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Research Article

Characterization of C/C Composites Produced Using 3D-preforms by CVD/CVI Method for Biomedical Applications

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ABSTRACT

In this study, carbon/carbon (C/C) composite structures were produced by depositing a pyrolytic carbon matrix around carbon fibers found in a three-dimensional (3D) preform using the Chemical Vapor Infiltration (CVI) method. The preforms used as starting materials were in orthogonal fiber geometry and 3D carbon fiber knitting structure. The CVI process performed in the CVD (Chemical Vapor Deposition) device was carried out at 1250 °C, under 2 mbar pressure, with a methane gas flow rate of 2.0 lt/min, for a total period of 312 hours gradually applied in an inert atmosphere consisting of argon and nitrogen gases. The produced block piece was processed to obtain small test samples; tensile and three-point bending tests were applied to the pieces, and their densities were measured. Samples were subjected to in-vitro biodegradation tests in 0.9 % isotonic sodium chloride solution by weight at 37 °C for a total of 21 days. The density and apparent porosity of the produced samples were measured to be 1.395 g/cm³ and 13.424%, respectively. The tensile strength and bending strength of the produced C/C composites were determined to be 252.5±6.20 MPa and 236.6±25.7 MPa, respectively. At the end of 21 days, the biodegradation ratio of C/C composites was calculated as 0.0095%.

1. Introduction

Carbon fiber is a material of great interest in various applications in the industry due to its extraordinary properties. This material, characterized by its high carbon content and fibrous structure, boasts a low density and outstanding mechanical characteristics, making it a highly desirable choice for a wide range of applications [1].

Carbon fiber fabrics are versatile materials that vary based on their application areas, manufacturing methods, and mechanical properties. In addition, they are characterized by weave types, weight per square meter, product manufacturing techniques, fiber orientation, and the polymer materials used. These fabrics are made into preforms on weaving machines using methods such as braiding, stitching, and pinning, depending on the weave type. Preforms for composite materials can be fabricated using various textile technologies, including weaving, knitting, and braiding, to produce one-dimensional (1D), two- dimensional (2D), or three-dimensional (3D) structures [2].

1D carbon fiber fabrics consist of fibers aligned in a single direction and are typically used in applications requiring high tensile strength. These fabrics provide maximum durability in the direction of fiber orientation, making them suitable for biomedical applications where localized load-bearing and high tensile strength

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are critical, such as in tendon or ligament repair materials. 2D carbon fiber fabrics, with fibers aligned in two directions (warp and weft), are produced using weaving or braiding techniques. These fabrics deliver high strength and stiffness in two-dimensional planes, which makes them applicable for prosthetics or load-bearing implants requiring planar strength and flexibility [3]. Among carbon fiber fabrics, 3D woven preforms offer distinct advantages over traditional 2D fabrics. 3D carbon fiber fabrics, composed of fibers aligned in three directions (warp, weft, and z-axis), are produced using advanced 3D weaving techniques [4]. Furthermore, novel 3D weaving techniques have been developed to continuously and rapidly produce 3D woven fabric preforms, addressing common issues such as delamination and fiber buckling found in 2D laminated composites [5]. These fabrics provide multidirectional load-bearing capacity, superior mechanical properties, enhanced durability, and structural integrity. In biomedical applications, their ability to evenly distribute mechanical stresses while maintaining biocompatibility makes them ideal for complex implants, bone scaffolds, artificial ribs, and surgical tools [6].

In the selection and design of carbon fiber preforms, not only physical, chemical, and mechanical properties but also biocompatibility, which is crucial in biomedical applications, must be considered. For instance, scaffolds must support cell adhesion, proliferation, and tissue integration in implants designed for bone replacement while withstanding mechanical loads [7]. With their exceptional strength, durability, and structural integrity, 3D woven carbon fiber fabrics meet all these requirements specified. Their capacity to accommodate multidirectional forces without compromising biocompatibility ensures they meet the rigorous demands of applications where compatibility with biological tissues is as critical as mechanical performance [8].

