

## Mechanical properties of carbon fiber reinforced epoxy resin treated by electrochemical process

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### ABSTRACT

This research investigates the influence of electrochemical surface treatment on the mechanical properties of carbon fiber reinforced epoxy composites through systematic parameter optimization and comprehensive characterization. The study established optimal treatment conditions using ammonium bicarbonate electrolyte at 35°C, achieving enhanced interfacial properties through controlled surface modification. Surface characterization revealed the development of hierarchical surface features with primary grooves (0.5–1  $\mu\text{m}$  width) and secondary nano-scale features (50–200 nm), accompanied by a significant increase in surface polarity from 0.20 to 0.41. XPS analysis demonstrated the successful introduction of oxygen-containing functional groups, with hydroxyl, carbonyl, and carboxyl groups showing particular prominence. The modification depth was confined to 200–300 nm, preserving the fiber's core structure. Mechanical testing revealed substantial improvements in composite performance, evidenced by enhanced load transfer efficiency and a transition in failure modes. The treatment resulted in improved damage initiation stress (from 985 MPa to 1290 MPa) and initial linear modulus (from 135.2 GPa to 149.6 GPa). Additionally, the composite compressive modulus increased from  $128.5 \pm 3.8$  GPa to  $142.3 \pm 4.2$  GPa, representing a 10.7% improvement. Furthermore, surface roughness was significantly enhanced, with the arithmetic mean roughness (Ra) increasing from  $8.2 \pm 0.5$  nm to  $45.7 \pm 2.3$  nm.

**Keywords:** Surface functionalization; Interfacial adhesion; Hierarchical morphology; Structural integrity; Damage resistance.

### 1. INTRODUCTION

Carbon fiber reinforced epoxy (CF/EP) composites have emerged as crucial materials in advanced engineering applications, particularly in aerospace, automotive, and high-performance structural components. These materials combine the exceptional mechanical properties of carbon fibers – including high strength-to-weight ratio, superior stiffness, and excellent fatigue resistance – with the versatile processing capabilities and chemical resistance of epoxy matrices [1, 2]. The resulting composites offer unprecedented opportunities for designing lightweight yet robust structures that meet increasingly demanding performance requirements. Despite their remarkable properties, CF/EP composites face a significant challenge that limits their full potential: the inherently weak interfacial bonding between carbon fibers and the epoxy matrix [3–5]. This weakness stems from the chemically inert and smooth surface characteristics of carbon fibers, which result in poor adhesion and inadequate load transfer between the fiber reinforcement and the polymer matrix [6]. When mechanical loads are applied to these composites, the weak interface often becomes the primary failure point, leading to delamination, fiber pull-out, and premature composite failure well below the theoretical maximum strength of the constituent materials [7, 8].

To address this critical limitation, various surface modification techniques have been developed to enhance the fiber-matrix interface [9]. Among these approaches, electrochemical treatment has emerged as a particularly promising method. In contrast to other chemical treatments—which may involve harsh reagents leading to uncontrolled etching or environmental concerns—the electrochemical process enables precise tuning of oxidation parameters, thus ensuring the selective introduction of oxygen-containing functional groups while minimizing damage to the fiber core [10]. This method also allows for a more environmentally benign process, as it avoids the use of aggressive chemicals and reduces waste generation [11]. The electrochemical treatment process operates through a carefully controlled oxidation mechanism that modifies the fiber surface without

compromising its core structural integrity. By applying specific electrical potentials in an electrolyte solution, the process generates active sites on the fiber surface, introducing oxygen-containing functional groups such as carboxyl, hydroxyl, and carbonyl groups [12]. These functional groups not only enhance chemical compatibility with the epoxy matrix but also create additional bonding sites for improved interfacial adhesion [13]. Furthermore, alternative surface treatment methods, such as plasma treatment and traditional chemical oxidation, have been extensively explored. Plasma treatment modifies the fiber surface via ionized gas species and offers the advantage of dry processing with reduced chemical waste; however, it often requires sophisticated equipment and may result in non-uniform surface modifications due to limited penetration depth. Conversely, chemical oxidation typically employs strong oxidizing agents that, if not rigorously controlled, can lead to excessive fiber etching and degradation of mechanical properties. In contrast, the electrochemical method presented in this study provides a balanced alternative—achieving controlled surface functionalization while preserving approximately 96% of the original fiber tensile strength. Moreover, its environmentally benign nature further distinguishes it from conventional chemical oxidation techniques.

