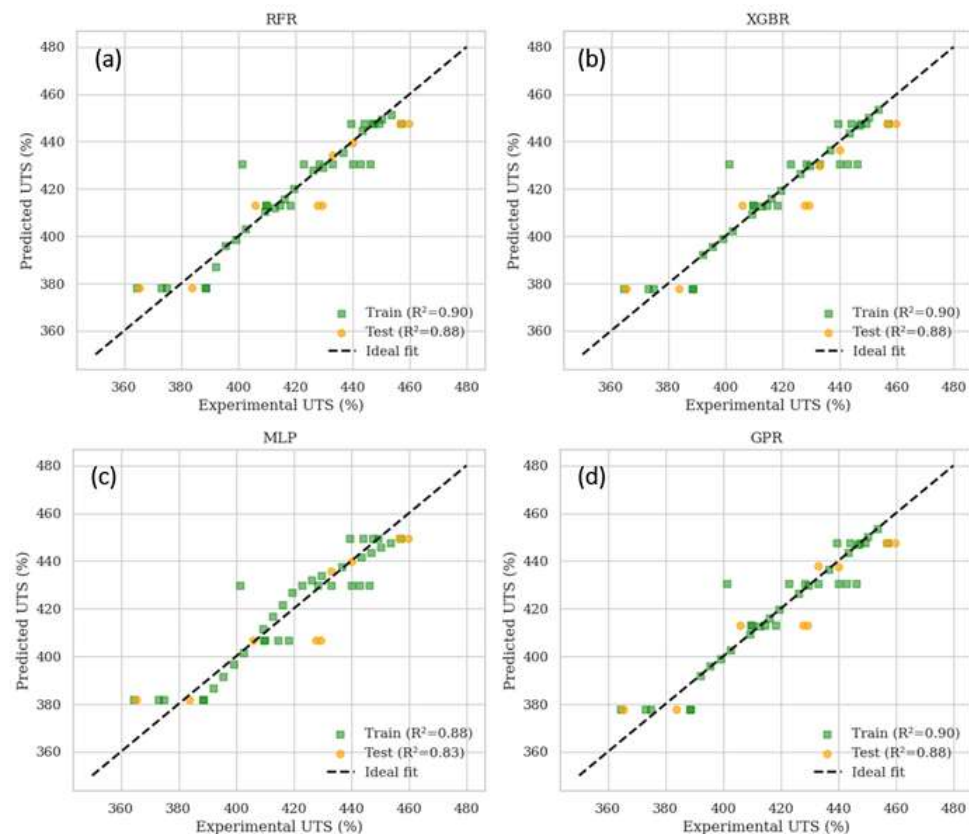


Figure 14. Model performance evaluation for predicting the ultimate strength in the flexural test of (a) random forest regressor (RFR), (b) Extreme gradient boosting regressor (XGBR), (c) Multi-layer perceptron (MLP), and (d) Gaussian process regressor (GPR).

Table 7. Comparative evaluation of the performance of the ML algorithms employed to predict the flexural strength for the training, testing, and cross-validation dataset.

Model	Training			Testing		
	MAE	MSE	R ²	MAE	MSE	R ²
RFR	4.915	59.043	0.902	8.863	107.454	0.880
XGBR	4.393	57.822	0.904	9.429	109.110	0.878
MLP	6.538	72.772	0.879	9.235	154.505	0.827
GPR	4.442	57.833	0.904	9.447	109.923	0.877
	Cross-validation			LOOCV		
	MAE	MSE	R ²	MAE	MSE	R ²
RFR	7.933	127.321	0.625	7.296	95.441	0.856
XGBR	8.716	136.947	0.606	7.743	97.729	0.852
MLP	8.293	118.957	0.684	7.588	98.251	0.852
GPR	10.033	219.530	0.544	7.816	101.362	0.847

The metrics obtained in different datasets for predicting flexural strength show more accurate results compared to tensile testing. Predicting flexural response is often easier than predicting tensile response in composite materials because flexural behavior is typically more stable, less sensitive to defects, and governed by a more localized set of deformation mechanisms [85,86]. In flexural testing, the stress state is non-uniform and dominated by a combination of tension and compression, with failure often initiating gradually in the most

highly stressed region. This tends to smooth out the influence of local flaws, voids, or weak interfaces, leading to more repeatable and less noisy data [85,87].

