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Composites & nanocomposites

Effect of nanoclay on the mechanical and thermal properties of glass fiber-reinforced epoxy composites

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ABSTRACT

The effects of nanoclay (NC) addition on the thermal and mechanical properties of glass fiber-reinforced epoxy composites were investigated experimentally in this study. Nanocomposite plates were produced for this purpose using three different NC ratios (0.5%, 1%, and 1.5% by weight). Thermal characteristics of nanocomposites were investigated using dynamic mechanical analysis, differential scanning calorimetry, and thermogravimetric analysis. The mechanical and thermal results obtained from composites with three different NC ratios were compared with the results obtained from pure composites. The structures of nanocomposites were investigated with the help of SEM–EDS analyses. Furthermore, the effect of nanoclay on the failure behavior of composites was also investigated. In this study, the highest values in all mechanical properties were obtained from samples with a 1% NC-added. Obtained from 1% NC-added samples: tensile, compressive, shear strengths, elasticity modulus, shear modulus, and Poisson's ratio values were 31.06%, 4.25%, 14.30%, 7.35%, 11.94%, and 12.5% higher, respectively, than the values obtained from pure samples. Maximum loss modulus and maximum storage modulus were obtained from samples with 1.5% and 0.5% NC-added, respectively. In samples with 1.5% NC-added, the highest Tan δ value was obtained. Glass transition temperatures increased with the added NC. It was observed that the fiber–matrix interfaces were more clearly separated in the samples with 1.5% NC-added.

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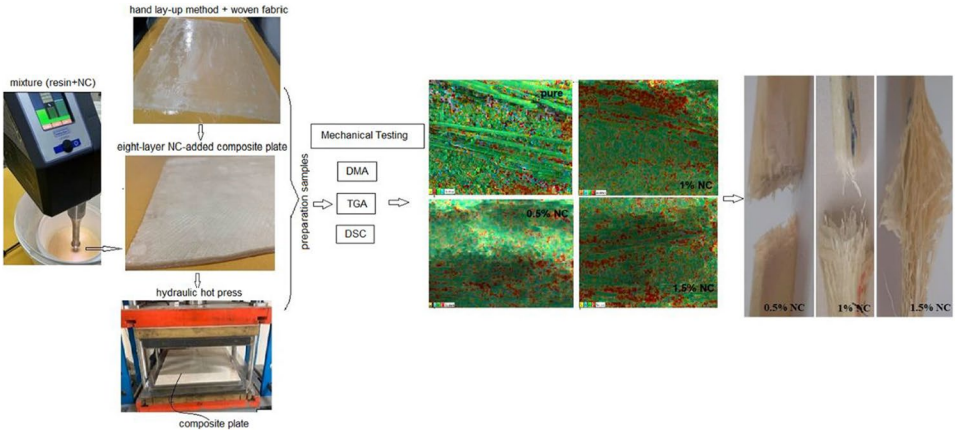
GRAPHICAL ABSTRACT



Introduction

Nanoparticles are used to improve the mechanical [1], thermal, and electrical properties of composites [2], depending on their application areas and types. Especially when added to polymer systems, highly effective polymer nanocomposites are obtained. One of the nanoparticles added as additives to polymeric composites is nanoclay. Clay is widely used due to its advantages, such as its abundance in nature, low material cost, and high size-to-strength ratio [3]. Clay nanocomposites show distinctive properties such as exceptional thermal stability, enhanced flame retardancy [4], high-dimensional stability, reduced gas permeability, and enhanced anticorrosive and mechanical properties [2, 5]. Therefore, it is a vital nanofiller that significantly affects the properties of the polymer by supporting improved polymer processing [6].

In this regard, many researchers have investigated into the effects of adding NC to epoxy and polymer resins. Merah et al. [7] and Dias et al. [8]. investigated the effects of NC addition on the mechanical [8] and physical properties of the composite. They reported studies on epoxy clay nanocomposites [7]. Merzah et al. [9] and Sabu et al. [10] added NC to epoxy matrix at (1, 2, and 3%) [9] and (1, 3, and 5%) [10] by weight, respectively. The authors reported that tensile strength [9, 10], impact strength, hardness, and Young's modulus [9] increased with the addition of NC. Kiran et al. [11] studied the effects of epoxy–glass–clay nanocomposites (by varying the NC weight percent, glass fiber volume percent, and fiber orientation angle) on the tensile and bending properties. In their study, they stated that optimum values for ultimate tensile strength and flexural strength were obtained from samples with 2.5% NC, 31.5% (by volume) glass fiber, and 0°/90° fiber orientation. Ankit et al. [12] investigated the mechanical properties of a Luffa/epoxy composite material with the addition of different NC (0, 1, 2, 3%). It was stated that the mechanical properties increased up to an addition of 2.0 NC, and then the values became insignificant. Kowshik et al. [13] and Devi et al. [14] investigated the flexural [13] and impact strengths [13, 14] of glass fiber-reinforced epoxy composites with NC added at (0.2–4%) [13], (1.5, 3.5, and 5%) [14], respectively. The authors reported that the flexural and impact strengths of the composites at 0.2–4% [13] and the impact strength of the composite at 3.5% increased [14]. Kirve et al. [15] and Verma et al. [16] added NC additives between (0–7%) [15] and (0.5–4%) [16] by weight to bamboo/epoxy composites [15] and E-glass unidirectional fiber composites with different arrangement orders [16]. In their studies, they stated that a 3% NC additive had a positive effect on tensile, flexural [15, 16], and impact strength. Agnihotri et al. [17] investigated the effect on the mechanical properties of carbon fiber-reinforced polymer composites by adding 0, 1.25, 2.50, or 3.75% by weight of NC to epoxy. They concluded that there was a sustained increase in flexural, ILSS, and tensile strengths of up to 2.5% by weight in NC. Pumchusak et al. [18] also investigated the effects of 0.5–2.5 wt% organomodified montmorillonite NC (O-MMT) on short carbon fiber-reinforced phenolic (CFRP) composites. The authors reported that the use of 1.5%

O-MMT increased the tensile strength of CFRP by 20% and Young's modulus by 11%. Annappa et al. [19] investigated the effect of organoclays on the mechanical properties of glass–epoxy nanocomposites for 2% filler loading. In the experimental study, it was stated that there were improvements in tensile and bending strength. Theja et al. [20] synthesized NC with rubber composites to obtain nanocomposites. In the study, it was stated that the modulus, hardness, and wear resistance of the material were greatly improved with the addition of NC. Selly et al. [21, 22] investigated the effect of different NC reinforcements [22], silanized NC [21], unmodified clay, and organically modified clay [22] on the mechanical properties of epoxy-based glass fiber-reinforced composite plates. They found significant improvements in all mechanical properties, including impact strength, flexural properties [21, 22], fracture toughness [22], and tensile properties [21]. Sapiai et al. [23] hybridized kenaf composites with glass fiber and used NC at 1%, 3%, and 5% weight ratios. They stated that the addition of 1% and 3% by weight of NC improved the tensile properties of NC-filled Kenaf (KFRP) composites. Shettar et al. [24, 25] obtained NC at 0.2, 4% by weight, to obtain "NC-epoxy composites" [24] and "Polyester-NC composites" [25]. They stated that the maximum tensile strength was obtained by adding 2% [25] and 4% NC [24] by weight. In carbon/fiberglass hybrid composites, Oner et al. [26] used NC (0%, 0.5%, 0.75%, and 1.25% by weight). They stated that the sample containing 0.75% NC by weight had the highest tensile strength. Awan et al. [27] developed NC (3% by weight) and calcium carbonate (CaCO3)-coated, reinforced binary, and triple high-density polyethylene (HDPE) composites. They stated that there was an increase of 27.7% in tensile strength, 118% in toughness, and 15% in hardness. Jeyakumar et al. [28] stated that the compression, tensile strength, tensile modulus, and hardness of glass fiber-reinforced epoxy composites increased significantly with the addition of NC and then decreased with the addition of NC. Beesetty et al. [29] changed the NC ratios (0.5, 1, 2, 5%) and blended it with HDPE (high density polyethylene) to develop the filament to be used in 3D printers. They stated that the tensile modulus and the strength of the filament increased with the addition of NC in the HDPE matrix. Krushnamurty et al. [30] investigated the fracture toughness of the epoxy matrix by adding organically modified NC (ranging from 0 to 5% by weight) to epoxy and E-glass–epoxy composites. In the addition of 3% by weight NC of epoxy composites, they stated that the toughness, flexural strength, and tensile strength increased by 25%, 13%, and 11%, respectively. Kumar et al. [31] investigated the vibration and low-velocity impact behavior of AlMg4.5Mn-reinforced NC nanocomposites at room temperature. They produced composites containing NC with different weight percentages (0, 1.5, and 2.5%). They found that, in addition to the weight percent of NC, the hardness value and impact natural frequencies of the composite increased. Muralishwara et al. [32] evaluated the epoxy clay nanocomposite coating in terms of surface hardness, surface roughness, surface morphology, and topography. They conducted an experimental experiment by considering the parameters of NC loading, sonication amplitude, and sonication time. They stated that the maximum hardness value was obtained when the NC loading was 1% by weight, the sonication time was 20 min, and the sonication amplitude was 100%. Chen et al. [33] investigated the mechanical properties of NC-containing BFRPs (basalt fiber-reinforced polymers) by preparing NCs with different morphologies and structures. They successfully improved the weak inherent interfacial adhesion of inorganic basalt fiber by adding NCs. Hashemi-Nasirabad et al. [34] investigated the mechanical properties of a urea–formaldehyde (UF) resin binder used in the production of fiberglass mats. They stated that adding 4% weight-based NC resulted in an additional 33% increase in tensile strength. Maksimov et al. [35] prepared a polymer nanocomposite by combining an aqueous polymer emulsion with an aqueous dispersion of Montmorillonite (MMT) clay. They noted that when they used a relatively small amount of montmorillonite clay, the mechanical properties of the material improved significantly. At a content of 15% by weight, i.e., only 7% by volume, of MMT, they found that its strength and elastic modulus increased by 1.4 and 3 times, respectively, compared to the pure polymer. Anandraj et al. [36] investigated the mechanical properties of the compound, which consisted of 30% abaca fiber content, 2% NC content, and 8% NaOH concentration content. They stated that the increased content of abaca fiber and NC causes a decrease in mechanical properties. Vivekanandhan et al. [37] investigated the effects of NC at ratios of 2–10% by weight on the mechanical properties of epoxy composites. The authors reported that tensile and

flexural strength decreased with increasing NC. Fakhreddini-Najafabadi et al. [38] synthesized nanoclay/epoxy nanocomposites in order to compare the mechanical properties of the samples and find the appropriate methodology. They stated that the tensile strength improved with the methodologies they applied.

