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Araştırma Makalesi - Research Article

Mechanical, Tribological, and Biological Properties of Short Carbon Fiber/ Nano Hydroxyapatite Reinforced Hybrid Epoxy Composites

Kısa Karbon Fiber/Nano Hidroksiapatit Takviyeli Hibrit Epoksi Kompozitlerin Mekanik, Tribolojik ve Biyolojik Özellikleri

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ABSTRACT

This investigation intends to examine the mechanical, tribological, and biological properties of hybrid epoxy composites reinforced with nanohydroxyapatite (nHA) and short carbon fiber (SCF). Due to its advantageous mechanical, tribological, and biocompatibility features, the proposed E/SCFs-nHA hybrid composites are meant to be recommended for composite structures that can be used to develop fixation plates used in orthopedic applications. In this study, single-layer hybrid composites reinforced with SCFs and nHA in varying ratios, as well as pure epoxy (E) and epoxy-carbon fiber composites, were all fabricated by hand lay-up method. Tensile tests, 3-point bending tests, and Izod impact tests were performed to investigate their mechanical characteristics. Moreover, the hybrid composite samples were tested for their biological properties in simulation body fluid (SBF). Mechanical and biological properties were found to be enhanced according to the results. Consequently, the hybrid composite (E-10CF-3nHA) of 10% carbon fiber (CF) and 3% nanohydroxyapatite (nHA) performed the best in all tests.

Keywords- Carbon Fiber, Epoxy, Mechanical Properties, Composite Materials

ÖZ

Bu çalışmanın amacı, kısa karbon elyaf (SCF) nano hidroksiapatit (nHA) ile güçlendirilmiş hibrid epoksi kompozitlerinin mekanik, tribolojik ve biyolojik özelliklerini incelemektir. Sahip oldukları mekanik, tribolojik ve biyouyumlulukları ile E/SCF-nHA hibrid kompozitleri, ortopedik uygulamalarda gerekli sabitleme plakalarını üretmek için kullanılabilecek kompozit yapılar için önerilmektedir. Bu çalışmada, SCF ve nHA ile çeşitli oranlarda güçlendirilmiş tek katmanlı hibrit kompozitler, saf epoksi (E) ve epoksi karbon elyaf kompozitler, el yatırması yöntemiyle üretilmiştir. Mekanik özelliklerini incelemek için çekme testleri, 3 noktalı eğme testleri ve Izod darbe testleri yapılmıştır Ayrıca, hibrit kompozit numunelerin biyolojik özellikleri yapay vücut sıvısında (SBF) bekletilerek incelenmiştir. Test sonuçları, mekanik ve biyolojik özelliklerin iyileştirildiğini göstermiştir. Buna

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göre, %10 karbon fiber (CF) ve %3 nano hidroksiapatit (nHA) içeriğine sahip E-10CF-3nHA hibrit kompozit, yürütülen tüm testlerde en iyi sonuçları göstermiştir.

Anahtar Kelimeler- Karbon Elyaf, Epoksi, Mekanik Özellikler, Kompozit Malzemeler

I. INTRODUCTION

Bone fractures are a significant public health issue everywhere. The characteristics and functions of human organs vary. Therefore, replacing the damaged pieces is a big challenge. For the replacement of body parts, biomaterials are the material of choice [1]. In the past, orthopedic implants frequently use metals such as stainless steel, titanium alloys, and cobalt-chromium alloys. However, compared to human bone, these metallic implants are much stiffer. Because the implant takes on the majority of the stress and the bone is fully stress-free, the stress-shielding effect causes bone loss [2]. Additionally, the human body corrodes metals (which have a pH of 7.4 and a temperature of 37 °C) [3]. In addition to implant loss, metal implant corrosion can cause the release of dangerous metallic ions and/or particles. Furthermore, they cannot produce artifact-free radiography images because of their radiolucency.

In an attempt to eliminate the aforementioned problems, polymer matrix composites have been examined in several studies [4]. Many types of polymer-based composite materials reinforced with carbon, aramid, glass, and natural fibers have been studied for bone fracture plating because of their excellent properties such as superior strength-to-weight ratio, non-corrosiveness, tolerability, and radiolucency [5]. The epoxy matrix reinforced with carbon fiber has been employed in a variety of bone treatments including bone plates, spinal cord plates and rods, and total hip replacement [1]. The use of CF in biomedical composites leads to increase in tensile strength and wear resistance of the epoxy matrix composites [4,6]. Many studies have included the investigation of carbon fiber and epoxy composites. Ozsoy et al [7] determined that the CFRE composites were stronger than the GFRE composites based on the results of the tensile and three point bending tests. While Mahalakshmi et al [8] reported that the fabrication of carbon fiber reinforced epoxy composites with different fiber weights is possible by a simple hand lay-up technique, and they had noticed that the mechanical properties of the composites, such as tensile strength, flexural strength, and impact strength, are greatly influenced by the fiber weights.