In contrast, tensile response is highly sensitive to the weakest link in the material, such as micro voids, imperfect bonding, or slight variations in fiber or particle distribution [88]. Therefore, small defects or voids can lead to an early failure which may possibly produce a certain scattering in the experimental dataset which complicates the learning stage of the model development. From a modeling perspective, the lower noise and more monotonic trends typical of flexural tests eases the property prediction, when using algorithms that depend on structure–property relationships. Collectively, the results demonstrate that machine learning regressors can capture both the global trend and local variations in tensile and flexural strength behavior in nanoparticle-reinforced composites, confirming the suitability of data-driven approaches for mechanical property prediction even in modest datasets.

4. Conclusions

In this study, the characterization of a GFRBE composite reinforced with TiO₂ nanoparticles was developed. The main conclusions can be summarized as follows:

- The addition of TiO₂ nanoparticles to the bio-epoxy resin allowed to improve the mechanical resistance, both tensile and flexural, for the totality of composite formulations developed. The composite containing 1 wt.% of the reinforcement displayed the best tensile property results with an improvement of 13.6% for tensile strength and 12.8% for tensile modulus when compared to the unreinforced GFRBE. Similarly, the composite with 2 wt.% reinforcement obtained the best flexural response with an improvement of 19.3% and 39.6% for the flexural strength and modulus, respectively. The obtained results confirm that controlled nanoparticle addition can significantly improve load transfer and improve the overall mechanical resistance on GFRPs.
- SEM micrographs demonstrate that the improvement in mechanical performance of the composites were associated with the modification of the fracture surfaces, which allowed crack pinning mechanisms to develop.
- EDS mapping determined the manufacturing process used in this study was adequate to obtain a homogenous TiO₂ nanoparticle dispersion in composite matrix.
- Across both mechanical tests—tension and flexure—the ML models consistently showed that the mechanical response of TiO₂-reinforced composites is governed by nonlinear structure–property relationships. In both datasets, ensemble regressors such as Random Forest, ExtraTrees, and XGBoost demonstrated the highest predictive stability, confirming that nanoparticle-induced mechanisms (matrix stiffening, enhanced interfacial bonding, and restricted deformation) produced trends that are not adequately captured by linear or low-complexity models.
- The agreement among the top-performing regressors in both tensile and flexural predictions demonstrates that supervised machine learning can reliably characterize and generalize the mechanical behavior of TiO₂-modified composites, even with modest dataset sizes. The cross-validated consistency observed in both studies indicated that the learned relationships are robust rather than model-dependent, providing strong evidence that data-driven methods are suitable for predicting and optimizing multiple mechanical properties in nanoparticle-reinforced polymer composites.
- Overall, this study demonstrates that the integration of a thorough characterization process of materials accompanied by machine learning prediction models constitutes a powerful tool to provide an optimization for composite materials' formulations. Undoubtedly, the observed interaction between machine learning prediction and

experimental validation can become the foundation for keen design strategies, which may optimize conventional trial and error experimentation.

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Data Availability Statement: The data presented in this study are available on request from the corresponding authors. The data are not publicly available because the raw and processed data required to reproduce these findings is part of an ongoing study. The ML dataset is available at <https://github.com/GermanOmar/Composite2> (accessed on 20 February 2026).

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Abbreviations

The following abbreviations are used in this manuscript:

GFRP	Glass fiber reinforced polymer
SEM	Scanning electron microscopy
EDS	Energy-dispersive spectroscopy
ML	Machine learning
TS	Tensile strength
GO	Graphene oxide
G-SVM	Gaussian support vector
ANN	Artificial neural networks
BFRP	Basalt fiber reinforced polymer
GFRBE	Glass fiber reinforced bio-epoxy
ASTM	American Society for Testing and Materials
FT-IR	Fourier-transform infrared spectroscopy
FS	Flexural strength
RMSE	Root mean square error
GPR	Gaussian process regression
RFR	Random forest regression
MSE	Mean square error
MAE	Mean absolute error
MLP	Multi-layer perceptron