Pyrolytic carbon is a synthetic biomaterial first introduced in the 1960s and known for its outstanding biocompatibility and mechanical properties [9]. These unique properties have made it a fundamental material for many medical applications, especially implantable devices such as heart valve prostheses, orthopedic implants, and vascular grafts [10]. The biocompatibility of pyrolytic carbon stems from its ability to resist immune rejection and support tissue integration, making it a reliable choice for long-term implantation [11]. One of the key factors contributing to its widespread adoption in the medical field is its ability to endure harsh physiological conditions within the human body. Pyrolytic carbon exhibits excellent corrosion, wear, and fatigue resistance, even under continuous mechanical stress and exposure to bodily fluids [12].

Its surface properties, including smoothness and hydrophobicity, also minimize platelet adhesion and thrombus formation, making it particularly suitable for cardiovascular devices [13]. Pyrolytic carbon is typically produced through the CVI process, a technique wherein amorphous carbon is deposited from solid or gaseous hydrocarbons onto the surface of a substrate, such as graphite or carbon fiber woven preforms [14]. This process not only ensures precise control over material thickness, density, and microstructure but also enables the production of materials with enhanced durability and performance. Layered structures in pyrolytic carbon can be fabricated via the CVI process, allowing the material to exhibit anisotropic mechanical properties. This tailored anisotropy, characterized by high strength and flexibility, renders pyrolytic carbon particularly advantageous for specialized applications, including those in the biomedical field [15, 16].

C/C composites having the capacity to withstand temperatures up to 3000 °C without melting are materials known for their thermal stability and resistance to high temperatures. Their non-flammability, high corrosion resistance, and oxidation stability ensure durability and reliability, even in chemically aggressive or high-stress environments [17]. In addition to these excellent properties, C/C composites have unique physical and mechanical properties that enable their use as biomaterials [18]. These composite materials can absorb and convey magnetic and electrical energy with high efficiency, exhibiting greater mechanical strength than steel, all while being considerably lighter and maintaining a strong strength-to-

weight ratio. Their superior fatigue and wear resistance further enhance their suitability for biomedical applications, particularly in load-bearing or high-friction environments [19].

CVI, Polymer Impregnation and Pyrolysis (PIP), which are the basic production methods of C/C composites, differ in infiltration mechanisms and lead to dissimilar material properties. CVI utilizes gaseous precursors for adequate densification, while PIP uses liquid polymer precursors to offer a more scalable yet less homogeneous process [20]. In the CVI process, a gaseous hydrocarbon precursor is introduced into a heated chamber containing a porous carbon preform. At high temperatures, the precursor decomposes and deposits pyrolytic carbon on the internal surfaces of the preform. This controlled, layer-by-layer densification minimizes voids and creates a high-purity carbon matrix with superior mechanical strength, thermal stability, and uniformity [14]. With its ability to produce defect-free, structurally reliable materials, the CVI method is especially valuable for biomedical applications. These methods enable precise control over the material's microstructure and properties, ensuring biocompatibility, structural integrity, and longevity in biomedical settings [21, 22]. Figure 1 presents the manufacturing flowchart of C/C composites by the CVD/CVI method.

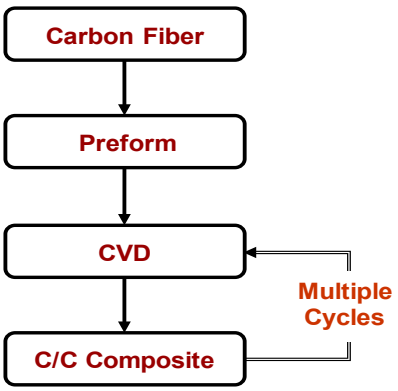

Figure 1. The manufacturing flowchart of C/C composites by the CVD/CVI method [3]

This study aimed to produce C/C composites using 3D carbon fiber preforms via the isothermal CVI method, to characterize the produced composites and reveal their potential for use in biomedical applications through in-vitro biodegradation tests. There is not enough information in the literature regarding using these composites in biomedical applications. The applied biodegradation tests will reveal the potential of the produced composites to maintain stability and structural integrity over extended periods in biological environments, thereby making a significant contribution to the literature.