Hydroxyapatite (HA), a bioceramic material, when added to a polymer, improves the material's bioactivity due to its structural similarities to the mineral part of human bone [9]. Many studies deal with the importance of using hydroxyapatite. Ślósarczyk et al [10] investigated the use of non-coated and coated carbon fiber in HA composites. They discovered that composites having hydroxyl groups on the surface of the fibers had the highest strength.

Several researchers have performed research concerning on carbon fiber and hydroxyapatite reinforced composites. Zhao et al [11] developed HAp/CF/Epoxy and HAp/Epoxy composites. According to the findings, CF-reinforced composites have five times the flexural strength of HAp/epoxy composites. Solomon et al [12] investigated the influence of varying carbon fiber percentages (30%, 35%, and 40%) on epoxy matrix composites and discovered that composites with a carbon content of 35% wt. have improved mechanical strength. Khun et al [13] examined the tribological properties of epoxy composites reinforced with short carbon fibers (SCFs). Based on the results, introducing SCFs to epoxy-based composites increased their tribological properties.

In the literature search, it was seen that carbon fiber/hydroxyapatite-reinforced epoxy matrix composites were produced for multilayer structures or carbon fiber fabrics were used for as reinforcement [11,14,15]. However, although it has been seen that hybrid composites are produced by reinforcing different polymer matrices such as PMMA and HDPE with short carbon fiber/hydroxyapatite, no work has been seen on mechanical, tribological and biological properties of short carbon fiber/hydroxyapatite reinforced epoxy composites. As a result, hybrid composites with carbon fiber and nano hydroxyapatite-reinforced epoxy matrix were produced in this study.

Hand lay-up will be utilized in this study to manufacture composites with varied quantities of carbon fiber and nanohydroxyapatite, and mechanical, tribological, and biological tests will be performed on the created composites. The study's goal is to develop composite structures that can be used to make fixation plates for orthopedic applications.

II. EXPERIMENTAL

A. Materials and Fabrication

Chopped carbon fibers (SCFs) with average dimensions of 3–6 mm and a 7 μm average fiber diameter, were purchased from Dost Kimya, Turkey. Hydroxyapatite particles with an average diameter of 5 to 10 nm were

purchased from Nanografi LLC (Turkey). The epoxy resin and hardener were provided by Power Time (Turkey). Hanks' balanced salt solution used as SBF was purchased from Lonza (Belgium).

To create hybrid composites, various ratios of epoxy were employed as the matrix material, together with short carbon fiber and hydroxyapatite as reinforcement components. The chemical composition of the samples is shown in Table 1 following their coding.

Table 1. Composition of Hybrid Composites

Sample	Epoxy Resin Ratio (wt.%)	Short Carbon Fiber Ratio (wt.%)	Nano-hydroxyapatite (wt.%)
E	100	-	-
E-3CF	97	3	-
E-5CF	95	5	-
E-10CF	90	10	-
E-10CF-1nHA	89	10	1
E-10CF-3nHA	87	10	3
E-10CF-5nHA	85	10	5

The steps in the composite production process were as follows: To create pure epoxy, the epoxy resin was first placed inside a beaker and then mixed with a hardener in a 2:1 ratio. As shown in Figure 1, the manufacturing procedure is carried out using two different silicone molds using the hand layup method at room temperature and without applying any pressure [16]. For composites fabrication, firstly epoxy and additives were mixed and then a quick mixing procedure with hardener was applied followed by casting and rolling with brush. The sample was supposed to be ready for testing 48 hours after the curation process was finished. Following the completion of the curing process, the sample surfaces were polished using 120-grit sandpaper.

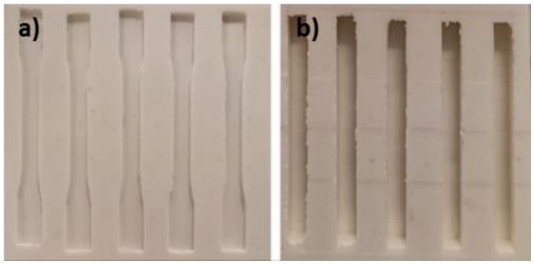


Figure 1. Mold for, (a) Tensile, flexural composite samples and (b) Izod impact composite samples

Due to the inert nature of the carbon fiber surface, the matrix material cannot adhere to it. Surface treatment of the fibers is a suggestion for enhancing adhesion between the fiber and matrix [17]. The surface was treated using the oxidation method. The treatment process was carried out by placing a quantity of short carbon fiber in the oven at 300 °C for 90 minutes.

B. Characterization

The tensile test was performed at room temperature using MTS Landmark servo-hydraulic tensile testing equipment, with a crosshead speed of 2 mm/min in compliance with the ASTM D3039 standard. A 3-point bending test was done following ASTM D790 utilizing a 600 kN Zwick Roell test machine at a loading rate of 2 mm/min. One test was performed on each composite and hybrid specimen, and the results were reported. 3-izod impact tests were utilized to conduct impact testing on each sample following the ASTM D256 standard, and mean values were determined. For these studies, the 450J loading capacity of the Zwick Roell RKP 450 test apparatus was utilized (Figure 2).