References

1. Hsissou, R.; Seghiri, R.; Benzekri, Z.; Hilali, M.; Rafik, M.; Elharfi, A. Polymer composite materials: A comprehensive review. *Compos. Struct.* **2021**, *262*, 113640. [\[CrossRef\]](#)
2. Navas-Pinto, W.; Cree, D.E.; Wilson, L.D.; Barrionuevo, G.O.; Sánchez-Sánchez, X. Mechanical response of bio-epoxy resin composites reinforced with graphene oxide: Machine learning approach for property prediction. *Funct. Compos. Struct.* **2025**, *7*, 035002. [\[CrossRef\]](#)
3. Radzi, F.S.M.; Ali, A.; Anwar, U.K.; Khalina, A.; Mansor, M.R.; Sani, M.S.M. Effect of reinforcement of Alkaline-treated sugar palm/bamboo/kenaf and fibreglass/Kevlar with polyester hybrid biocomposites: Mechanical, morphological, and water absorption properties. *J. Mater. Res. Technol.* **2023**, *24*, 4190–4202. [\[CrossRef\]](#)
4. Xu, Y.; Chung, D.D.L.; Mroz, C. Thermally conducting aluminum nitride polymer-matrix composites. *Compos. Part A Appl. Sci. Manuf.* **2001**, *32*, 1749–1757. [\[CrossRef\]](#)
5. Zhou, Y.; He, J.; Fu, H.; Zhou, X. Fabrication and characterization of aluminum nitride polymer matrix composites with high thermal conductivity and low dielectric constant for electronic packaging. *Mater. Sci. Eng. B* **2012**, *177*, 892–896. [\[CrossRef\]](#)
6. Loeffen, A.; Cree, D.E.; Sabzevari, M.; Wilson, L.D. Effect of Graphene Oxide as a Reinforcement in a Bio-Epoxy Composite. *J. Compos. Sci.* **2021**, *5*, 91. [\[CrossRef\]](#)
7. Chen, R.S.; Muhammad, Y.H.; Ahmad, S. Physical, mechanical and environmental stress cracking characteristics of epoxy/glass fiber composites: Effect of matrix/fiber modification and fiber loading. *Polym. Test.* **2021**, *96*, 107088. [\[CrossRef\]](#)
8. Morampudi, P.; Namala, K.K.; Gajjala, Y.K.; Barath, M.; Prudhvi, G. Review on glass fiber reinforced polymer composites. *Mater. Today Proc.* **2021**, *43*, 314–319. [\[CrossRef\]](#)
9. Yang, G.; Park, M.; Park, S.J. Recent progresses of fabrication and characterization of fibers-reinforced composites: A review. *Compos. Commun.* **2019**, *14*, 34–42. [\[CrossRef\]](#)
10. Patel, A.; Kravchenko, O.; Manas-Zloczower, I. Effect of Curing Rate on the Microstructure and Macroscopic Properties of Epoxy Fiberglass Composites. *Polymers* **2018**, *10*, 125. [\[CrossRef\]](#)
11. Megahed, M.; Fathy, A.; Morsy, D.; Shehata, F. Mechanical Performance of glass/epoxy composites enhanced by micro- and nanosized aluminum particles. *J. Ind. Text.* **2021**, *51*, 68–92. [\[CrossRef\]](#)
12. Sajan, S.; Selvaraj, D.P. A review on polymer matrix composite materials and their applications. *Mater. Today Proc.* **2021**, *47*, 5493–5498. [\[CrossRef\]](#)
13. Haeri, Z.; Ramezanzadeh, B.; Ramezanzadeh, M. Recent progress on the metal-organic frameworks decorated graphene oxide (MOFs-GO) nano-building application for epoxy coating mechanical-thermal/flame-retardant and anti-corrosion features improvement. *Prog. Org. Coat.* **2022**, *163*, 106645. [\[CrossRef\]](#)
14. Jin, K.; Luo, H.; Wang, Z.; Wang, H.; Tao, J. Composition optimization of a high-performance epoxy resin based on molecular dynamics and machine learning. *Mater. Des.* **2020**, *194*, 108932. [\[CrossRef\]](#)
15. Zhou, S.; Zhang, W.; Wang, C.; Zhang, J. Highly improving the mechanical and thermal properties of epoxy resin via blending with polyetherketone cardo. *Compos. Commun.* **2019**, *13*, 80–84. [\[CrossRef\]](#)
16. Ke, J.; Li, X.; Liao, H.; Wang, J. Critical transition of epoxy resin from brittleness to toughness by incorporating CO₂-sourced cyclic carbonate. *J. CO₂ Util.* **2018**, *26*, 302–313. [\[CrossRef\]](#)
17. Luo, J.; Zhang, Y.; Wang, Y.; Li, Y.; Zhao, Y. Simultaneous optimization of the thermal conductivity and mechanical properties of epoxy resin composites through PES and AgNP functionalized BNs. *Compos. B Eng.* **2023**, *248*, 110373. [\[CrossRef\]](#)
18. Wang, H.; Lu, R.; Li, L. Strong, tough, and thermally conductive nacre-inspired boron nitride nanosheet/epoxy layered nanocomposites. *Nano Res.* **2024**, *17*, 820–828. [\[CrossRef\]](#)
19. Azman, M.A.; Asyraf, M.R.M.; Khalina, A.; Petrú, M.; Ruzaidi, M.S.; Sapuan, S.M.; Wan Aman, V.G.; Ishak, M.R. Natural Fiber Reinforced Composite Material for Product Design: A Short Review. *Polymers* **2021**, *13*, 1917. [\[CrossRef\]](#)
20. Pappa, C.; Feghali, E.; Vanbroekhoven, K.; Triantafyllidis, K.S. Recent advances in epoxy resins and composites derived from lignin and related bio-oils. *Curr. Opin. Green Sustain. Chem.* **2022**, *38*, 100687. [\[CrossRef\]](#)
21. Ebrahimnezhad-Khaljiri, H.; Ghadi, A. Recent advancement in synthesizing bio-epoxy nanocomposites using lignin, plant oils, saccharides, polyphenols, and natural rubbers: A review. *Int. J. Biol. Macromol.* **2024**, *256*, 128041. [\[CrossRef\]](#) [\[PubMed\]](#)
22. Hidalgo, P.; Salgado, L.; Ibacache, N.; Hunter, R. Influence of Biochar and Bio-Oil Loading on the Properties of Epoxy Resin Composites. *Polymers* **2023**, *15*, 1895. [\[CrossRef\]](#) [\[PubMed\]](#)
23. Navas-Pinto, W.; Pila-Quishpe, M.; Velasquez, S.; Valadez-Gonzalez, A.; Espinoza-Santos, C. Comprehensive characterization of Yanchama (*Poulsenia Armata*) fiber cloth from the Ecuadorian Amazon region: Towards sustainable reinforcement materials. *J. Mater. Res. Technol.* **2025**, *38*, 6053–6062. [\[CrossRef\]](#)
24. Birleanu, C.; Udrioiu, R.; Cioaza, M.; Pustan, M.; Paul, B.; Vilau, C. The Effect of Fiber Weight Fraction on Tribological Behavior for Glass Fiber Reinforced Polymer. *Polymers* **2025**, *17*, 720. [\[CrossRef\]](#)
25. Matykiewicz, D. Hybrid Epoxy Composites with Both Powder and Fiber Filler: A Review of Mechanical and Thermomechanical Properties. *Materials* **2020**, *13*, 1802. [\[CrossRef\]](#)