2. Experimental

2.1. Production of C/C composite samples

In this study, the preform, having a 3D carbon fiber weave structure with orthogonal fiber geometry supplied by a domestic institution (3DWovens Composite Ltd.), was used. The first step applied before the CVI process is removing the polymeric sizing coating on the carbon fiber filaments. Subsequently, the initial weight of the preform was measured and subjected to a total of six CVD/CVI processes, varying in duration between 24, 48, and 72 hours each, at 1250 °C and 2 mbar pressure in a CVD system on an industrial scale (Figure 2). CVI processes were carried out with a methane gas (purity: 99.5%) flow rate of 2.0 lt/min in an inert atmosphere consisting of a combination of argon (purity: 99.999%) and nitrogen (purity: 99.999%) gases.

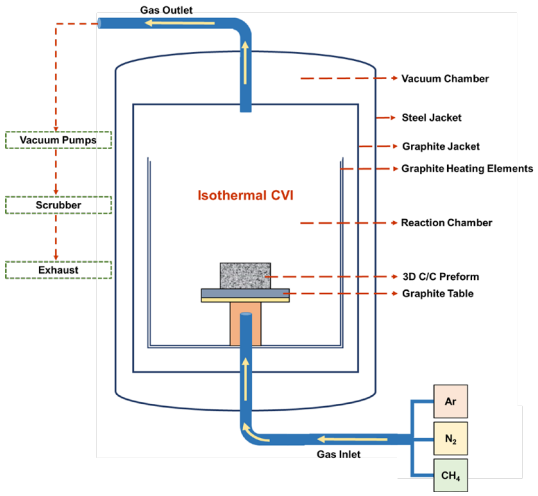

Figure 2. Schematically illustration of the CVD system utilized to perform CVI processes

At the end of each cycle, the CVD apparatus was turned on, and the preform was removed to note the weight increase resulting from pyrolytic carbon matrix deposition. At the end of 6 cycles, a total of 312 hours of CVD/CVI process was applied to the preform.

After a certain period in CVI processes, the fringes on the sample were trimmed and removed from the sample surface to increase the efficiency of subsequent CVI processes. Therefore, the change in weight ratio was revised by calculating the new initial weights. The CVI processes applied to the preform and the weight changes occurring in the preform are presented in Table 1.

Table 1. The CVI processes applied to the preform and the weight changes occurring in the preform

Process	Process Cycle Number	CVI Process Time (h)	Preform	
			Weight (gr)	Weight Change (%)
Initial weight		---	198.5	---
Initial weight after sizing*		---	191.5	---
	1	48	243.5	27
	2	72	290.9	52
Removal of fringers**		---	288.5	---
Initial weight (Revision – 1)*		---	189.9	---
	3	24	298.8	57
	4	72	320.1	69
Trimming***		---	305.3	---
Initial weight (Revision – 2)***		---	182.7	---
	5	48	319.7	75
	6	48	324.1	77
Trimming ****		---	144.7	---
Total:		**312**		

NOTES:

Sizing* : The weight values measured after removing of the polymeric coating on the raw fibers were taken as the initial weight in the calculations.

Removal of Fringes** : To increase the CVI efficiency, the fringes on the sample were cut with scissors. The proportional decrease in weight was applied to the initial weight to find the revised initial weight.

Trimming*** : Cutting from the edges with a milling machine to obtain a smooth prismatic structure. The proportional decrease in weight was applied to the initial weight to find the revised initial weight.

Trimming**** : Giving the samples their final shape by cutting with a milling machine.

As seen in Table 1, a 77% weight increase, indicating carbon accumulation, occurred in the 3D preform densified using the CVI method. The visual presentation of the C/C composite block obtained from the studies is presented in Figure 3. The test pieces required for characterization studies were extracted from this block.

2.2. Characterization of C/C composites

The phase composition of samples was determined using a PANalytical X'Pert PRO MPD model diffractometer operating with Cu-Kα (λ = 1.54056 Å) radiation. The XRD patterns of samples were obtained at 45 kV and 40 mA. The measurements were carried out in the 2–70° 2θ scanning range, with a measurement step of 0.03° 2θ and a duration of 10 minutes.

The morphologies of samples were examined by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS) (JEOL-JSM 6510 LV). The density and apparent porosity of samples were measured using the Archimedes method in distilled water.