Figure 2. a) A Tensile testing machine, b) a Flexural bending test machine, and c) an impact testing machine.

Wear tests were conducted using a UTS tribometer (Figure 3a) under 20 and 40 N loads with an AISI 52100 steel ball for a sliding distance of 100 m, a sliding rate of 30 mm/s, and a stroke of 5 mm. The wear rate was calculated using Archard's coefficient formula (Eq.1). The mean values of each sample were computed.

$$\text{Wear Rate} = W_v / l \quad (1)$$

Where:

W_v= worn volume

l = sliding distance [18].

After completing the wear test, the samples were subjected to a surface roughness test to calculate the worn volume of the samples using a Mitutoyo SJ.410 machine (Figure 3b) according to the ISO 4287-1997 standard [19]. The test was performed on samples subjected to a 40 N and 20 N load, and the reading was collected three times for each sample before calculating the average result. Furthermore, the biological characteristics of the samples were evaluated by immersing them in SBF solution. The chemical composition of simulated body fluid was shown in Table 2. The samples were put in a 37°C oven. After 21 days, the samples were removed, rinsed in water, and allowed to dry at room temperature before being utilized for further analysis, Figure 4. Furthermore, the damaged (broken and worn) surfaces of the samples, as well as those immersed in the SPF, were studied using a scanning electron microscope (SEM) after being plated with gold to increase surface conductivity using a sputter coater (Quorum, Q150R ES Plus).

Figure 3. a) Wear test machine. b) The surface roughness test machine.

Table 2. Chemical composition of simulated body fluid (SBF) [20].

Reagent	Amount
NaCl	8.036 g
NaHCO3	0.352 g
KCl	0.225 g
K2HPO4.3H2O	0.230 g
MgCl2.6H2O	0.311 g
1 M HCl	40 ml
CaCl2	0.293 g
Na2SO4	0.072 g
Tris (hydroxymethyl) aminomethane	6.063 g

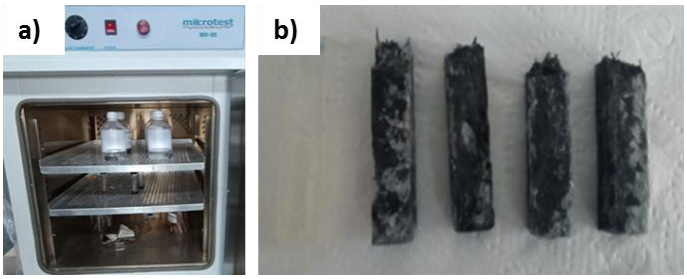


Figure 4. a) Samples in the SBF solution in the oven b) Samples after 21 days.

III. RESULTS AND DISCUSSION

A. Mechanical Properties

- *Tensile Test Results*

Stress-strain results of the samples were presented in Figure 5. The tensile tests showed that the tensile strength of composite materials began to improve with short carbon fiber addition to the epoxy matrix. This can be attributed to CF's axial load-bearing capacity [21]. According to the results, the E-10CF sample (10% wt. CF) had the highest strength. However, it was also detected that there is a reduction in the strain values of composites.

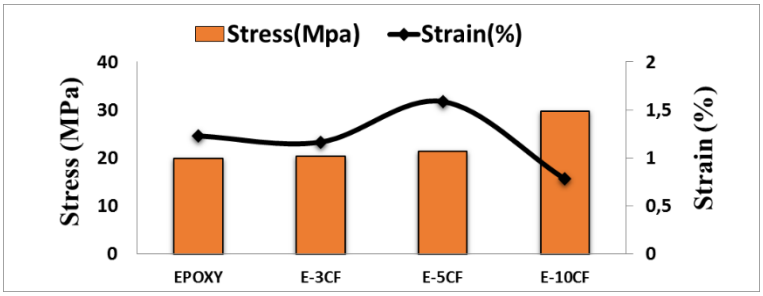


Figure 5. Stress-strain results diagram.

As shown in Figure 6, although the strength of the composites decreased after the surface treatment applied to the carbon fibers, the elongation at break values (strain values) were significantly improved. As a result, short carbon fibers were subjected to a surface oxidation process at 300 °C to improve the weak interface between matrix and epoxy.

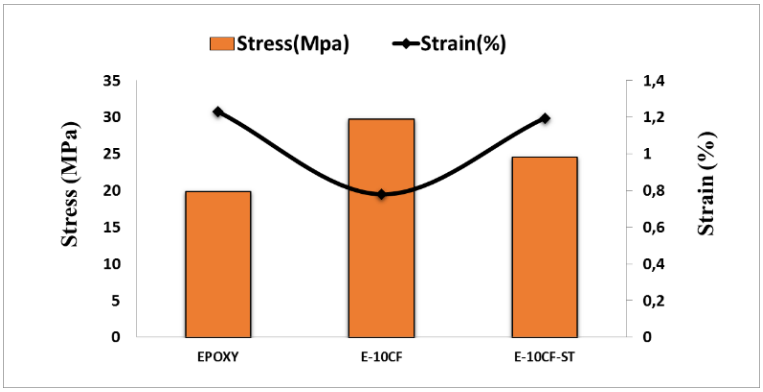


Figure 6. Stress-strain results diagram.