26. Chinnasamy, V.; Subramani, S.P.; Palaniappan, S.K.; Mylsamy, B.; Aruchamy, K. Characterization on thermal properties of glass fiber and kevlar fiber with modified epoxy hybrid composites. *J. Mater. Res. Technol.* **2020**, *9*, 3158–3167. [\[CrossRef\]](#)
27. Colombo, M.; Pinho, S.T.; Greenhalgh, E.S. Interfacial Chemistry Behind Damage Monitoring in Glass Fiber-Reinforced Composites: Attempts and Perspectives. *Polym. Compos.* **2026**, *47*, 2925–2954. [\[CrossRef\]](#)
28. Wang, Z.; Zhao, X.L.; Xian, G.; Wu, G.; Raman, R.K.S.; Al-Saadi, S. Durability study on interlaminar shear behaviour of basalt-, glass- and carbon-fibre reinforced polymer (B/G/CFRP) bars in seawater sea sand concrete environment. *Constr. Build. Mater.* **2017**, *156*, 985–1004. [\[CrossRef\]](#)
29. Januš, O.; Girgle, F.; Rozsypalová, I.; Kostiha, V.; Štěpánek, P. Influence of test parameters on the interlaminar shear strength of FRP bars. *Compos. Struct.* **2022**, *299*, 116061. [\[CrossRef\]](#)
30. Alsaadi, M.; Ugla, A.A.; Erklig, A. A comparative study on the interlaminar shear strength of carbon, glass, and Kevlar fabric/epoxy laminates filled with SiC particles. *J. Compos. Mater.* **2017**, *51*, 2835–2844. [\[CrossRef\]](#)
31. Gao, H.; Zhang, J.; Li, Y.; Zhao, Y. Enhanced interfacial adhesion in glass fiber fabric/epoxy composites employing fiber surface treatment with aminosilane-functionalized graphene oxide. *Text. Res. J.* **2021**, *91*, 790–801. [\[CrossRef\]](#)
32. Megahed, M.; Youssef, S.M.; Ali-Eldin, S.S.; Agwa, M.A. Upgraded Mechanical Properties of Diluent Nano-filled Glass/Epoxy Composites Fabricated by Vacuum Assisted Resin Infusion. *Fibers Polym.* **2021**, *22*, 1063–1081. [\[CrossRef\]](#)
33. Kose, H.; Bayar, I.; Ergün, R.K. Experimental optimization of CuO and MgO hybrid nanoparticle reinforcement ratios to enhance fatigue life of GFRP composites. *Polym. Compos.* **2024**, *45*, 11125–11137. [\[CrossRef\]](#)
34. Nawaz, H.; Abbas, N.; Arshad, M.A.; Ahmad, S.; Shakoor, A. Polymer Nanocomposites Based on TiO₂ as a Reinforcing Agent: An Overview. *Adv. Eng. Mater.* **2022**, *24*, 2200844. [\[CrossRef\]](#)
35. Singh, S.; Kumar, V. Investigation on the titanium dioxide nanoparticles infusion on tensile behavior of optimized stacking pattern of hybrid fiber-reinforced polymer composite. *Adv. Compos. Mater.* **2025**, *34*, 901–927. [\[CrossRef\]](#)
36. Wang, N.; Zhang, J.; Li, Y.; Zhao, Y. Preparation and Characterization of Polyimide/TiO₂ Nano-composites for Non-lethal Weapon. *J. Phys. Conf. Ser.* **2021**, *1759*, 012005. [\[CrossRef\]](#)
37. Liu, Z.; Bobrinetskiy, I.; Morozov, R.; Lisenkov, S. Reinforcement of polymer composite materials by titanium dioxide nanoparticles synthesized in plasma discharge under ultrasonic cavitation. *J. Phys. Conf. Ser.* **2022**, *2231*, 012012. [\[CrossRef\]](#)
38. Radhakrishnan, S.; Siju, C.R.; Mahanta, D.; Patil, S.; Madras, G. Conducting polyaniline–nano-TiO₂ composites for smart corrosion resistant coatings. *Electrochim. Acta* **2009**, *54*, 1249–1254. [\[CrossRef\]](#)