Many researchers have examined the effect of clay on some mechanical properties as well as its effect on thermal properties. Zawawi et al. [39] used poly (lactide) acid (PLA) and polypropylene (PP) with a constant PLA/PP ratio of 75–25% by weight and added 3% by weight MAgPP to their polymer blends as a compatibilizer. They added organo-nanoclays of montmorillonite (OMMT) to the polymer mixtures (1, 3, 5, and 7% by weight). They achieved the greatest tensile strength in composites containing 1% NC by weight. The TGA results stated that the degradation temperature was improved by adding 5% NC by weight to the PLA/PP blend. Karippal et al. [40] used 0–6% wt NC to produce epoxy/glass/NC hybrid composites. They noted that the tensile strength, or Young's modulus, increased by up to 5% by weight with the increase in NC loading. Basara et al. [41] obtained epoxy nanocomposites reinforced with diglycidyl ether of bisphenol-A (epoxy), containing 0.5–11% by weight of natural (Cloisite Na) and organically modified (Cloisite 30B) montmorillonite species. They stated that the glass transition temperature increased from 73 °C in unfilled epoxy resin to 83.5 °C in nanocomposite with 9% Cloisite 30B. They stated that the tensile strength reached the maximum level at 1% by weight of modified clay. Nayak et al. [42] investigated the effect on flexural and thermal properties by adding reinforced nanosilica (1% by weight) and NC (2% by weight) in glass fiber/epoxy composites. They stated that 1% by weight of silica nanoparticles and a mixture of 2% by weight of NC and epoxy decreased the tg compared to the pure samples. Salih et al. [43] obtained polymer composites by using NC in different ratios (3, 5, and 7%) and mixtures of epoxy and polymethyl methacrylate (PMMA) as a matrix. The authors stated that in addition to the improved mechanical properties, NC makes a positive contribution to sound insulation, impact resistance, thermal conductivity value, and bending strength. Öner et al. [44] investigated thermogravimetric and differential scanning calorimetry of a composite wall made of glass/carbon/glass layers with NC epoxy resin in different ratios (0.50, 0.75, and 1.25%). The authors stated that the lowest mass loss was obtained from samples containing 0.75% NC, and the highest glass transition temperature was obtained from samples containing 1.25% NC. Moyo et al. [45] investigated the thermo-mechanical properties of kenaf fiber-reinforced polylactic acid (PLA) biocomposites doped with clay (0, 3, 5, and 7% by weight) nanoparticles. In the dynamic mechanical analysis (DMA) results, it was determined that the storage and loss modules increased up to 5% wt NC loading and then a decrease was observed, and that the increase in the NC ratio decreased the glass transition temperature (tg) of the biocomposites. Dorigato and Pegoretti [46] stated in their study that the glass transition temperature increased with the added clay ratio (0.5, 3, or 5%). Dean et al. [47] studied the effect of organoclay nanoparticles on the rheology, morphology, and properties of epoxy/organoclay nanocomposites. According to the tan graph obtained by DMA analysis, the glass transition temperature increased. Gudapati et al. [48] stated in their study that the thermal characterization of wild cane grass fiber-reinforced polyester composites showed very good results with the addition of 4% NC. They stated that with the increase in fiber content, the thermal conductivity of the composite decreased, and quite the opposite trend was observed with respect to the temperature. Bakar et al. [49] investigated the effects of organomodified montmorillonite (MMT) content (1, 2, and 3%) and mixing conditions on the mechanical properties, structure, and morphology of rubber. In their study, they stated that changes in mixing amplitudes (162, 216, and 270 m) and MMT content did not affect the DSC and TGA thermograms, confirming the unchanged thermal stability of the nanocomposites. De Maria et al. [50] modified Sodium clay and organo-clay via silylation reaction. These silylated clays were characterized by IR, XRD, and thermogravimetric analyses. They stated that the nanocomposites had better thermal properties and increased glass and decomposition temperatures. Mohan and Kanny [51] fabricated a hybrid composite of glass fiber-reinforced epoxy polymer filled with NC particles (0–5 wt%) using the VARIM technique. The authors reported

that the storage modulus, glass transition temperature (Tg), and $Tan\ \delta$ curve were affected. Mazlan et al. [52] studied the thermogravimetric and dynamic mechanical analysis of woven glass/fiber/epoxy hybrid nanocomposites filled with 1.0 wt% silicone-modified epoxy resin and varying clay loadings of 1, 3, and 5 wt%. They reported that the highest storage modulus was obtained at 1 wt% NC.

When the studies in the literature are examined, it becomes clear that the effects of NC on the mechanical and thermal properties of composites have been investigated separately. However, in this study, the mechanical properties of the composite produced with different weight percentages (0, 0.5%, 1%, and 1.5%) were determined, as well as TGA, DMA, and DSC analyses that were performed and evaluated in tandem. At the same time, SEM–EDS analyses of the samples were made, and failure modes were examined after the experiment. The findings of this study constitute an important data area in terms of a comprehensive assessment of the effect of NC on the thermal and mechanical properties of the composite.

Materials and experimental procedure

Mechanical tests

In the study, E-glass fiber-reinforced woven fabric was used. In the resin system, epoxy resin with a density of 1.15 g/cm^3 (FRES 21, Fibermak, Turkey) was used. The organomodified NC was obtained from Eczacıbaşı Company (Turkey). The interfacial area can influence the properties of nanocomposites; hence, it is important that the NC be homogeneously distributed throughout the epoxy resin [26]. In order to ensure the dispersion of NC in the polymer matrix, an ultrasonic mixer (Hielscher UP400S) was used for mixing. The device (Fig. 1a) uses sonication to create sound waves that disintegrate the mixture's agglomerates. Therefore, the mixture was mixed with the help of the device for 45 min. Using a laser thermometer, the mixture's temperature was continuously monitored as it was being mixed. Sonication stopped until the mixture's temperature decreased when it was getting close to 50 °C. A hardener (FHARD 22, Fibermak, Turkey) with a density of 1 g/cm^3 was added to the mixture

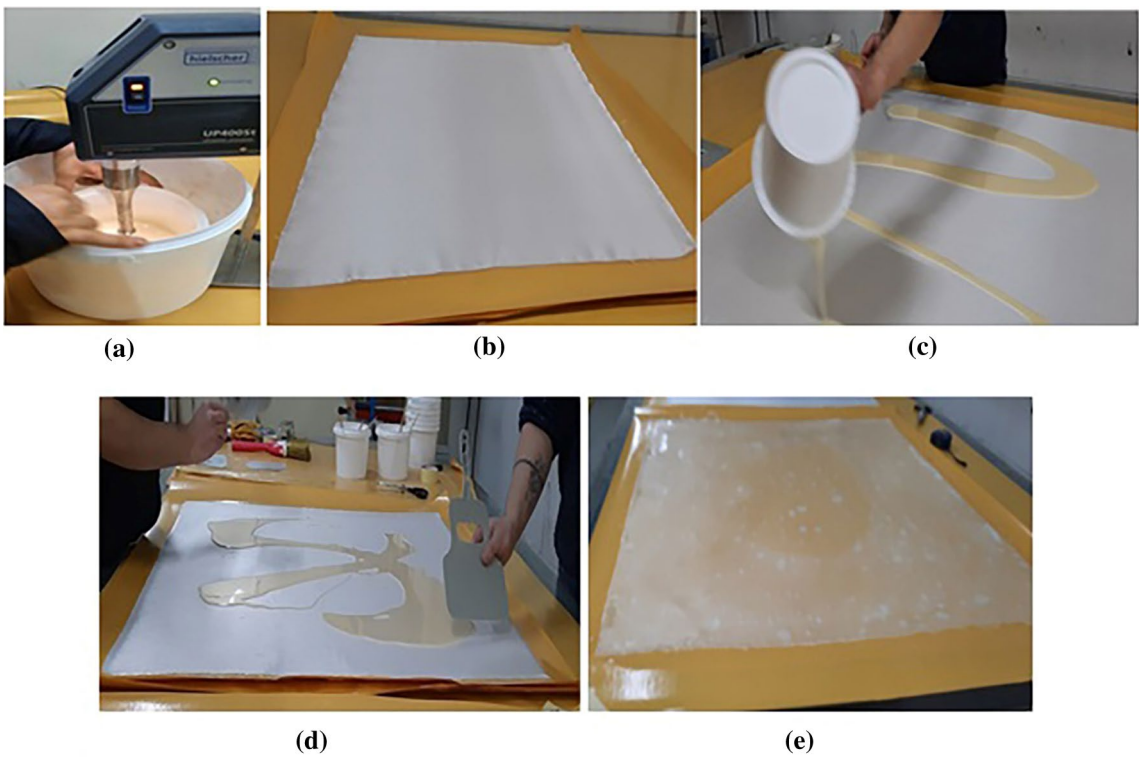


Figure 1 Hand lay-up production steps: **a** ultrasonic mixer; **b** glass fiber epoxy woven fabric; **c**, **d** hand lay-up process; **e** impregnated fabric.