Tensile tests of C/C composites prepared with a CNC lathe was carried out using a ZWICK Z250 Universal tensile device by ASTM C1275 standards at room temperature and a 2 mm/min speed. The initial gauge length (L_0) for strain measurement was 10 mm, measured using an extensometer. Test results were determined by calculating the arithmetic mean of the results of tests applied to three different samples.

Three-point bending tests of C/C composite samples prepared with a CNC lathe, with dimensions of 4x3x40 mm, were performed by ASTM C1161 standards, using a 5 kN load cell on a ZWICK Z250 Universal tensile device at a test speed of 1 mm/min at room temperature. The

distance between the supports and the diameter of the supports was 30 mm and 10 mm, respectively. Test results are determined by calculating the arithmetic average of the experiments.

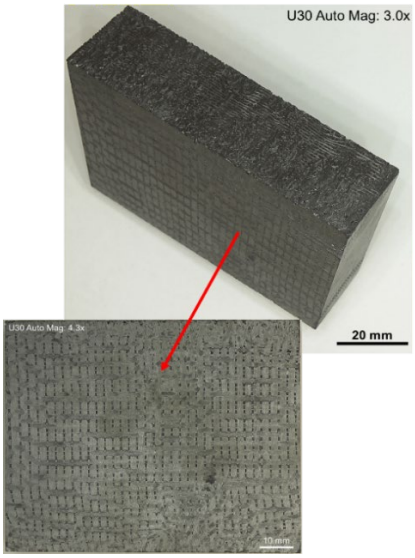

Figure 3. The visual presentation of the C/C composite block produced by CVD/CVI method

Hydrogels, biocomposites, and biodegradable implant materials used in biomedical applications are commonly subjected to in vitro biodegradation testing to evaluate their biocompatibility or biodegradability. Accordingly, biodegradation tests were conducted on C/C composite samples as well. Each sample was divided into pieces and weighed using an electronic balance with an accuracy of 0.0001 g. Subsequently, the samples were immersed in a 0.9 wt% isotonic sodium chloride solution (NaCl, I.V. Infusion) at 37 °C for 1, 3, 5, 7, 14, and 21 days [23-25].

At the end of each period, the samples were removed from the prepared solution, washed with distilled water, cleaned ultrasonically, dried for 30 minutes, and then their weights were measured again. A fresh solution was used during each incubation period, and the experiment was conducted on three samples. Weight loss was calculated according to the weight recorded before and after the incubation period [26], and the results were determined by the arithmetic mean. The biodegradation ratio was calculated using Equation 1:

$$\text{Biodegradation ratio (\%)} = \frac{w_0 - w_f}{w_0} x100 \qquad (1)$$

where; w_0 and w_f are the weights of C/C composite samples before and after in vitro biodegradation test, respectively.

3. Results and Discussion

X-ray diffractometry (XRD) analysis was conducted to identify the phases in the C/C composite densified using the CVI process, and the obtained XRD pattern is presented in Figure 4. According to the analysis result obtained from the sample surface, it was seen that the present phase was only carbon within the detection limits of XRD.

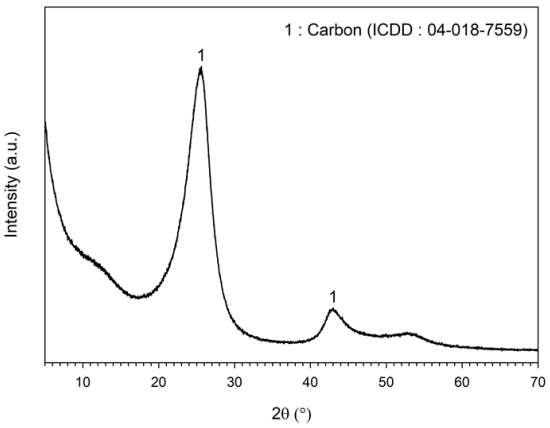

Figure 4. XRD pattern of the sample after the CVD/CVI process

Figure 5 shows the regional EDS analysis results obtained from two different regions. The presence of 100% carbon in both regions indicates that pyrolytic carbon accumulated both on the surface of the carbon fibers and between the fibers using the CVI method. Additionally, the EDS analysis results are consistent with and support the XRD analysis results.