39. Díez-Pascual, A.M.; Díez-Vicente, A.L. Nano-TiO₂ reinforced PEEK/PEI blends as biomaterials for load-bearing implant applications. *ACS Appl. Mater. Interfaces* **2015**, *7*, 5561–5573. [\[CrossRef\]](#)
40. Prasad, V.; Sekar, K.; Varghese, S.; Joseph, M.A. Evaluation of interlaminar fracture toughness and dynamic mechanical properties of nano TiO₂ coated flax fibre epoxy composites. *Polym. Test.* **2020**, *91*, 106784. [\[CrossRef\]](#)
41. Perumal, K.P.S.; Boopathi, R.; Saravanan, P.; Selvarajan, L. Effect of zircon and anatase titanium dioxide nanoparticles on glass fibre reinforced epoxy with mechanical and morphological studies. *Ceram. Int.* **2023**, *49*, 21667–21677. [\[CrossRef\]](#)
42. Elamvazhudi, B.; Gopalakannan, S. Effect of nanoclay and nanoscale TiO₂ on carbon/glass fibrereinforced polymer composites. *Mater. Res. Express* **2020**, *7*, 075303. [\[CrossRef\]](#)
43. Barrionuevo, G.O.; Ramos-Grez, J.A. Machine Learning for Optimizing Technological Properties of Wood Composite Filament-Timberfill Fabricated by Fused Deposition Modeling. *Commun. Comput. Inf. Sci.* **2019**, *1194*, 119–132. [\[CrossRef\]](#)
44. Liu, J.; Zhang, Y.; Zhang, Y.; Kitipornchai, S.; Yang, J. Machine learning assisted prediction of mechanical properties of graphene/aluminium nanocomposite based on molecular dynamics simulation. *Mater. Des.* **2022**, *213*, 110334. [\[CrossRef\]](#)
45. Kim, Y.; Oh, H. Comparison between Multiple Regression Analysis, Polynomial Regression Analysis, and an Artificial Neural Network for Tensile Strength Prediction of BFRP and GFRP. *Materials* **2021**, *14*, 4861. [\[CrossRef\]](#) [\[PubMed\]](#)
46. Hirani, H.; Verma, M. Effect of titanium dioxide (TiO₂) nanoparticles on the wear rate of marine journal bearings made of composite materials. *Results Eng.* **2025**, *27*, 105949. [\[CrossRef\]](#)
47. Khan, S.B.; Li, N.; Chen, S.; Liang, J.; Xiao, C.; Sun, X. Influence of nanoparticle size on the mechanical and tribological characteristics of TiO₂ reinforced epoxy composites. *J. Mater. Res. Technol.* **2023**, *26*, 6001–6015. [\[CrossRef\]](#)
48. Saffar Shamshirgar, A.; Toghyani, A.; Ghasemi-Ghalebahman, A.; Ahmadihah, M. Raman spectroscopy study of the role of dispersion and interfaces in the improvement of the mechanical properties of epoxy-TiO₂ composites. *J. Mater. Res. Technol.* **2025**, *34*, 1744–1758. [\[CrossRef\]](#)
49. Zeng, X.L.; Tan, L.S.; He, S.Y.; Zhang, J. Effect of graphene oxide size on interlaminar shear strength of glass fabric/epoxy composites. *Mater. Res. Express* **2019**, *6*, 105306. [\[CrossRef\]](#)
50. Parveen, S.; Pichandi, S.; Goswami, P.; Rana, S. Novel glass fibre reinforced hierarchical composites with improved interfacial, mechanical and dynamic mechanical properties developed using cellulose microcrystals. *Mater. Des.* **2020**, *188*, 108448. [\[CrossRef\]](#)
51. Harsha, M.S.; Shivamurthy, B.; Anandkumar, R. Synergistic effects of titanium dioxide and graphene nanofillers on delamination and thrust forces in machining glass fiber reinforced nanocomposites. *Sci. Rep.* **2025**, *15*, 1–18. [\[CrossRef\]](#) [\[PubMed\]](#)