SEM analysis was used to investigate the surface properties of the fibers and the changes in shape caused by oxidation (Figure 6). It can be seen that the surface morphology of 300 °C oxidized fiber is nearly similar to that of untreated fiber. However, it can be said that lines are deeper, and the porosity becomes clearer.

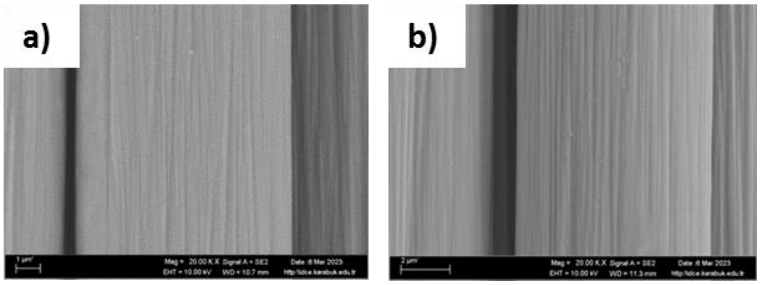


Figure 7. Surface morphologies of carbon fibers: (a) Carbon fiber without surface treatment; (b) 300 °C oxidized fiber

Then, after including nHA, we observe a tiny reduction in the stress values, but the strain values are still maintained, and we observe that increasing the amount of hydroxyapatite, as in the sample E-10CF-5nHA, causes the mechanical characteristics to decline. While the average tensile strength of samples E-10CF-1nHA and E-10CF-3nHA, was 24.3 and 24.65 MPa, respectively, the value of sample E-10CF-5nHA decreased to 18.8 MPa (Figure 8). This is most likely due to a high nHA concentration and nonhomogeneous distribution of nHA in matrix, both of which reduce mechanical properties [22].

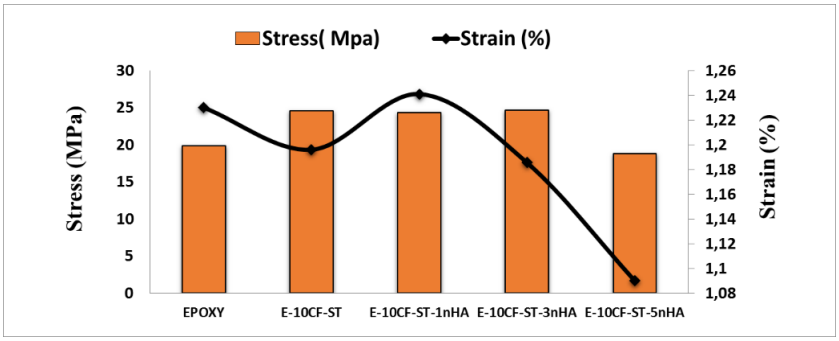


Figure 8. Stress-strain results diagram.

- *Bending Test Results*

In Figure 9, the flexural strength of the hybrid composites was presented. The flexural and tensile strengths of samples have a similar pattern. The bending strength grew together with the carbon fiber content. The rule of mixtures explains this occurrence by showing that flexural strength increases linearly with fiber content [23]. In comparison to pure sample E, the flexural strength of CF10-ST improved (Figure 9). Compared to the 31.18 MPa strength of the CF10-ST sample, the pure E sample exhibited 29.2 MPa in strength. After including nano hydroxyapatite, the hybrid composite that was formed also exhibited improvements in flexural values, as was clear in the samples E-10CF-1nHA and E-10CF-3nHA, which have 31.3 and 34.8 MPa, respectively. With more hydroxyapatite present, as in the sample E-10CF-5nHA, we observe a decline in mechanical characteristics. As previously noted, the mechanical characteristics decreased because of the high concentration of nHA.

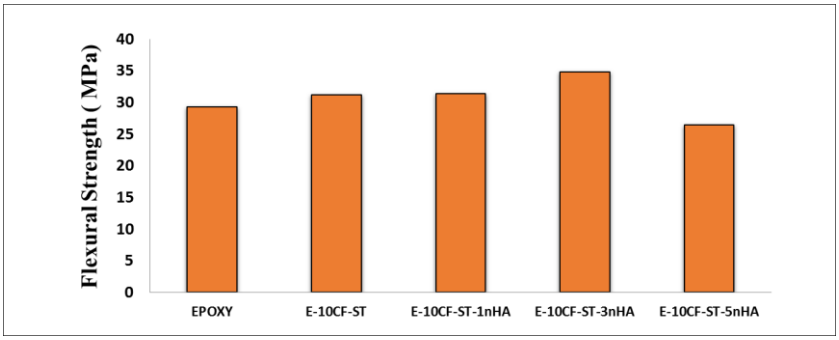


Figure 9. The flexural strength of samples.