Figure 2 NC-reinforced glass fiber-reinforced composite plate manufacturing impregnated fabric.

following sonication. The mixture was mixed with an ultrasonic mixer for another 10 min. Then, the mixture was applied to the glass fiber fabric (Fig. 1b) by hand lay-up method (Fig. 1c, d, e) and production was carried out. The production processes were repeated in the same way by adding NC at rates of 0.5%, 1%, and 1.5% by weight.

The fabrics, which were prepared separately and impregnated with a NC resin mixture, were left to dry at room temperature for 10 days. Semi-finished fabrics were stacked (Fig. 2a), wrapped with fireproof film, and then placed in a hydraulic hot press (Fig. 2b). First, the fabrics were exposed to 10 bars of pressure. Then the temperature was increased from room temperature to 125 °C. The plate was left in these conditions for one hour. At the end of the time, the heater was turned off, and the plates were brought to room temperature. Composite plates with 8 layers and 2.5 mm thickness were cut with the help of a CNC machine in accordance with ASTM standards for experimental processes. Since it is stated in the literature that sample preparation according to standards has significant effects on the experimental results [53], these parameters were prepared carefully. Five samples were prepared for each test.

The tests were carried out in the Dicle University DÜBTAM laboratory at room temperature (23 ± 3 °C) and 50 °C ± 10 °C relative humidity [54, 55]. For tensile tests, samples of 250 × 25 mm^2 (Fig. 3a) were prepared according to the ASTM D3039-17 standard [56], and for compression tests, samples of 140 × 13 mm^2 (Fig. 3b) were prepared according to the ASTM D6641-16 standard [57]. Shear test samples were prepared according to the ASTM D7078-20 standard [58] (L = 76 mm, w = 56 mm, d = 31 mm, r = 1.3 mm) (Fig. 3c). Tensile and shear tests were carried out at a speed of 2 mm/min, and compression tests were carried out at a speed of 1 mm/min.

To prevent stress concentrations, composite joints were adhered to the ends of the tensile test specimens with adhesive. Experiments were conducted in an Instron brand 8801-type device with a capacity of

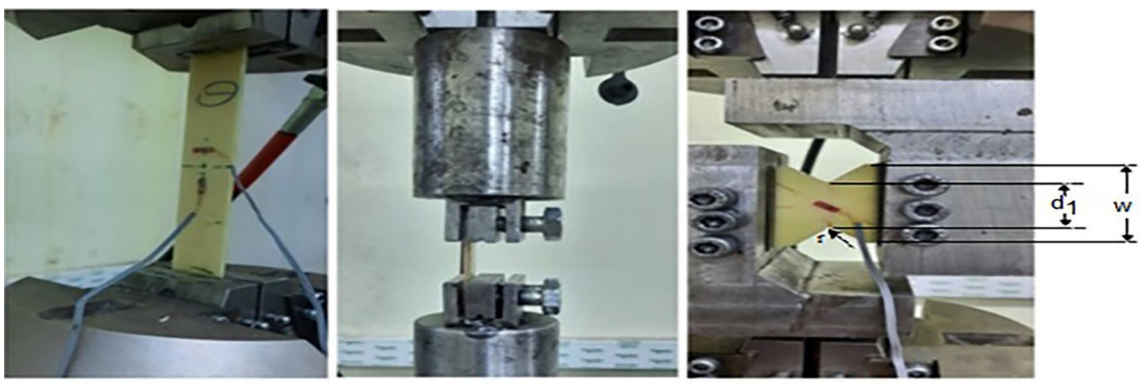


Figure 3 Mechanical tests **a** Tensile test **b** Compression test **c** Shear test.

100 kN. At the same time, experiments were carried out for the modulus of elasticity, Poisson's ratio, and shear modulus by making a strain gauge connection to the samples.

Hardness of composites was measured in Vickers test mode with an AOB brand test device at 0.3 kg load and a 10-s dwell time (HV 0.3/10). Five tests were performed on each sample, which were then averaged.

DMA analysis

The samples were subjected to dynamic mechanical analysis (DMA) on a TA Instruments Q800 DMA device at the Ege University Central Research Test and Analysis Laboratory Application and Research Center. Sample dimensions (56 mm × 12 mm) were produced in accordance with the ASTM D7028-07 [59] standard. A dual cantilever mode was applied to the samples as a test method. The test was carried out with a frequency of 1 Hz, a force of 1 N, an amplitude of 15 m, a heating rate of 5 °C per minute, and temperatures ranging from room temperature to 150 °C. The storage modulus (E′), loss modulus (E″), and tan δ -temperature curves of the samples were obtained as a result of this analysis.

DSC analysis

The glass transition temperatures (tg) of the samples weighing 0.5 mg were determined by differential scanning calorimetry (DSC) analysis. This analysis was performed in a Schimadzu DSC-60 type device by gradually increasing the temperature to 500 °C at a rate of 10 °C per minute.

TGA analysis

Thermogravimetric analysis (TGA) was performed using DTG-60H Shimadzu brand device. Samples (TGA) were made by heating from 23 to 800 °C at a heating rate of 10 °C/min.

SEM and EDS analysis

SEM–EDS analysis is an analytical technique used to perform nano-characterization. EDS (Energy-Dispersive Spectrograph) provides data on the chemical composition of the sample and provides additional data on the structure observed in SEM (Scanning Electron Microscopy). SEM and EDS analyses of the samples were performed in Munzur University's Materials Laboratory. Analyses were performed with a JEOL JSM-5600 scanning electron microscope.

Results and discussion

Mechanical properties

Pure and three different amounts of NC (0.5%, 1%, and 1.5% by weight) added glass fiber-reinforced epoxy composites; tensile strengths, compressive strengths, shear strengths, elasticity modules, shear moduli, Poisson's ratios, and hardness values were obtained as a result of mechanical tests, and the results are given in Table 1. Additionally, the stress–strain graphs obtained as a result of tensile (Fig. 4a), compression (Fig. 4b), and shear tests (Fig. 4c) for all samples are given comparatively in Fig. 4.

As a result of the tensile tests, the values obtained from all NC-added samples were higher than the

Table 1 Mechanical properties of glass fiber-reinforced epoxy composites

Mechanical properties	Pure	Nanoclay		
		0.5%	1%	1.5%
Tensile strength (Mpa)—(X_T)	448.38	569.32	587.64	545.83
Compressive strength (MPa)—(Xc)	292.87	276.77	305.32	287.95
Shear strength (MPa)—(S12)	119.81	134.53	136.94	129.71
Modulus of elasticity (MPa)—(En)	22,085.98	23,047	23,709.53	23,326.72
Shear modulus (MPa)—(G_{L2})	4087.78	4497.24	4576.04	4398.13
Poisson's ratio—(i;o)	0.16	0.20	0.18	0.18
Hardness (HVO.3/10)	31	38.83	41.1	39.05

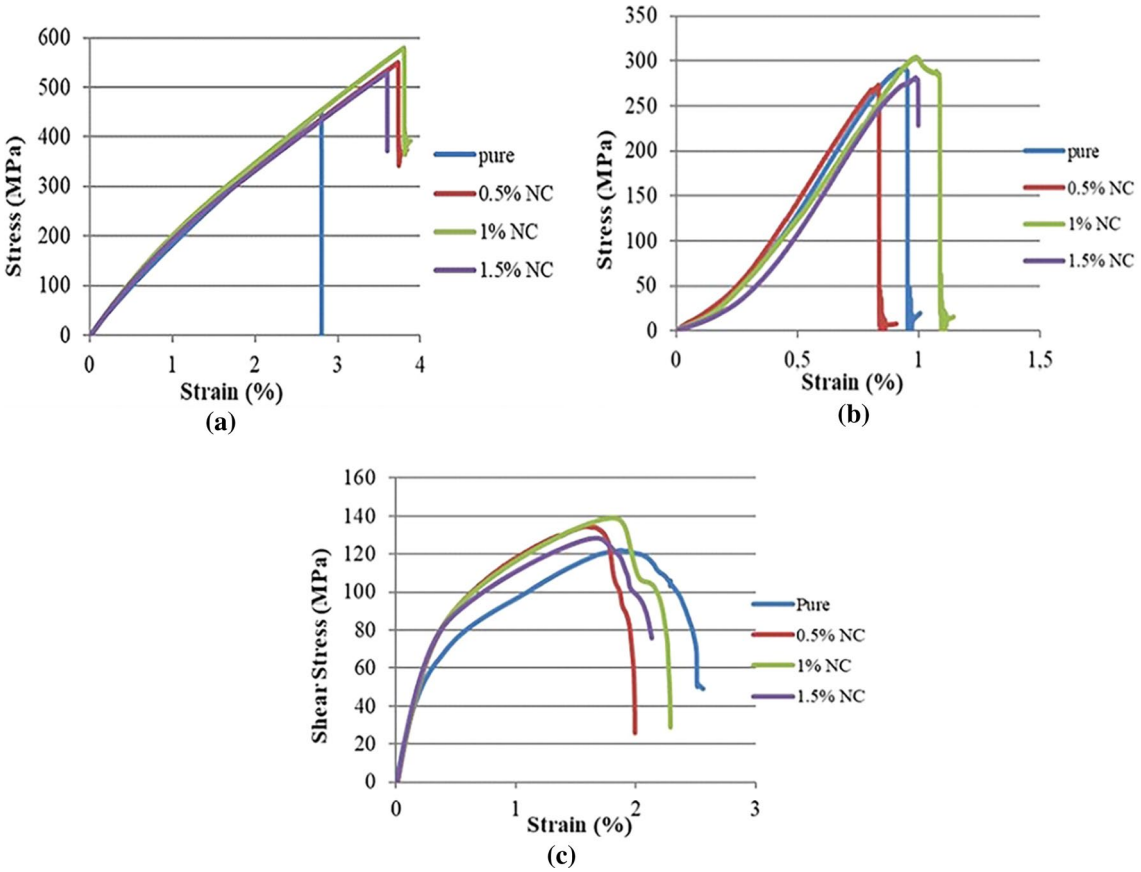


Figure 4 Comparative graphs **a** Tensile test **b** Compression test **c** Shear strength.

values obtained from the pure sample (Table 1, Fig. 4a). The highest values were obtained from 1% NC-added samples. It was determined that the tensile strength value obtained from the pure sample was 26.97%, 31.06%, and 21.73% lower than the values obtained from the samples with 0.5%, 1%, and 1.5% NC-added, respectively (Table 1).