52. Varghese, A.S.; Sreekanth, M.S. Investigation on the effect of multiwalled carbon nanotubes in carbon fiber-reinforced epoxy composites manufactured using vacuum infusion process and hand layup process followed by vacuum bagging. *Polym. Compos.* **2024**, *45*, 1600–1618. [CrossRef]
53. Amar, T.A.; Kumar, A.; Yadav, D.K. A Comparative Study on the Properties of Carbon Fiber-Reinforced Polymer Composites Developed by Hand Layup and Vacuum Bagging Molding Techniques. *J. Inst. Eng. (India) Ser. D* **2024**, *106*, 263–275. [CrossRef]
54. Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials. Available online: https://store.astm.org/d3039_d3039m-08.html (accessed on 25 October 2025).
55. Standard Test Method for Flexural Properties of Polymer Matrix Composite Materials. Available online: https://store.astm.org/d7264_d7264m-21.html (accessed on 25 October 2025).
56. Navas-Pinto, W.; Cree, D.E.; Wilson, L.D.; Barrionuevo, G.O.; Sánchez-Sánchez, X.; Calvopiña, H. Characterization of graphene oxide synthesized through a modified Hummers method. *Ingenius* **2025**, *34*, 31–42. [CrossRef]
57. Abdelhamied, M.M.; Atta, A.; Alotaibi, B.M.; Al-Harbi, N.; Henaish, A.M.A.; Rabia, M. Synthesis of flexible polymer nanocomposites based on methyl cellulose/copper oxide with desired dielectric properties for electrical applications. *Inorg. Chem. Commun.* **2023**, *157*, 111245. [CrossRef]
58. Çağlar, S.; Gaylan, Y. Optimization of the microstructural, mechanical, and radiation shielding properties of Al-30B4C-25 W hybrid composites with Gd₂O₃ reinforcement. *Sci. Rep.* **2025**, *15*, 29867. [CrossRef]
59. Noman, M.T.; Amor, N.; Petru, M.; Mahmood, A.; Kejzlar, P. Photocatalytic Behaviour of Zinc Oxide Nanostructures on Surface Activation of Polymeric Fibres. *Polymers* **2021**, *13*, 1227. [CrossRef]
60. La Fé-Perdomo, I.; Loyola, O.; Barrionuevo, G.O.; Ramos-Grez, J.A. Adiabatic efficiency and mechanical properties optimization for the laser-based powder bed fusion process of 316L stainless steel alloy. *Mater. Today Commun.* **2025**, *49*, 113616. [CrossRef]
61. Bukichev, Y.S.; Bogdanova, L.M.; Lesnichaya, V.A.; Chukanov, N.V.; Golubeva, N.D.; Dzhardimalieva, G.I. Mechanical and Thermophysical Properties of Epoxy Nanocomposites with Titanium Dioxide Nanoparticles. *Appl. Sci.* **2023**, *13*, 4488. [CrossRef]
62. Yang, J.; Dai, J.; Liu, X.; Fu, S.; Zong, E.; Song, P. A lignin-based epoxy/TiO₂ hybrid nanoparticle for multifunctional bio-based epoxy with improved mechanical, UV absorption and antibacterial properties. *Int. J. Biol. Macromol.* **2022**, *210*, 85–93. [CrossRef] [PubMed]
63. Xia, R.; Xi, J.; Zhang, Z.; He, Y.; Yu, Z. Curing Behavior of UV-Initiated Surface-Modified Nano-TiO₂/Epoxy Resin Prepolymers and the Properties of Cured Composites. *Polymers* **2023**, *15*, 1756. [CrossRef] [PubMed]
64. Goyat, M.S.; Rana, S.; Halder, S.; Ghosh, P.K. Facile fabrication of epoxy-TiO₂ nanocomposites: A critical analysis of TiO₂ impact on mechanical properties and toughening mechanisms. *Ultrason. Sonochem.* **2018**, *40*, 861–873. [CrossRef] [PubMed]