- *Impact Test Results*

According to the Izod impact test results, adding SCFs to a pure epoxy matrix directly improves the energy absorption of composites. This could be explained by an increase in the number of SCFs, which increases the load that the reinforcement can properly support as the total fiber interaction surface with the matrix increases [24]. As indicated in Figure 10, while pure epoxy was 0.86 J, the sample with CF10-ST had 1.26 J. Wong et al [24] observed similar results, in which increasing the fiber content resulted in increased energy absorption of composites. After adding hydroxyapatite, firstly, it was seen that there is a slight decrease in impact values, but impact values increased when the proportion of hydroxyapatite in the composite is increased.

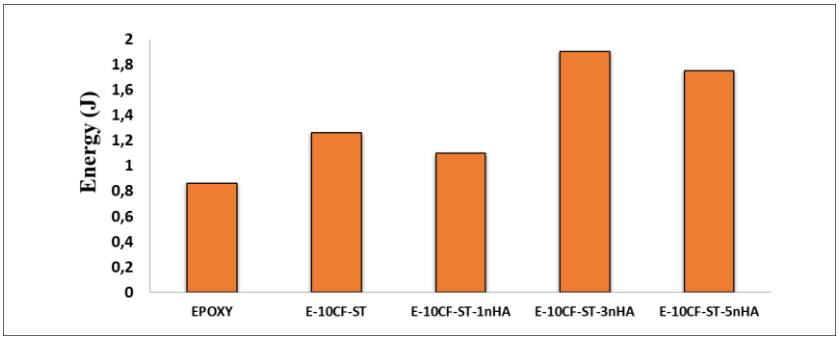


Figure 10. Impact resistance of samples.

SEM examination was performed to determine how the fracture occurred. Agglomeration and debonding (interaction losses between SCFs and epoxy matrix) effects were seen in the CF10-ST composite, according to

Figure 11 (a). Therefore, these might be a factor in the impact value reduction [25]. Furthermore, extra pores formed in the composite as a result of the increased viscosity in the sample caused by the increase in SCFs concentration [26]. In Figure 11 (b), it can be seen that one of the failure modes in fiber-reinforced composite materials, is fiber pullout. Weak bonding is the reason behind fiber pull-out [27]. The appearance of pores and agglomerations is evident in Figure 11 (c and d), which is because when adding different amounts of hydroxyapatite, the mixture becomes more difficult to mix due to an increase in the viscosity of the mixture, and thus the mixture is not well homogenized, increasing the formation of agglomerations as the amount of hydroxyapatite increases.