The microstructure and morphology from both the surface and the cross-sectional of C/C composites densified using the CVI process were thoroughly examined using SEM, and the obtained images were presented in Figures 6, Figure 7 and Figure 8.

The x-y-z fiber bundles, individual carbon fibers, and macro-pores created by the orthogonal weave texture were clearly visible in the images. It was observed that the pyrolytic carbon layer deposited via the CVD/CVI method formed on

both the fiber bundles and individual fibers (Figure 6).

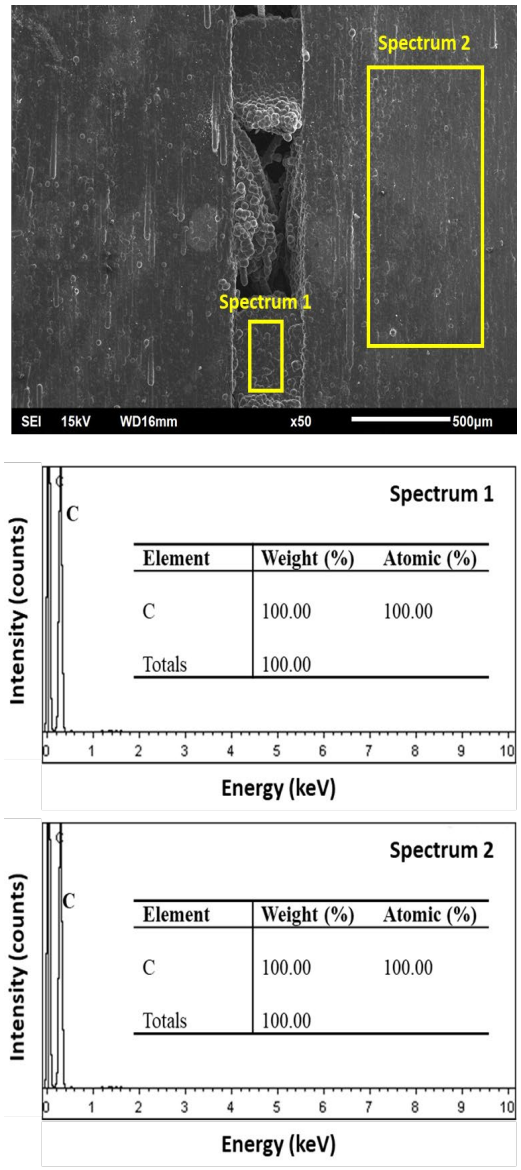

Figure 5. Regional EDS analysis results after the CVD/CVI process

The average fiber diameter was measured at 5–6 µm, while the thickness of the pyrolytic carbon layer on the fibers averaged 0.6 µm (Figure 7). Additionally, the average size of the macro voids along the y-axis of the preforms was determined to be 230 µm (Figure 8).

The orientation of carbon fiber strands, the type of weave, and the manufacturing method applied directly affect the density of the C/C composite. For example, a composite made with densely woven fibers provides higher density and mechanical properties [27]. The density of the C/C composite produced in this study, measured by the Archimedes principle, was 1.395±0.0065 g/cm³, and the amount of apparent porosity was calculated as 13.424±0.572%. These values are also presented graphically in Figure 9. E. Fitzer and M. Manocha reported that the density of C/C composites produced by various methods varies between 1.4 and 1.9 g/cm³, while H. O.

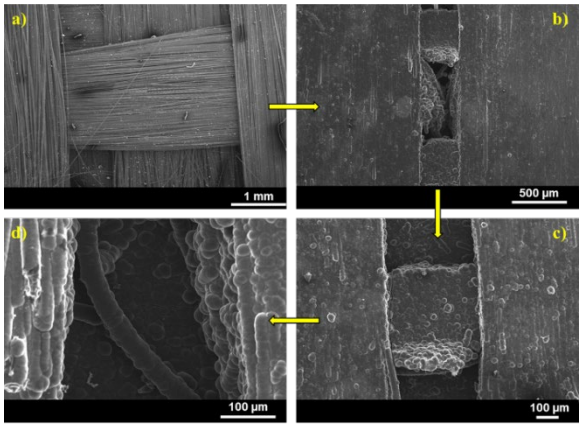

Figure 6. SEM micrographs of the C/C composites showing a) fiber bundles in the x-y axes, b) fiber bundles in the x-axis, c) fiber bundles in the y-axis, and d) pyrolytic carbon deposits on the fibers