65. Yadhav, B.R.L.; Govindaraju, H.K.; Kiran, M.D.; Suresha, B. Three-point bending and impact behaviour of carbon/epoxy composites modified with titanium dioxide nanoparticles. *Mater. Today Proc.* **2021**, *43*, 1755–1761. [CrossRef]
66. Hatata, S.M.; Salman, H.H. Study of Impact Resistant and Flexural Strength for Epoxy Composite Reinforced by Nano and Micro Materials of Silica and Titanium Oxide. *AIP Conf. Proc.* **2023**, *2845*, 070036. [CrossRef]
67. Kamesh, B.; Singh, L.K.; Kassa, M.K.; Arumugam, A.B. Synergetic effect of incorporating graphene, CNT and hybrid nanoparticles on the mechanical properties of glass fiber reinforced epoxy laminated composites. *Cogent Eng.* **2023**, *10*, 2232604. [CrossRef]
68. Pinto, D.; Bernardo, L.; Amaro, A.; Lopes, S. Mechanical properties of epoxy nanocomposites using titanium dioxide as reinforcement—A review. *Constr. Build. Mater.* **2015**, *95*, 506–524. [CrossRef]
69. Kumar, A.; Saha, A.; Kumar, S. Structural analysis of sol-gel derived TiO₂ nanoparticles: A critical impact of TiO₂ nanoparticles on thermo-mechanical mechanism of glass fiber polymer composites. *J. Polym. Res.* **2021**, *28*, 441. [CrossRef]
70. Perumal, K.P.S.; Selvarajan, L.; Kumar, P.M.; Shriguppikar, S. Enhancing mechanical and morphological properties of glass fiber reinforced epoxy polymer composites through rutile nanoparticle incorporation. *Prog. Addit. Manuf.* **2024**, *10*, 831–848. [CrossRef]
71. Dasari, S.; Lohani, S.; Dash, S.S.; Fulmali, A.O.; Prusty, R.K.; Ray, B.C. A novel study of flexural behavior of short glass fibers as secondary reinforcements in GFRP composite. *Mater. Today Proc.* **2021**, *47*, 3370–3374. [CrossRef]
72. Abebe, Y.; Palani, S.; Sirahbizu, B. Effect of Al₂O₃ nanoparticle on mechanical properties of polyester/ glass-wool fiber reinforced polymer composites. *Hybrid Adv.* **2025**, *10*, 100472. [CrossRef]
73. Barbaz-Isfahani, R.; Shariati, M.; Jalali Azizpour, M. Synergistic Effects of Incorporating Various Types of Nanoparticles on Tensile, Flexural, and Quasi-static Behaviors of GFRP Composites. *Fibers Polym.* **2022**, *23*, 2003–2016. [CrossRef]
74. Turaka, S.; Chintalapudi, R.; Geetha, N.K.; Pappula, B.; Makgato, S. Experimental and numerical analysis of the Microstructure and mechanical properties of unidirectional glass fiber reinforced epoxy composites. *Compos. Struct.* **2024**, *331*, 117887. [CrossRef]
75. Ojha, S.; Bisaria, H.; Mohanty, S.; Kanny, K. Fractographic analysis of fiber-reinforced polymer laminate composites for marine applications: A comprehensive review. *Polym. Compos.* **2024**, *45*, 6771–6787. [CrossRef]
76. Howlader, M.I.; Islam, M.S.; Haque, M.M. Mechanical and morphological analysis of natural fiber-reinforced epoxy and polyester hybrid composites. *Results Eng.* **2025**, *26*, 105010. [CrossRef]
77. Zhang, W.; Yin, L.; Zhao, M.; Tan, Z.; Li, G. Rapid and non-destructive quality verification of epoxy resin product using ATR-FTIR spectroscopy coupled with chemometric methods. *Microchem. J.* **2021**, *168*, 106397. [CrossRef]