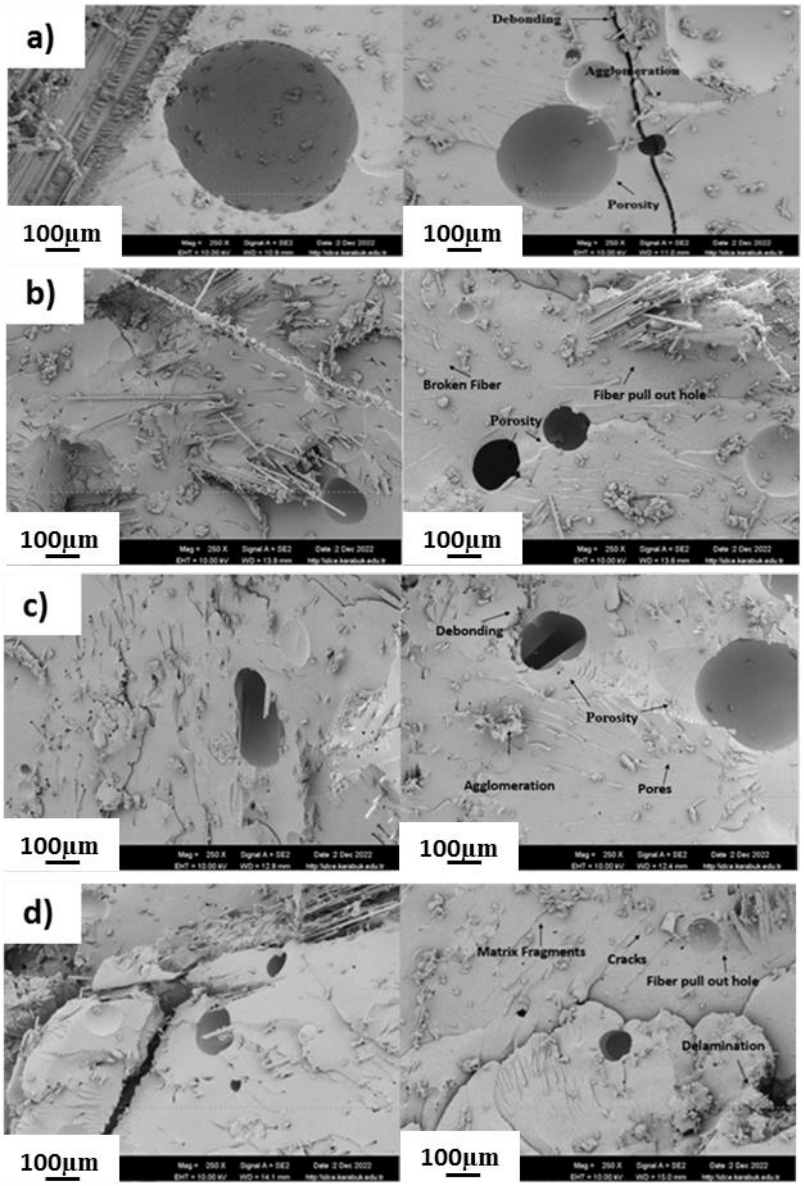


Figure 11. SEM images of broken surfaces of samples (a) E-10CF-ST (b) E-10CF-1nHA, (c) E-10CF-3nHA, and (d) E-10CF-5nHA.

B. Tribological Properties

The tribological characteristics of plastic components are fundamental in engineering applications [28]. The calculated wear test results of the produced samples under loads of 20 and 40 N are shown in Figure 12. The results revealed that as the applied load increased, the wear rates of samples raised. The CF10-ST sample indicated that the wear resistance deteriorated when carbon fiber was added, because the formation of wear debris was increased with the addition of 10% carbon fiber. The higher strength increases the wear resistance properties of composites [29]. However, partial agglomeration of SCFs can cause to decrease in the wear resistance of composite [30]. In similar results, Xian and Zhang discovered that smaller fiber amounts resulted in higher wear resistance [31].

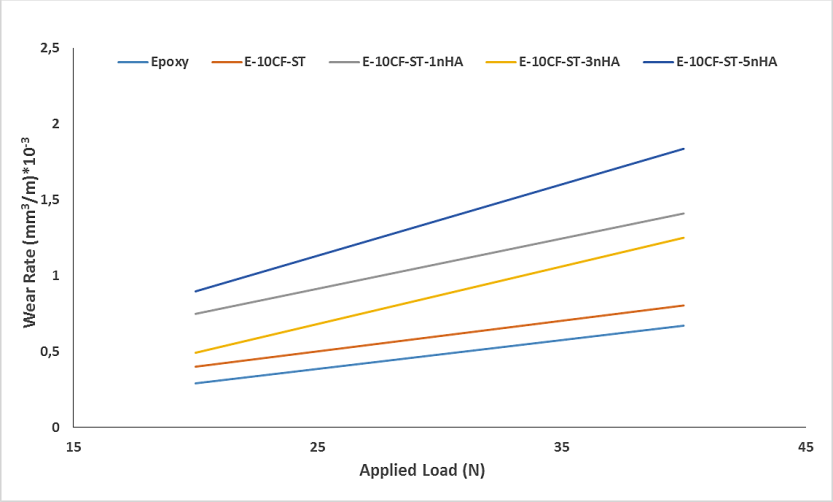


Figure 12. Wear rate under the applied loads.

On the other hand, it was observed that the wear rate increased with the addition of hydroxyapatite. Also, the E-10CF-ST-5nHA sample has the highest wear rate. Furthermore, a scanning electron microscope (SEM) was used to analyze the worn surfaces of both pure epoxy (E) and composite materials. According to the SEM test results, the pure epoxy sample appeared to have layers, as shown in Figure 13 (a), which depicts the formation of layers one by one. However, we notice a significant increase in wear debris formation after adding 10% SCF, Figure 13 (b), an increase in wear debris caused a high wear rate, and when we can see debris, that means the wear mechanism is an abrasive wear mechanism. When hydroxyapatite is added to the composite, we see a remarkable improvement in the wear rate. In comparison to the E-10CF-5nHA sample as seen in Figure 13 (e), the worn surface of the E-10CF-3nHA sample as seen in Figure 13 (d) have relatively smooth surface, this could indicate that this sample has better wear resistance. However, as the amount of hydroxyapatite increases, the sample deteriorates, and agglomerations occur due to the difficulty of mixing resulting from the increase in the viscosity of the composite.