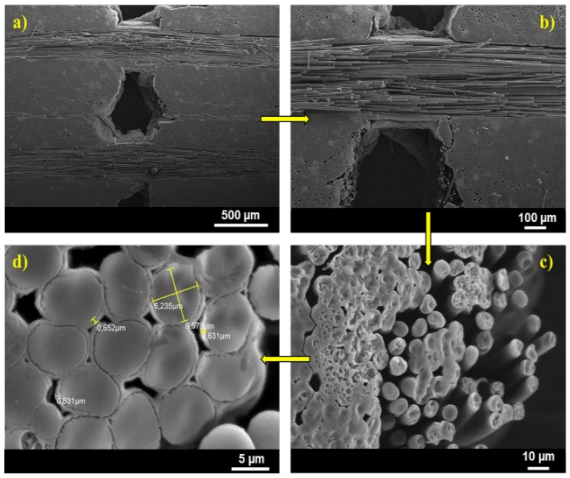

Figure 7. SEM micrographs of the C/C composites showing a) fiber bundles along the y-axis, b) enlarged view of the fiber bundles along the y-axis, c) cross-section of carbon fibers along the y-axis, d) fibers and the pyrolytic carbon layer along the y-axis

Pierson expressed the density of C/C composites as 1.7 and 1.84 g/cm³ [28, 29]. As seen, the determined density value is close to and compatible with the density value ranges reported in the literature for C/C composites.

Tensile and three-point bending tests were conducted on the C/C composites produced by the CVD/CVI method. The data for the mechanical properties obtained from these tests

are presented in Table 2. Example results from both tests are shown in Figure 10.

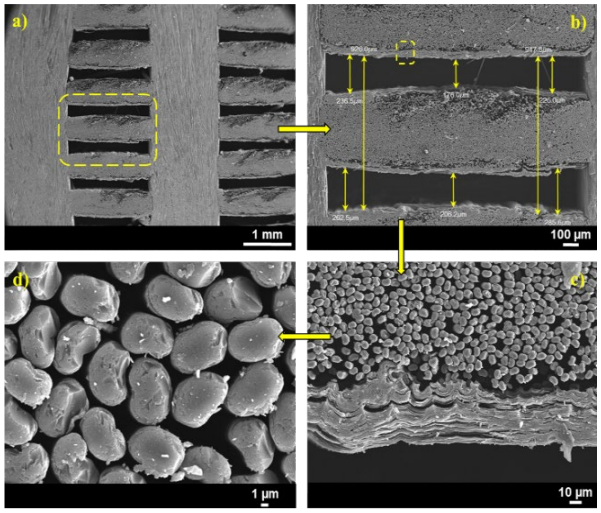

Figure 8. SEM micrographs of the C/C composites showing a) the cross-section of fiber bundles in the z-y axes, b) the cross-section of a fiber bundle in the y-axis, c) carbon fibers in the y-axis, and d) a magnified view of carbon fibers in the y-axis

E. Fitzer and M. Manocha [29] reported that the tensile strengths of C/C composites produced by various methods vary between 349 MPa and 1350 MPa, while their bending strengths range from 88 MPa to 450 MPa. H. O. Pierson [28] stated that the tensile strength of C/C composites is 270 MPa, while the bending strength is 303 MPa. As seen in Table 2, the tensile and bending strengths we found are within the ranges of values stated in the literature.

According to the results obtained from the in vitro biodegradation tests performed by immersing the samples in 0.9 wt% isotonic sodium chloride solution (NaCl, I.V. Infusion) at 37 °C for 1, 3, 5, 7, 14, and 21 days, C/C composites maintained their stability for 14 days and underwent minimal degradation in subsequent periods. The graph depicting the biodegradation ratio plotted depending on incubation time was presented in Figure 11. It is seen that the curve drawn by applying regression changes exponentially.