As a result of the compression test, the highest values were obtained from samples with a 1% NC-added (Fig. 4b, Table 1). The compressive strength values obtained from the samples with 0.5% and 1.5% NC-added were 5.82% and 1.71% lower, respectively, compared to the value obtained from the pure sample (Table 1). Shear strength values in all NC-added samples were higher than the shear strength values of the pure sample (Fig. 4c, Table 1). The strength value obtained from the pure sample was 12.29%, 14.30%, and 8.26% lower than the values obtained from the 0.5%, 1%, and 1.5% NC-added samples, respectively (Table 1). As the NC ratio increased, elasticity and shear modulus values increased. However, the highest values were obtained from samples containing 1% NC (Table 1). The elasticity modulus value obtained from the pure sample was 4.35%, 7.35%, and 5.62% lower than the elasticity modulus values obtained from the samples with 0.5%, 1%, and 1.5% NC-added, respectively (Table 1). The shear modulus value obtained from the pure sample was 10.02%, 11.94%, and 7.59% lower than the shear modulus values obtained from the samples with 0.5%, 1%, and 1.5% NC-added, respectively (Table 1). With the addition of NC, the Poisson's ratios to the samples increased. The highest value was obtained from the sample with 0.5% NC-added (Table 1).

As the NC ratio increased, the hardness values of the samples increased. The highest hardness values were obtained from the sample with a 1% NC-added. It was determined that the hardness value obtained from the pure sample was 25.26%, 32.58%, and 25.97% lower than the hardness values obtained from the samples with 0.5%, 1%, and 1.5% NC-added, respectively (Table 1).

It has been stated in the literature that NC has positive effects on tensile strength [9–12, 15, 16, 18,

19, 21, 23–30, 38–42], compressive strength [12, 28], and modulus of elasticity [9, 18, 29]. It has been evaluated in some studies [9, 20, 25, 27, 28, 31] that NC also has a positive effect on hardness. However, in some studies [12, 15, 17, 23, 28, 36, 37], it has been stated that strength decreases when the NC ratio increases. Similar results were obtained in this study, and lower values were obtained from samples with a 1.5% NC ratio.

In this study, the NC effect had a positive effect on all mechanical properties except those mentioned in the literature. The highest values were obtained from samples with a 1% NC ratio (except for the Poisson's ratio).

Results of DMA analysis

In DMA analysis, loss modulus (E″), storage modulus (E′), and tan δ values were obtained. The graph of the change with temperature of the storage modulus of the pure and NC-added samples is given in Fig. 5.

From the pure sample, the storage modulus was obtained at 15,190 MPa. The highest storage modulus was obtained from the 0.5% NC sample with a value of 16,820 MPa, and it decreased to 16,460 MPa in the 1% sample and 15,180 MPa in the 1.5% sample. It is seen that the storage modulus values of the pure sample and the 1.5% NC-added sample are close to each other and show similar behavior (Fig. 5). It has been determined that there is a difference of 0.066% between them. As the temperature increased, the storage modulus value decreased for all samples. Due to the glass transition zone, a large decrease occurred in all samples at about 60 °C. The decrease in storage modulus at 1.5% wt NC loading is thought to be due to agglomeration resulting in stress concentration regions that lead to premature damage [45]. After the glass transition temperature is exceeded, the region where the storage module becomes stable/appears to be at a constant width is the rubber plateau region [26, 47, 52]. The fastest transition to the rubber plateau region was observed in the 1.5% NC-added sample. After the glass transition, the lowest decrease in storage modulus was obtained from 86% of the 0.5% NC-added composite sample.

Figure 6 shows the graph of loss modulus–temperature change that was obtained from composite samples that were both pure and that had NC-added. It was shown that the inclusion of NC increased the loss modulus. This increase in loss modulus can be attributed to an increase in restriction to polymer chain molecular movement by nanoparticles [45, 47, 52]. The loss modulus of the pure sample was obtained at 1934 MPa. The maximum loss modulus was obtained from the 1.5% NC-added sample at 2139 MPa. After the glass transition temperature,

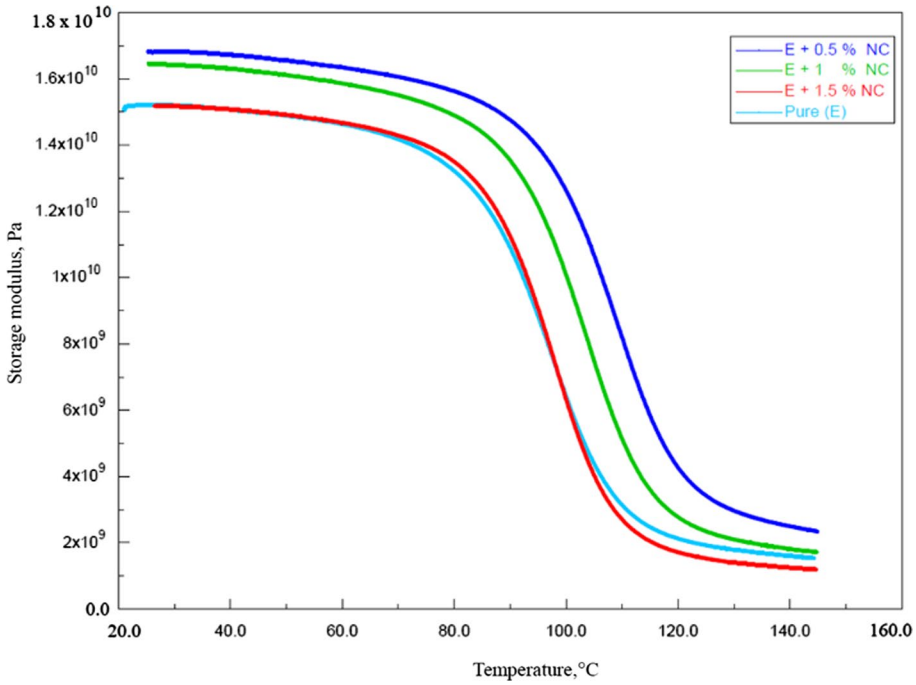


Figure 5 Storage modulus–temperature graph of pure and NC-added samples.

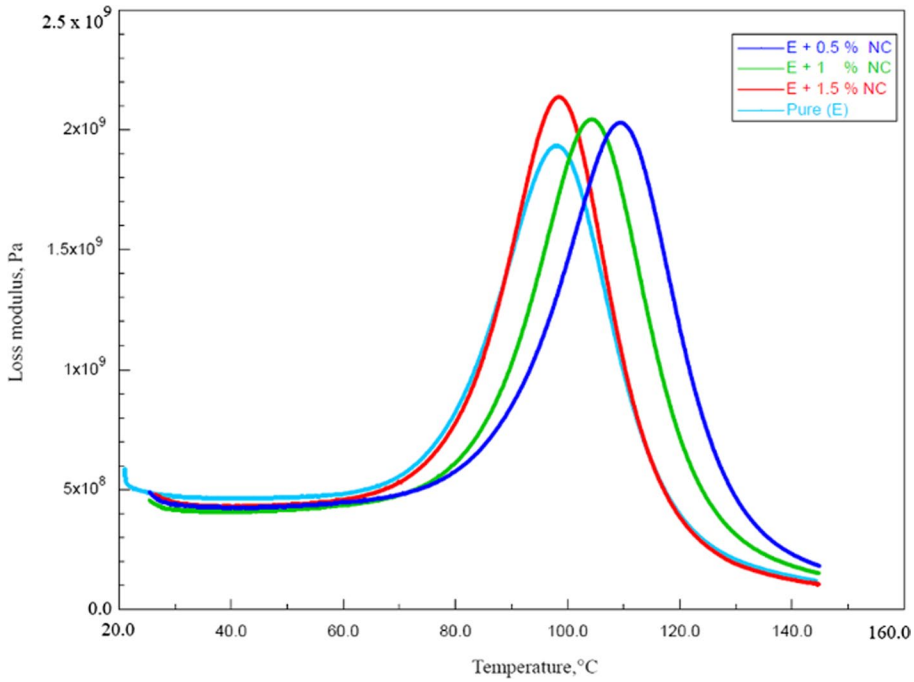


Figure 6 Loss modulus–temperature graph of NC-added samples at different rates.