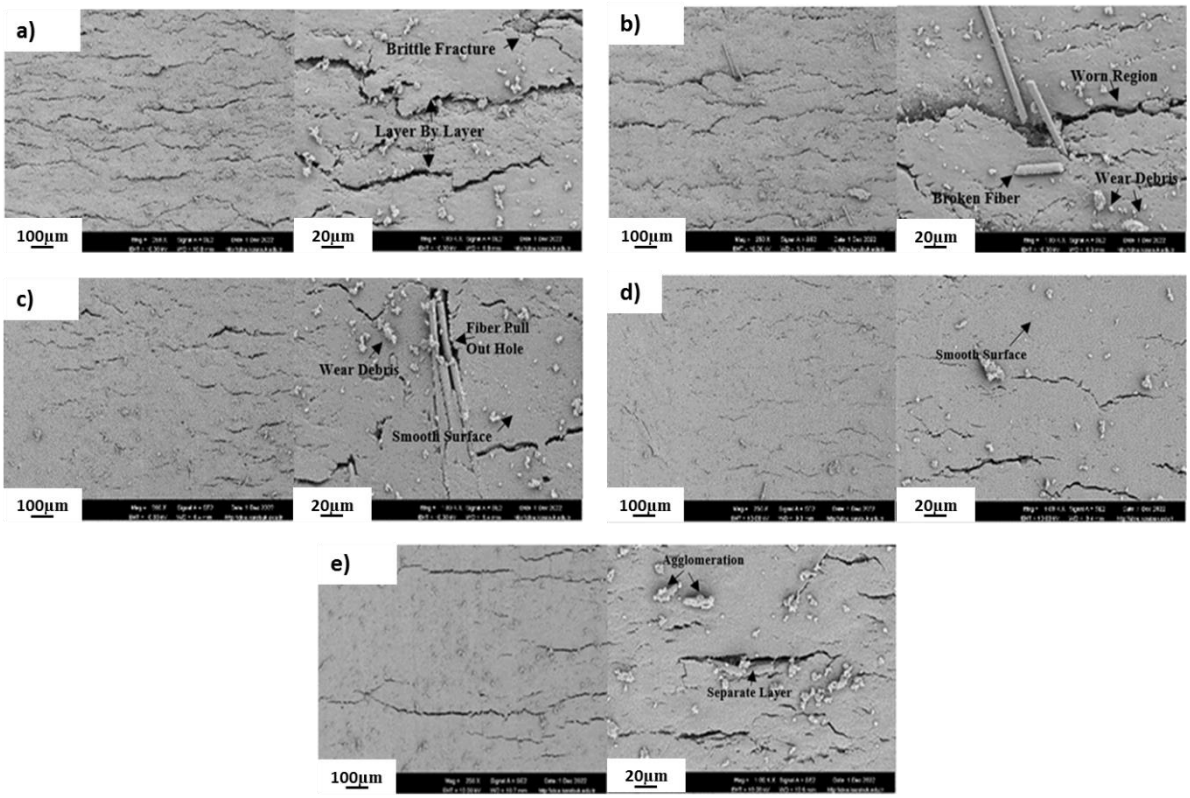


Figure 13. SEM images of worn surfaces of samples, (a) E, (b) CF10-ST, (C) E-10CF-1nHA, (d) E-10CF-3nHA, and (e) E-10CF-5nHA.

C. Bioactivity

To investigate the bioactivity of the hybrid composite, the composite was mixed with hydroxyapatite to improve its bioactivity, and after being incubated in simulated body fluid (SBF) for about 21 days at 37 °C, the apatite that developed on the composites was observed, as shown in Figure 14.

Researchers typically apply the SBF test to determine the ability of test samples to develop an apatite layer on their surfaces [32]. In SBF, the formation of apatite on the surface of a composite can predict in vivo bioactivity (bone-bonding capability) [33]. Since samples Figure 14 (a) and Figure 14 (b) did not contain hydroxyapatite in their compositions, there was no change, as was predicted. According to the findings, adding nHA increased the amount of apatite that was generated, which increased the samples' bioactivity. Kong et al [34] also noted a similar result in their research. Apatite formation increases as the proportion of hydroxyapatite in the hybrid composite, as shown in Figure 14 (c-e).

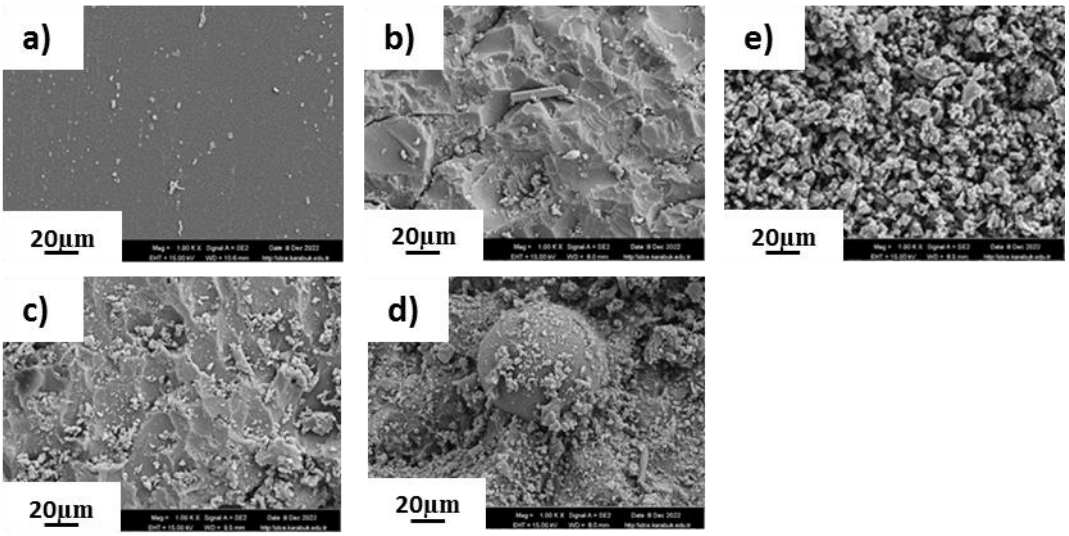
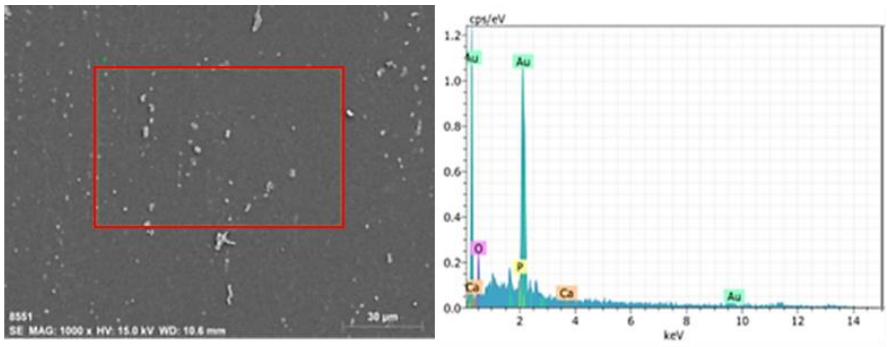