At the end of 21 days, the biodegradation rate of C/C composites was determined as 0.0095%. The results obtained suggest that the samples produced by the CVD/CVI process can be used in the implanted area without deformation, maintaining its integrity and stability for a long time.

In recent studies, C/C composites have been described for their exceptional mechanical, chemical, and biological properties, as well as their stability. For example, it was stated in studies by various researchers [30-32] that the pyrolytic carbon matrix and carbon fibers enhance the composite's resistance to chemical and thermal degradation, contributing to its long-term stability in different environments.

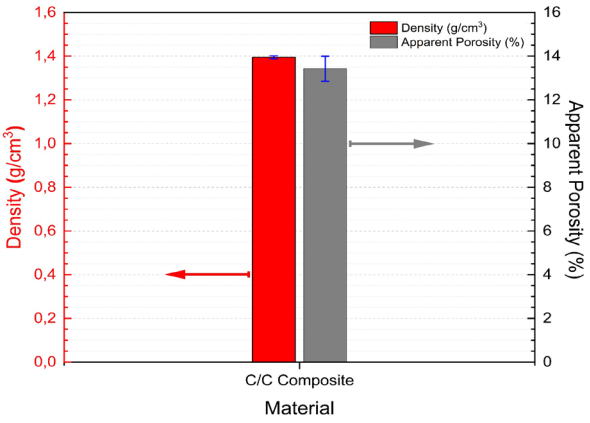

Figure 9. The density and apparent porosity graph of the C/C composites produced by CVI method

Table 2. The mechanical property data obtained from the tensile and three-point bending tests

Tests	Mechanical Properties	Value
Tensile Test	Tensile Strength (MPa)	252.5±6.20
	Elastic Modulus (GPa)	56.879±4.05
	Strain (%)	0.82±0.01
Bending Test	Bending Strength (MPa)	236.6±25.7
	Elastic Modulus (GPa)	20.318±3.39
	Strain (mm)	0.8±0.2

Wan et al. [33] studied carbon fiber-reinforced polylactide (C/PLA) composites to determine the influence of interfacial adhesion strength (IAS) on their in vitro degradation behavior in phosphate-buffered saline (PBS; pH 7.4, 37 ± 0.5 °C). They found that the PLA matrix in treated composites with nitric acid-oxidized carbon fibers absorbed less water and experienced lower mass and molecular weight loss compared to untreated composites. All samples (pure PLA and C/PLA) showed a reduced mass loss rate after 15 days of degradation. From day 16 to 25, the degradation rates were 0.51% for pure PLA and 0.27% for C/PLA composites, respectively.

Díaz et al. [34] studied the in vitro degradation of PCL and PCL/nHA composite scaffolds. They found that the polymer structures, molecular

weight, and other characteristics influenced the degradation rates. PCL, derived from fossil carbon, is hydrophobic, highly crystalline, degraded slowly, and shows only a 0.2% weight loss after 16 weeks. However, PCL/nHA composites with high oxide phase content exhibited more significant weight loss.