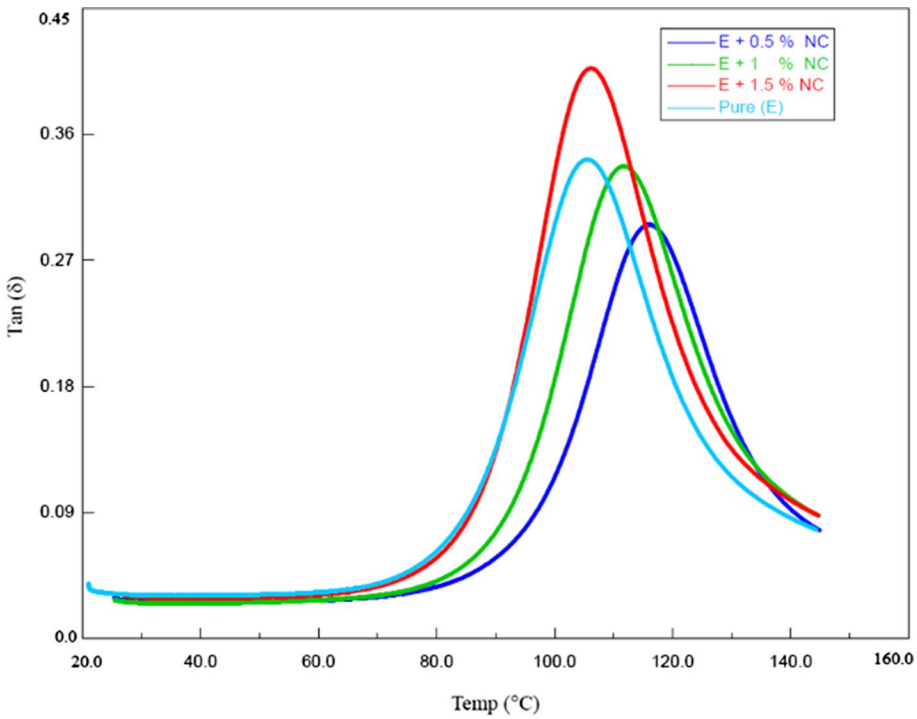


Figure 7 The tan δ-Temperature graph of NC-added samples at different rates.

the rate of decrease of the pure and 1.5% NC-added samples progressed similarly. A similar situation is seen in the storage module graph (Fig. 5). Loss modulus 2030 MPa was obtained from a 0.5% NC-added sample and 2044 MPa from a 1% NC-added sample.

The tan δ-temperature graphs obtained from the samples are given in Fig. 7. Tan δ values were obtained as 0.340 for the pure sample. It increased to 0.407 at 1.5% NC, decreased to 0.337 at 1% and 0.295 at 0.5%. In this case, Moyo et al. [45] stated that NC caused a decrease in tan δ values, but increased tan δ values in some studies.

In this study, it was determined that while the NC ratio was at the maximum rate (1.5%), the tan δ value was also maximum.

By using the Tan δ method, the glass transition temperatures (tg) of the pure, 0.5%, 1%, and 1.5% NC-added samples were obtained as 104.62 °C, 115.92 °C, 111.31 °C, and 105.94 °C, respectively. The addition of NC to the epoxy matrix caused an increase in the glass transition temperature.

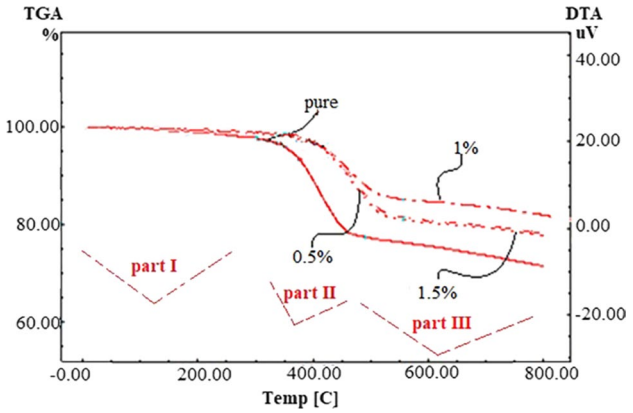


Figure 8 Comparative TGA plots of samples.

Results of TGA analysis

In Fig. 8 depicts the obtained TGA graphs in comparison. The approximate temperature ranges of the samples were determined and examined in three parts.

The first mass loss of the samples (Part I) occurred in the range of 0–300 °C. In this range, moisture, water, and volatile matter loss [21, 45] occurred in the samples. Then, a single-stage decomposition

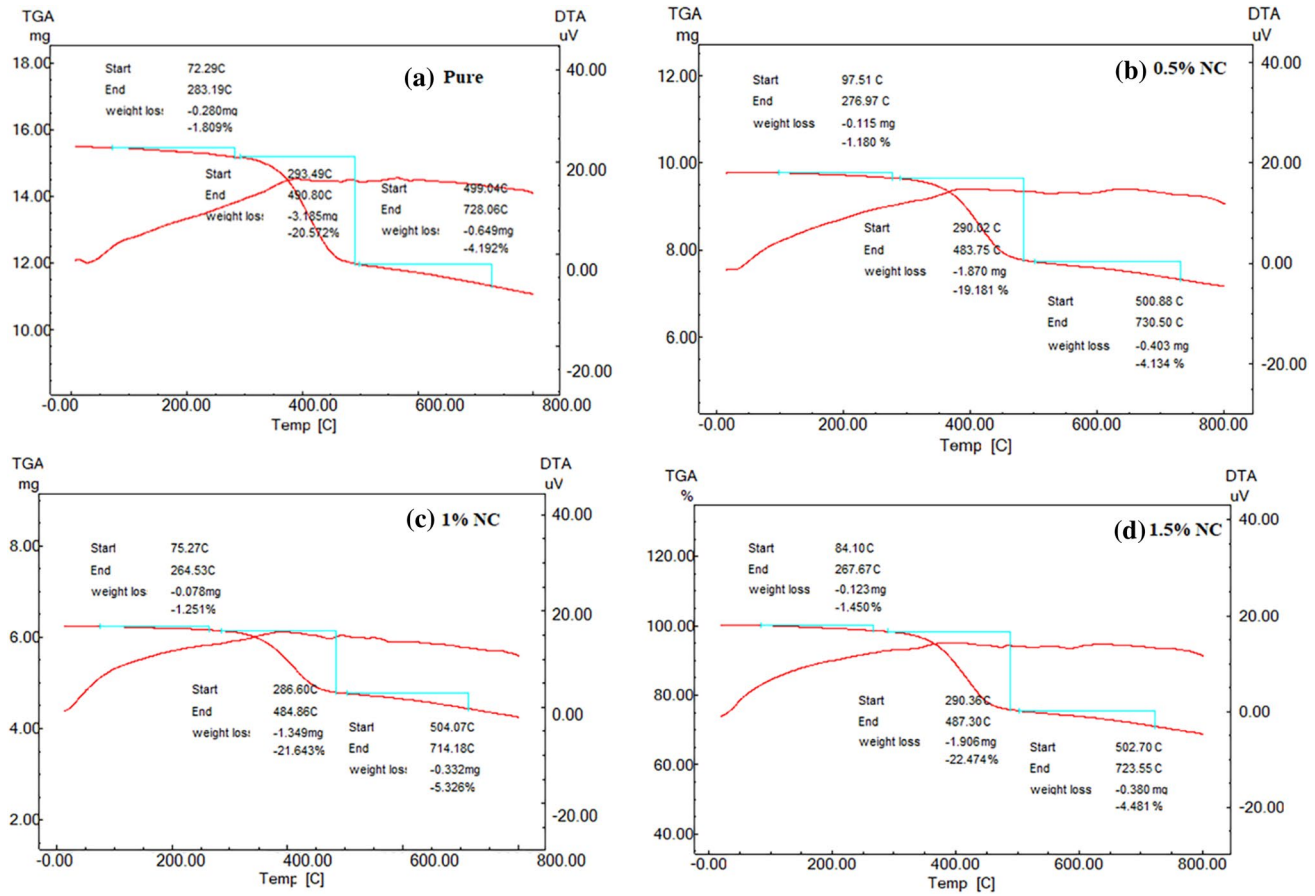


Figure 9 TGA plots of samples.

Table 2 TGA analysis results of samples

Specimen type	Start (°C)	End (°C)	Weight loss (%)
Pure	293.49	490.80	20.512
0.5%	290.92	483.75	19.181
1%	286.60	484.86	21.643
1.5%	290.36	487.30	22.474

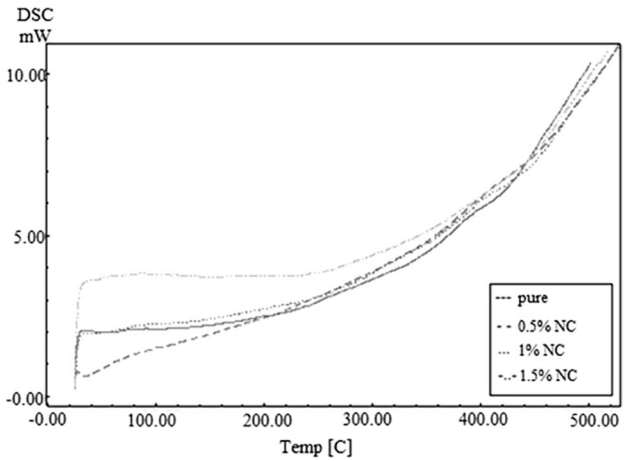


Figure 10 Comparison of DSC plots of samples.

takes place [46], the 300–490 °C interval is seen, and the II. part is described. The highest loss rates were realized in this region. The range of 490–800 °C after decomposition, III, showing residual mass loss, is specified as part. It also shows the variation of residual mass with NC content at 800 °C.

In Fig. 9, the mass losses of the pure (Fig. 9a), 0.5% (Fig. 9b), 1% (Fig. 9c), and 1.5% (Fig. 9d) samples with the initial and end temperatures for all three parts are given.

As an example, in Table 2, II, the initial and final temperatures of the samples in the region and the resulting mass loss rates are given. The highest weight loss was obtained from the samples with 1.5% NC. As the clay ratio increased, the weight loss ratio increased.