78. Pannico, M.; Mensitieri, G.; Musto, P. In-situ FTIR spectroscopy of epoxy resin degradation: Kinetics and mechanisms. *Front. Chem.* **2024**, *12*, 1476965. [[CrossRef](#)]
79. Mamudu, U.; Dufresne, A.; Lim, R.C. Synthesis and characterization of phosphorylated cellulose nanocrystals for enhanced UV stability of epoxy nanocomposite films. *Compos. Part C Open Access* **2023**, *12*, 100393. [[CrossRef](#)]
80. Nikolic, G.; Zlatković, S.J.; Živković, M.Ž.; Nikolić, A.S.; Kakurinov, V.B.; Živković, D.T. Fast Fourier Transform IR Characterization of Epoxy GY Systems Crosslinked with Aliphatic and Cycloaliphatic EH Polyamine Adducts. *Sensors* **2010**, *10*, 684–696. [[CrossRef](#)]
81. Gonçalves, F.A.M.M.; Ferreira, P.; Alves, P. Synthesis and characterization of itaconic-based epoxy resin: Chemical and thermal properties of partially biobased epoxy resins. *Polymer* **2021**, *235*, 124285. [[CrossRef](#)]
82. Girigoswami, A.; Deepika, B.; Pandurangan, A.K.; Girigoswami, K. Preparation of titanium dioxide nanoparticles from *Solanum Tuberosum* peel extract and its applications. *Artif. Cells Nanomed. Biotechnol.* **2024**, *52*, 59–68. [[CrossRef](#)] [[PubMed](#)]
83. Barrionuevo, G.O.; Walczak, M.; Ramos-Grez, J.; Sánchez-Sánchez, X. Microhardness and wear resistance in materials manufactured by laser powder bed fusion: Machine learning approach for property prediction. *CIRP J. Manuf. Sci. Technol.* **2023**, *43*, 106–114. [[CrossRef](#)]
84. Barrionuevo, G.O.; Ramos-Grez, J.A.; Walczak, M.; Betancourt, C.A. Comparative evaluation of supervised machine learning algorithms in the prediction of the relative density of 316L stainless steel fabricated by selective laser melting. *Int. J. Adv. Manuf. Technol.* **2021**, *113*, 419–433. [[CrossRef](#)]
85. Ramezani, M.; Choe, D.E.; Rasheed, A. Prediction of the flexural strength and elastic modulus of cementitious materials reinforced with carbon nanotubes: An approach with artificial intelligence. *Eng. Appl. Artif. Intell.* **2025**, *150*, 110544. [[CrossRef](#)]
86. Mirindi, D.; Sinkhonde, D.; Bezabih, T.; Mirindi, F.; Oshineye, O.; Mirindi, P. Prediction of flexural and split tensile strength of waste glass-concrete composite using machine learning algorithms. *Green Technol. Sustain.* **2026**, *4*, 100275. [[CrossRef](#)]
87. Sarmiento-Pupo, K.D.; Escalante-Tovar, J.D.; Altamiranda, J.E.; Abellan-Garcia, J.; Vélez, J.I. Predicting flexural strength in fiber-reinforced UHPC via random forest. *Sci. Rep.* **2025**, *15*, 42045. [[CrossRef](#)]
88. Fu, Y.; Yao, X. A review on manufacturing defects and their detection of fiber reinforced resin matrix composites. *Compos. Part C Open Access* **2022**, *8*, 100276. [[CrossRef](#)]

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