Figure 14. SEM images of samples, (a) E, (b) CF10-ST, (C) E-10CF-1nHA, (d) E-10CF-3nHA, and (e) E-10CF-5nHA, after 21 days in simulated body fluid (SBF).

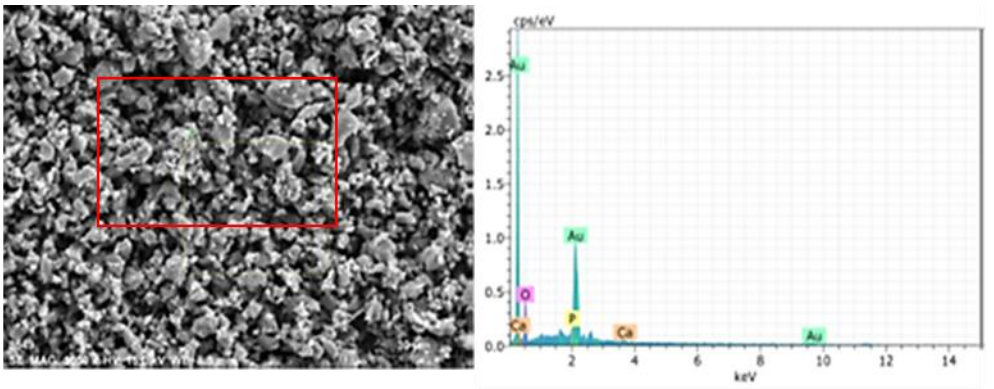
Additionally, apatite particle production on the surface of composites was confirmed using scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX). In contrast to Figure 15, which showed no apatite particles, Figure 16 showed the appearance of apatite particles for the E-10CF-5nHA sample.



Spectrum	Mass Percent (%)			
	O	P	Ca	Au
	6.28	2.08	0.00	47.99

Figure 15. SEM-EDX analysis of pure epoxy after 21 days of immersion in simulated body fluid (SBF).

Due to the absence of nano-hydroxyapatite in the combination, apatite does not form on the surface, as in Figure 15. With the increase in the percentage of hydroxyapatite, there is a noticeable increase in the amount of apatite on the surface of the hybrid composite as shown in Figure 16.



Spectrum	Mass Percent (%)			
	O	P	Ca	Au
	19.20	3.92	1.60	75.28

Figure 16. SEM-EDX analysis of E-10CF-5nHA sample after 21 days of immersion in simulated body fluid (SBF).

IV. CONCLUSIONS

In this study, various ratios of epoxy (E) as matrix material and short carbon fibers (SCFs) and nano hydroxyapatite (nHA) as reinforcements were used to produce composites that can be used to manufacture fixation plates needed in orthopedic applications. According to the results, Sample E-10CF had the highest tensile properties, and after conducting surface treatment at 300 °C to enhance interfacial adhesion between the fiber and the resin, hybrid composites were produced when hydroxyapatite was added. As a result, the E-10CF-3nHA sample performed well in all tensile, bending, impact, and wear tests. Regarding the biological properties, it was noted that as the nHA amount increased, its bioactivity also increased, so the E-10CF-5nHA sample indicated the best bioactivity. In summary, 10% treated short carbon fiber (10CF-ST) and nanohydroxyapatite are added to the composites to enhance their mechanical, tribological, and biological properties. Due to pore formation was not prevented when samples were prepared using the hand lay-up approach, it is believed that the outcomes will be better if these samples are created using a different method such as the compression molding method. The outcomes will be instructive for making composites that can be applied to orthopedic applications.

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