Results of DSC analysis

The DSC graphs obtained in Fig. 10 are indicated comparatively. As a result of the analysis, the tg temperatures of the samples increased. The tg temperatures of the samples pure, 0.5%, 1%, and 1.5% NC, were determined to be 98.38 °C, 110.72 °C, 109.76 °C, and 106.71 °C, respectively.

In the literature, it has been stated in some studies that tg decreases as the NC ratio increases [42, 45], while tg increases in some studies [40, 41, 46, 47, 50]. However, this increase and decrease varies according

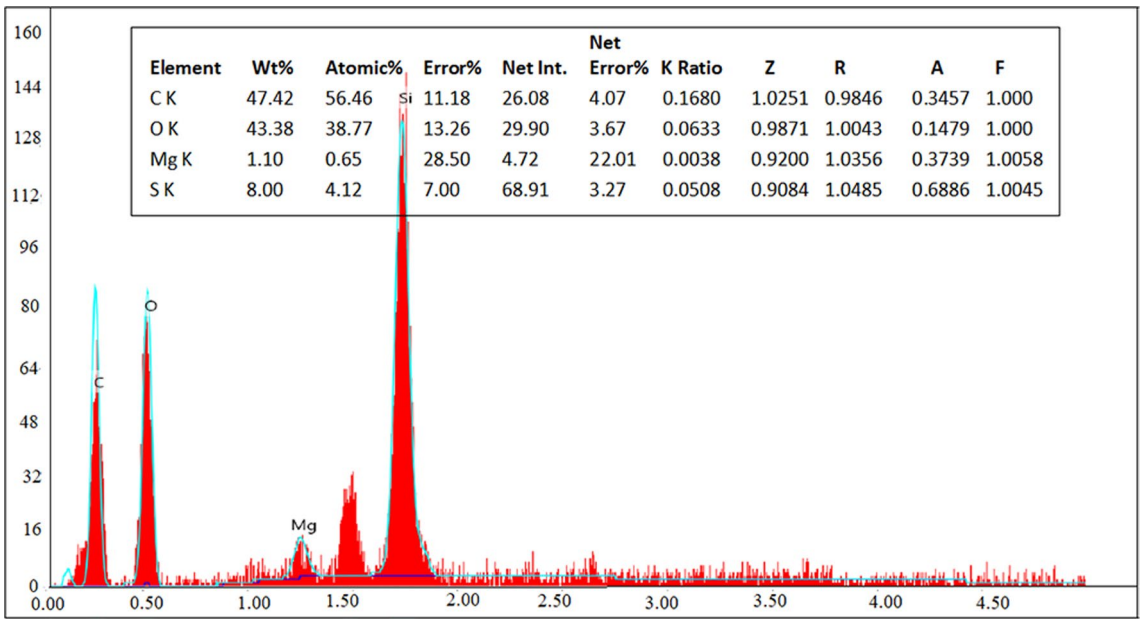
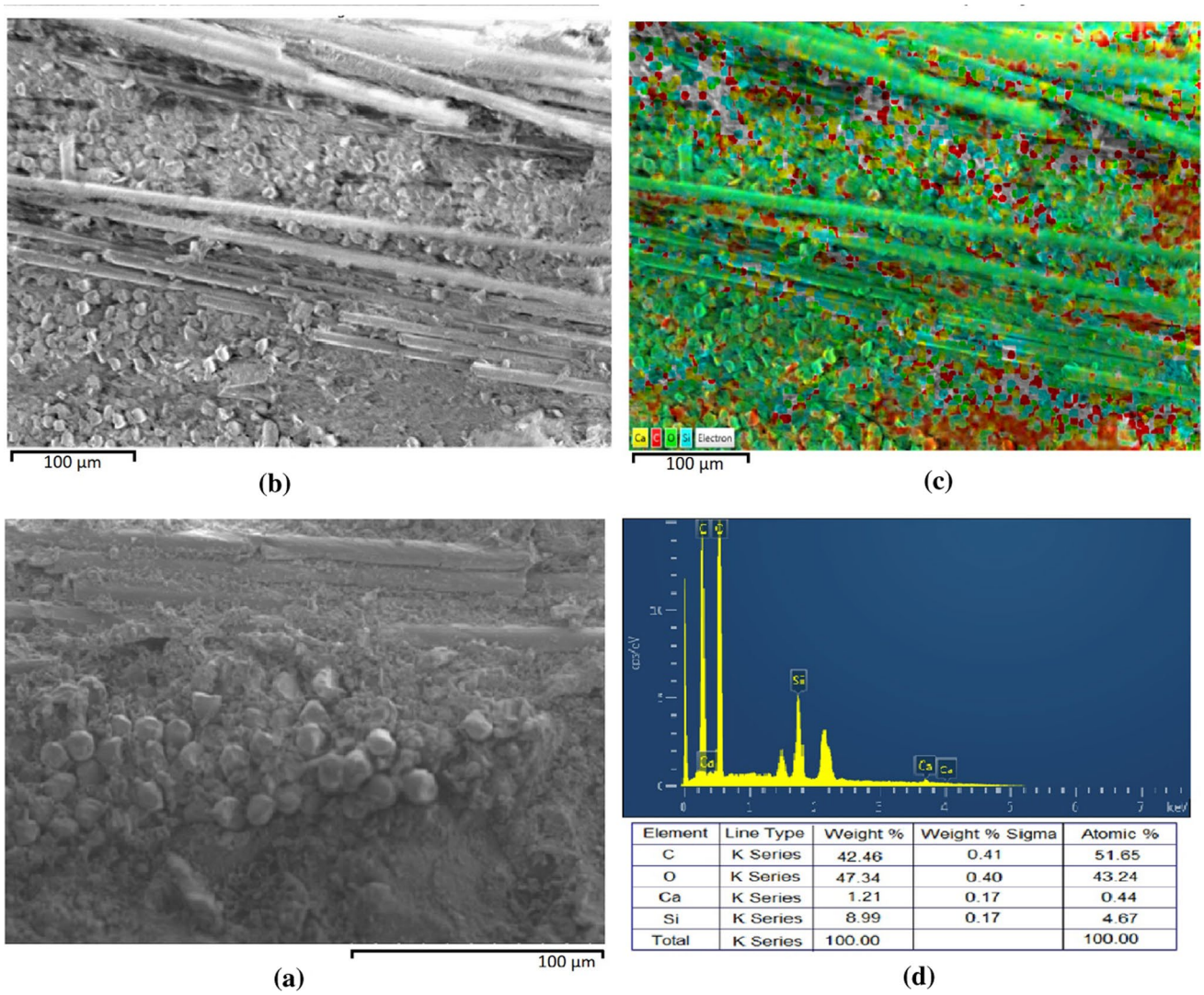


Figure 11 EDS analysis graph for NC.



Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K Series	42.46	0.41	51.65
O	K Series	47.34	0.40	43.24
Ca	K Series	1.21	0.17	0.44
Si	K Series	8.99	0.17	4.67
Total	K Series	100.00		100.00

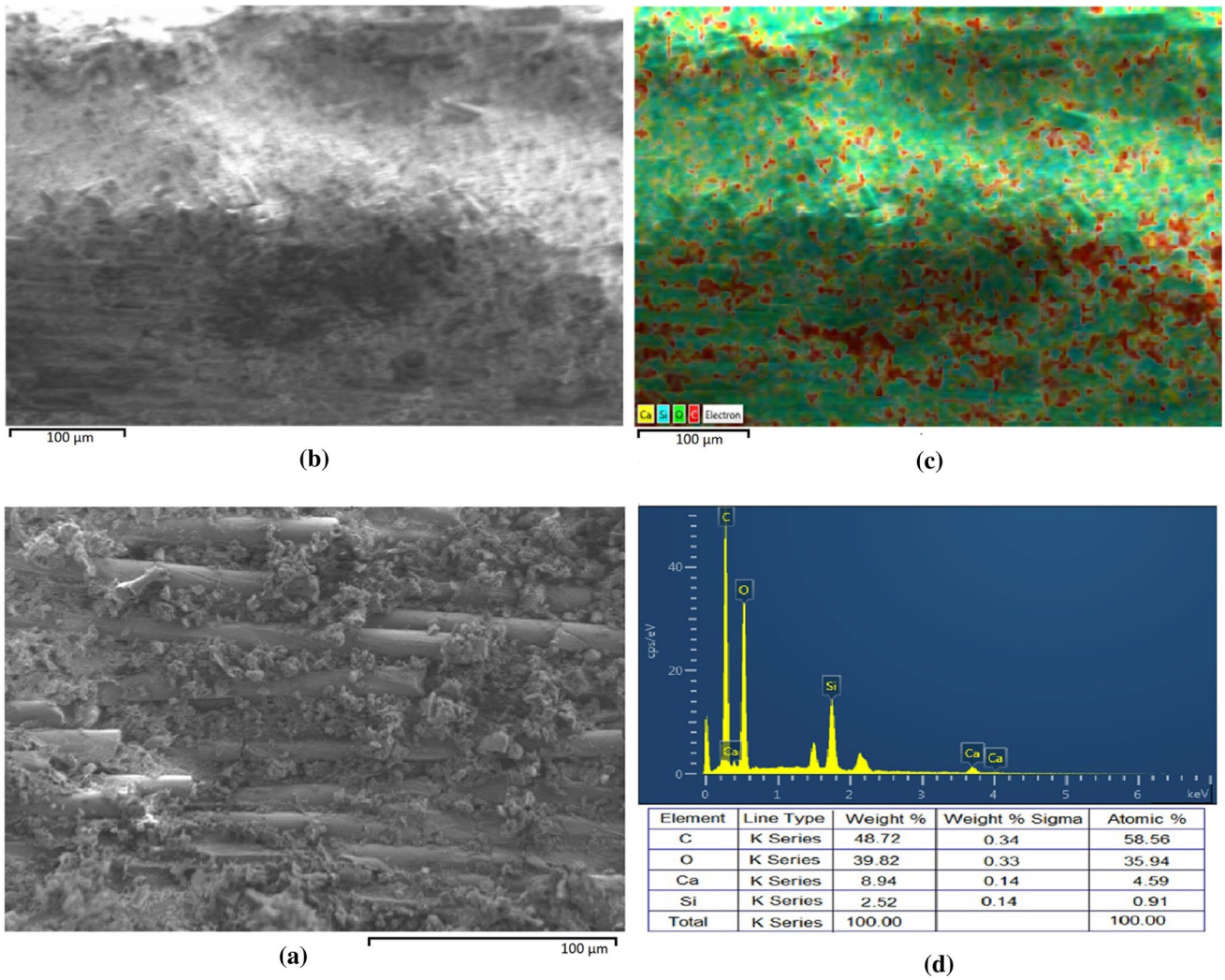
Figure 12 Pure **a** SEM analysis **b** EDS analysis **c** Mapping analysis **d** EDS peak.