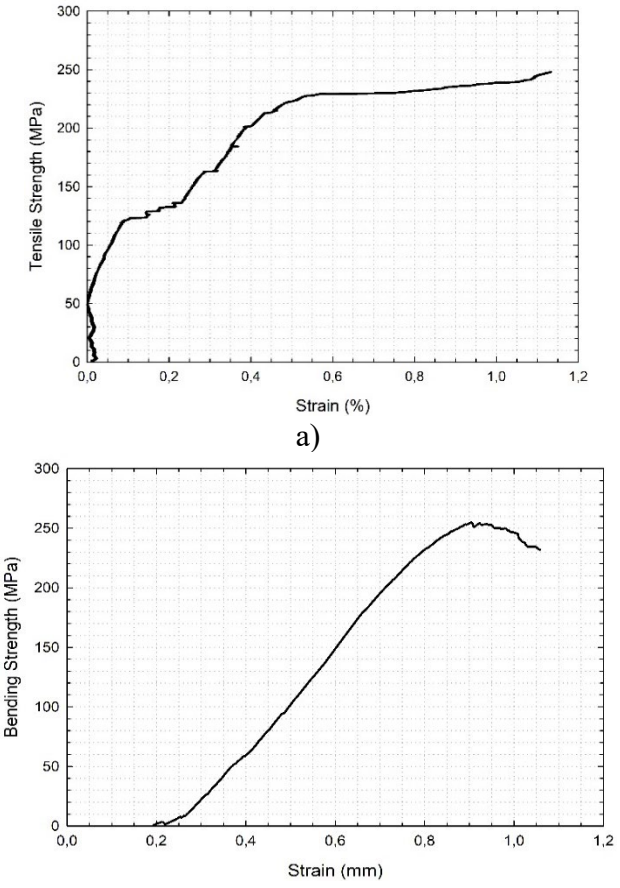

Figure 10. An example of the results from each of the two tests: a) tensile strength-strain curve, b) bending-strain curve

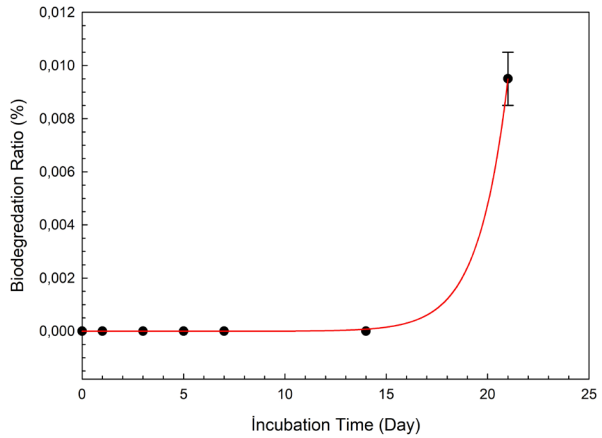

Figure 11. The biodegradation ratio graph plotted depending on incubation time

Similarly, Krishnakumar et al. [35] reported the biodegradation behavior of as-fabricated and annealed polylactic acid composites reinforced with varying carbon fiber (CF) volumes. They found that annealed carbon fiber composites exhibited improved mechanical properties and better degradation resistance than as-fabricated ones. Carbon fiber reinforcement accelerated degradation, resulting in significant changes in weight, pH, and mechanical properties of the composites immersed in simulated body fluid (SBF).

As seen, the degradation behavior in our study aligns with findings reported in the literature, where C/C composites exhibit minimal biodegradation over extended periods, even under physiological conditions.

4. Conclusion

In this study, the structural and mechanical properties of a carbon/carbon composite produced by consolidating a 3D preform woven from carbon fibers using the CVI method were examined. Additionally, the biodegradation of the produced C/C composite was investigated in 0.9 wt% isotonic sodium chloride solution (NaCl, I.V. Infusion) at 37 °C for different incubation periods. The obtained results are summarized below.

A 77% weight increase, which is an indicator of carbon accumulation in the preform, occurred in the 3D preform densified with the CVI method.

The final density of the produced samples reached the targeted level. The density and apparent porosity amount of the samples were calculated as 1.395±0.0065 g/cm³ and 13.424±0.572%, respectively.

The XRD analysis revealed that the present phase consisted solely of carbon, and EDS analyses supported this result.

SEM examinations revealed that the structure consists of a carbon matrix, fiber bundles, and pores, consistent with the structure expected from carbon/carbon composites.

Tensile and three-point bending tests applied to the samples showed that the tensile strength and bending strength of the samples were 252.5±6.20 and 236.6±25.7 MPa, respectively. Thus, applied mechanical tests revealed that the produced samples exhibited mechanical properties similar to those expected from biomaterials to be used as bone or hard tissue implants.

The in vitro biodegradation tests have shown that C/C composites maintained their stability for 14 days and underwent minimal degradation in subsequent periods. At the end of 21 days, the biodegradation rate of C/C composites was determined as 0.0095%.

Even though the biodegradation results are positive, it is necessary to perform more advanced in vitro and in vivo tests to confidently assert that these produced materials can be used as biomaterials.

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Authors' Contribution
Authors contributed equally to the study.

The Declaration of Conflict of Interest/ Common Interest
No conflict of interest or common interest has been declared by authors.

The Declaration of Ethics Committee Approval
This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics
Authors of the paper declare that they comply with the scientific, ethical, and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification of the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered and that this study has not been evaluated in any academic.

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