to the NC ratio and type used. In this study, the highest and lowest values were obtained from samples with 0.5% and 1.5% NC-added. The values obtained from samples with 0.5% and 1.5% NC-added were 12.54% and 8.47% higher, respectively, than the values obtained from the pure sample. The tg temperatures obtained with DSC and DMA are compatible with each other, and it has been determined that there is a maximum deviation of 6.24 °C and a minimum of 0.77 °C between the obtained values.

SEM and EDS analysis

SEM and EDS analyses of pure and NC-added samples are given in Fig. 12–15. At the same time, EDS analysis results for NC are given in Fig. 11. It is seen that there are Si, O, C, and Mg elements in the clay structure according to their maximum amounts, respectively.

The fiber–matrix structure may be seen in the pure sample's 500 × magnitude SEM picture (Fig. 12a). We can determine the components of the composite structure and their distribution ratios in the map analysis (Fig. 12c) when we look at the EDS regional analysis (Fig. 12b). These distributions were found to exhibit



Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K Series	48.72	0.34	58.56
O	K Series	39.82	0.33	35.94
Ca	K Series	8.94	0.14	4.59
Si	K Series	2.52	0.14	0.91
Total	K Series	100.00		100.00

Figure 13 NC, 0.5% **a** SEM analysis **b** EDS analysis **c** Mapping analysis **d** EDS peak.

ratios of 42.46% C (red), 47.34% O (green), 1.21% Ca (yellow), and 8.99% Si (blue) (Fig. 12 c-d).

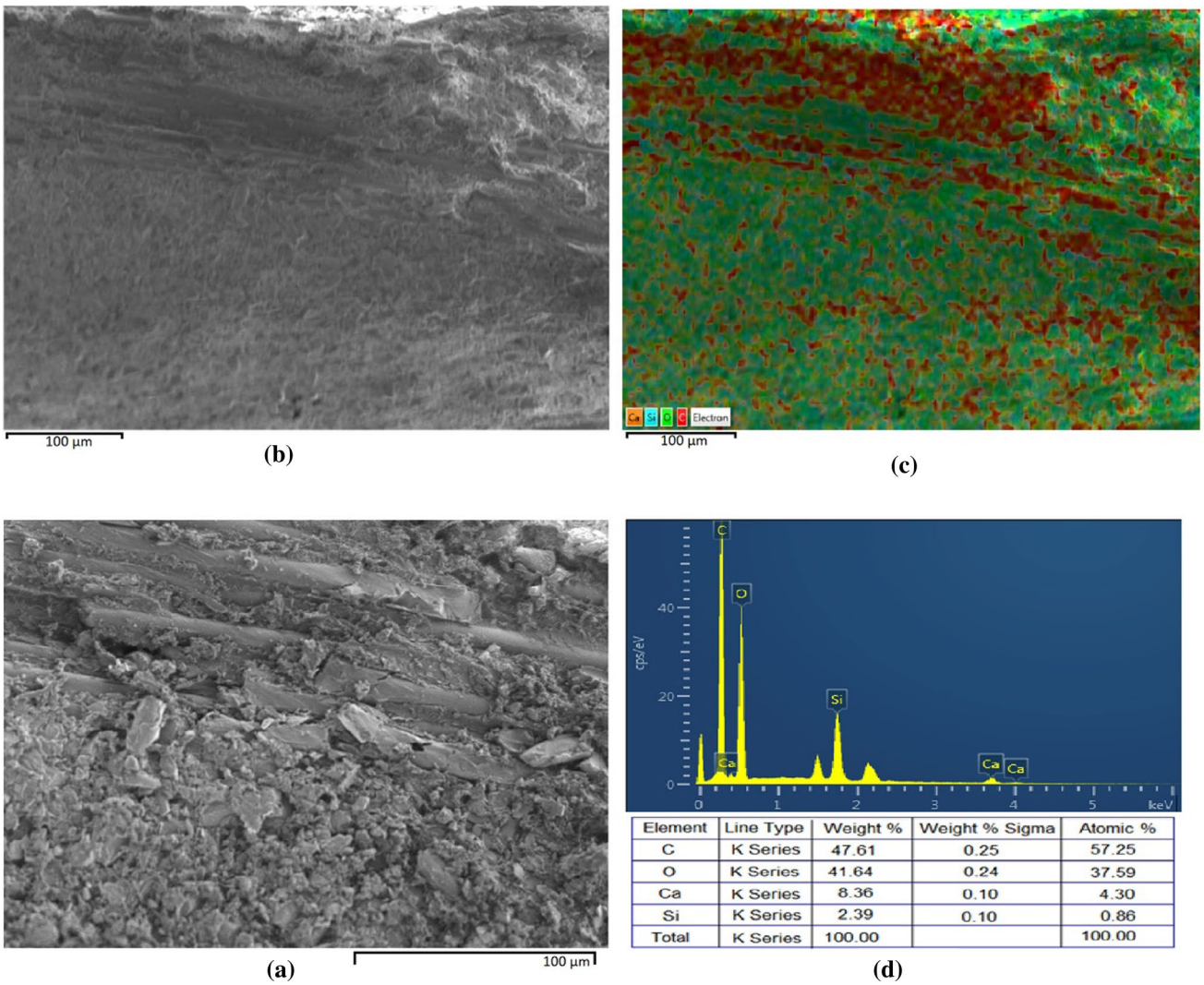
The EDS image obtained with the addition of 0.5% NC shows regional analysis of the element distribution (Fig. 13b) and map analysis (Fig. 13c). Ratios of 48.72% C, 39.82% O, 2.52% Ca, and 8.94% Si are observed when 0.5% NC is added (Fig. 13 d).

SEM and EDS studies that were obtained with 1% and 1.5% NC-added, respectively, are shown in Fig. 14 and Fig. 15. The ratios of 47.61% C, 41.64% O, 2.39% Ca, and 8.36% Si were determined after the addition of 1% NC (Fig. 14 d). 50.32% C, 40.57% O, 1.75% Ca, and 7.37% Si were found at 1.5% NC (Fig. 15 d).

In this study, the distribution of NC in the composite was better clarified by mapping analysis. As seen in Fig. 13c and Fig. 14c, homogeneously dispersed Si atoms were obtained when NC was added. The addition of NC increased the strength of the nanocomposites (Table 1).

Effect on failure modes

In this study, the effect of NC ratios on the failure mode of the samples was investigated after the tensile tests. Images of the failure modes obtained from samples with 0.5%, 1%, and 1.5% NC-added are shown in Fig. 16a–c, respectively.

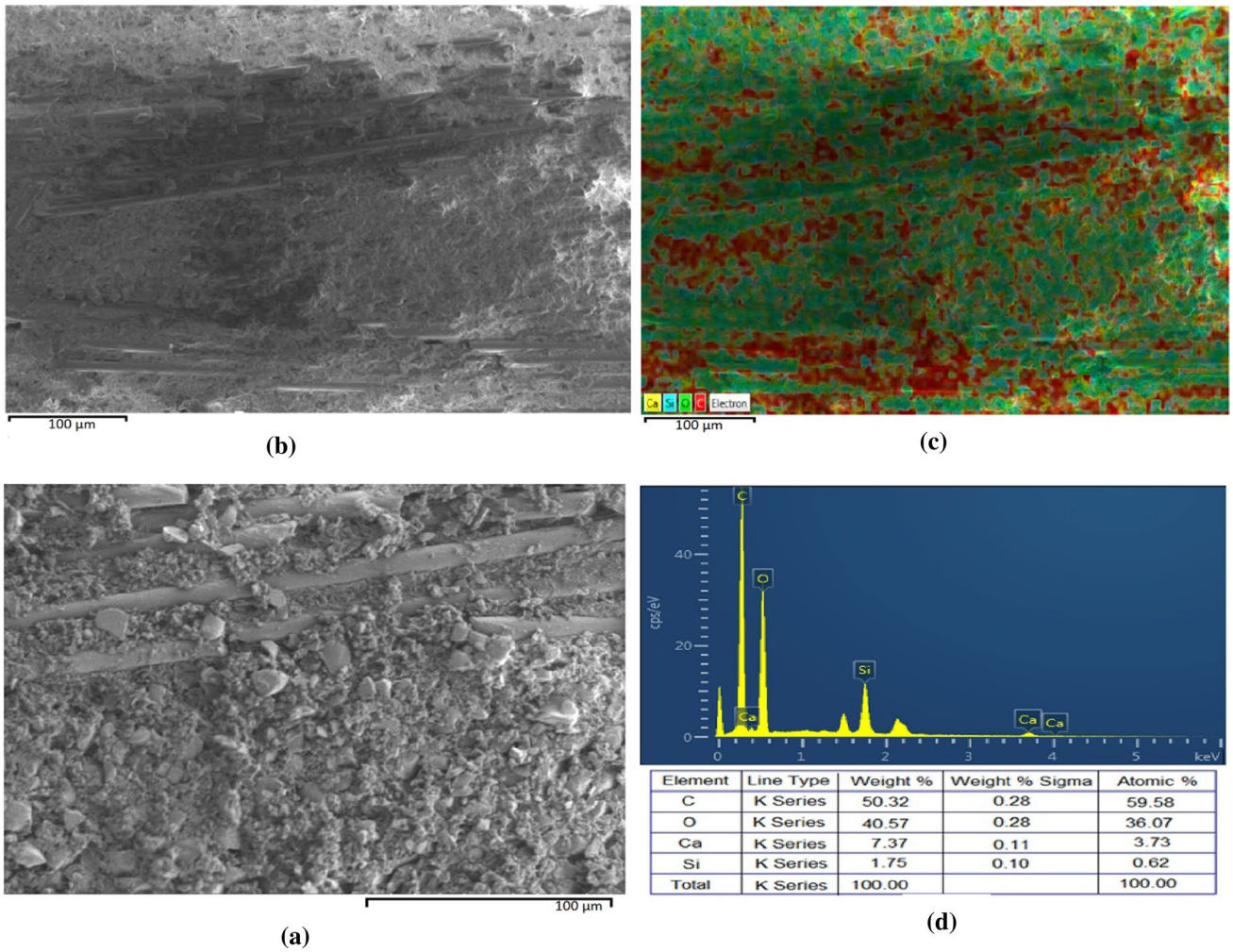


Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K Series	47.61	0.25	57.25
O	K Series	41.64	0.24	37.59
Ca	K Series	8.36	0.10	4.30
Si	K Series	2.39	0.10	0.86
Total	K Series	100.00		100.00

Figure 14 NC, 1% **a** SEM analysis **b** EDS analysis **c** Mapping analysis **d** EDS peak.

From samples with 0.5% NC-added, fiber rupture/fiber failure mode and matrix failure mode were observed. (Fig. 16a). Fiber–matrix failure mode and separation of the fiber–matrix interface were obtained from samples with 1% NC-added (Fig. 16b). In the tensile test of the samples, it was observed that fiber and matrix failure, decomposition of the fibers and conspicuously separation of the fiber–matrix interface occurred in all samples, especially with a nanoclay content of 1.5% (Fig. 16c). The addition of 1.5% NC resulted in the formation of microscale clusters over the NC network (Figs.15b and c). In the samples with 1.5% NC-added, it is seen in Fig. 15b that the surface becomes cloudy and rough. This indicates that the nanocomposites deteriorate due to power transfer from the resin matrix to the NC network [9] and do not exhibit holistic behavior.

Also, Jeyekumar et al. [28] stated in their research that the fiber–matrix interface was separated and broken fibers emerged. Kiran et al. [11] observed that in the fracture morphology of the NC-added samples, the cracks in the matrix propagate throughout the matrix, followed by fiber failure. This behavior supports up both the failure mode result we found in the study and the tensile test results (Table 1), which show that the sample's strength decreased when the NC ratio was 1.5%. At the same time, it shows that when the NC ratio increases, there is no good interface, and interlayer adhesion is not good either.



Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K Series	50.32	0.28	59.58
O	K Series	40.57	0.28	36.07
Ca	K Series	7.37	0.11	3.73
Si	K Series	1.75	0.10	0.62
Total	K Series	100.00		100.00

Figure 15 NC, 1.5% **a** SEM analysis **b** EDS analysis **c** Mapping analysis **d** EDS peak.

Conclusion

In this study, NC-added glass fiber-reinforced epoxy composite plates were obtained by adding 0.5%, 1%, and 1.5% NC by weight. The mechanical and thermal properties of the obtained plates were investigated experimentally. For this purpose, mechanical tests, DMA, TGA, DSC analysis, and SEM–EDS analysis were performed. Additionally, the effect of NC on failure was examined. The experiments' and analysis findings were assessed, and the conclusions obtained are given below.

- The addition of NC had a positive effect on all mechanical properties. The highest tensile strength, compressive strength, shear strength, elasticity modulus, shear modulus, and hardness values were obtained with the addition of 1% NC.
- The values of the glass transition temperature obtained from DMA and DSC analysis have been found to be proportional.
- The addition of NC increased the tg temperature in the samples. The highest and lowest T_g temperatures were obtained from samples with 0.5% and 1.5% NC-added, respectively.
- In the data obtained from TGA, it was found that the weight change with temperature increased with the addition of NC, increasing the decomposition temperatures.

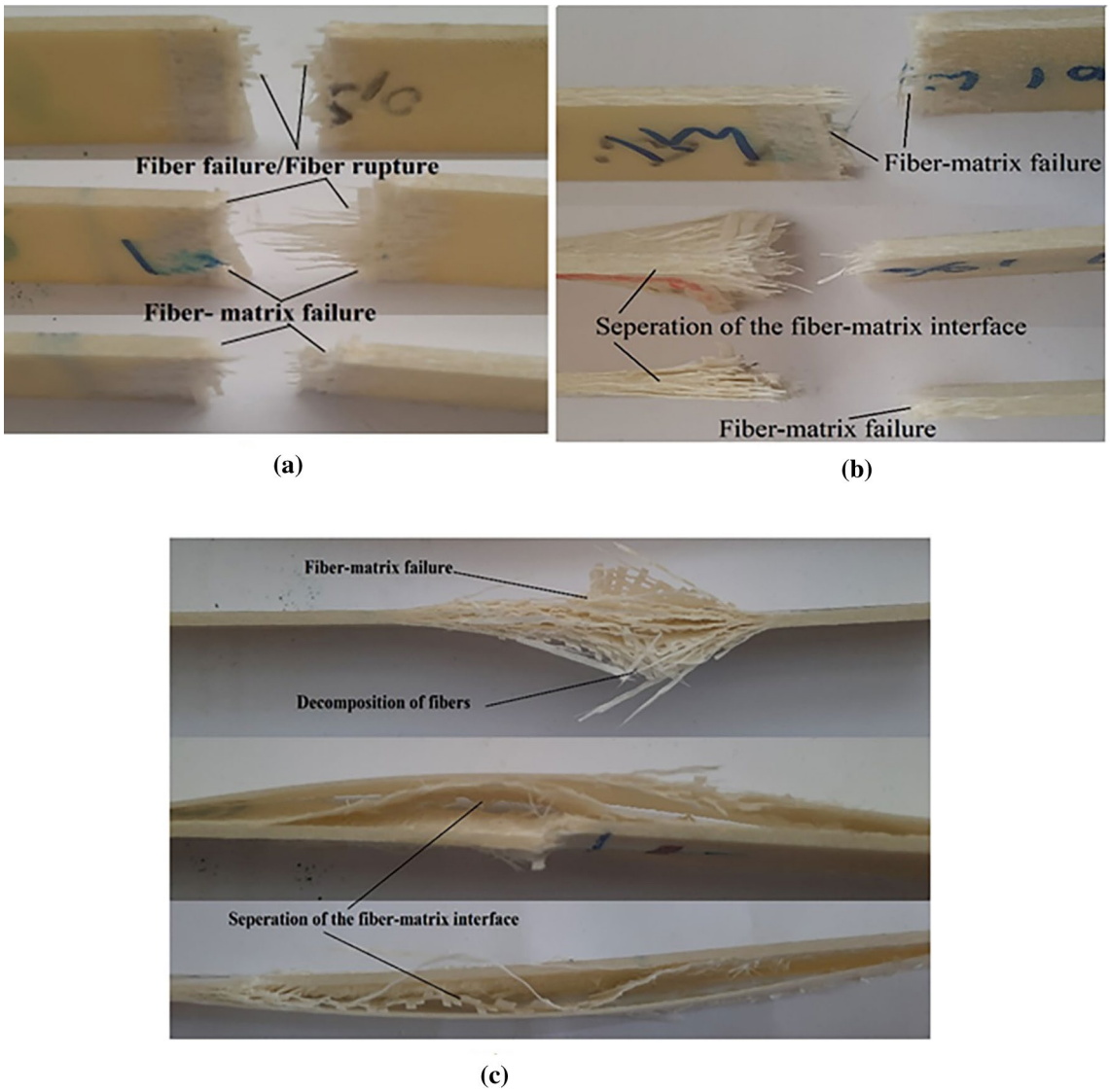


Figure 16 Failure mode of samples with NC-added **a** 0.5% **b** 1% **c** 1.5%.

- The highest E′ value was obtained from the sample with 0.5% NC-added, and it was found to be 10.73% higher than the value obtained from the pure sample.
- The highest and lowest E″ values were obtained from samples with 1.5% and 0.5% NC added, respectively. Value obtained from a 1.5% NC-added sample: 10.60%, 4.65%, and 5.37% higher were obtained as compared to the values from pure, 1%, and 0.5% NC-added samples.
- The highest tan δ value was obtained from the sample with a 1.5% NC-added. It was 19.71% higher than the value obtained from the pure sample.
- Distinctly, separation at fiber/matrix interfaces was observed in samples with 1.5% NC-added.

This study is significant in terms of comprehensively evaluating the influence of nanoclay additions on the mechanical and thermal properties of glass fiber-reinforced epoxy composite materials. In this sense, it is thought that the findings obtained from this study will contribute to the literature.

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Data availability

The datasets generated and analyzed during the current study are available from the corresponding author upon reasonable request.

Declarations

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

Ethical approval Not applicable.

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