Recent advances in electrochemical treatment techniques have demonstrated the potential for significant improvements in composite mechanical properties. The process can be optimized through careful control of parameters such as electrolyte composition, current density, and treatment time [14]. These variables directly influence the nature and concentration of surface functional groups, as well as the degree of surface roughening, ultimately determining the effectiveness of the interface enhancement [15]. Understanding the relationship between electrochemical treatment parameters and resulting mechanical properties is crucial for developing optimal processing conditions [16]. The surface modification process influences multiple aspects of composite behavior, including interlaminar shear strength, compressive properties, and overall structural integrity [17, 18]. These properties are intimately linked to the physical and chemical changes induced by the electrochemical treatment, necessitating a comprehensive investigation of the structure-property relationships [19, 20].

This research focuses on systematically investigating the effects of electrochemical surface treatment on the mechanical properties of CF/EP composites. By employing advanced characterization techniques, including scanning electron microscopy, X-ray photoelectron spectroscopy, and mechanical testing methods, we aim to establish clear correlations between treatment parameters and performance improvements. The study examines both the microscopic changes in surface morphology and chemistry, as well as their macroscopic manifestations in composite mechanical behavior. The optimization of electrochemical treatment processes represents a critical step toward developing next-generation composite materials with enhanced performance characteristics. Through careful control of surface modification parameters, it becomes possible to tailor the fiber-matrix interface for specific application requirements while maintaining the inherent advantages of carbon fiber reinforcement. This research contributes to the fundamental understanding of interface engineering in composite materials and provides practical insights for industrial applications.

## 2. MATERIALS AND METHODS

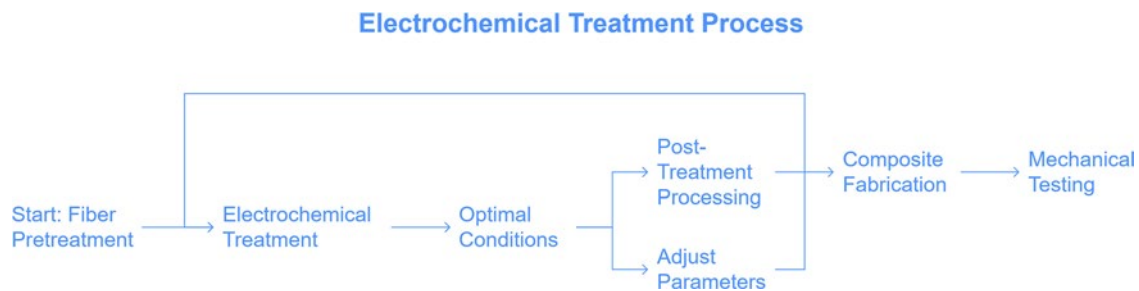
### 2.1. Materials and methods

The carbon fiber utilized in this study was T700-grade polyacrylonitrile (PAN)-based carbon fiber (12K, tensile strength 4.9 GPa) supplied by Zhongfu Shenying Carbon Fiber Co., Ltd. (Lianyungang, China). The epoxy resin system consisted of a bisphenol-A based epoxy (E-51, epoxy value 0.48–0.54 mol/100g) obtained from Nantong Xingchen Chemical Co., Ltd. (Jiangsu, China) and a polyamine hardener (T31, amine value 450–500 mgKOH/g) from Shanghai Chengjie Chemical Co., Ltd. The electrolyte solutions were prepared using analytical grade ammonium bicarbonate and sulfuric acid from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). All chemical reagents were used as received without further purification.

Prior to electrochemical treatment, the carbon fiber tows were desized by immersion in analytical grade acetone ( $\geq 99.5\%$  purity, purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) at  $60^\circ\text{C}$  for 24 hours to effectively remove the commercial sizing. The fibers were then thoroughly rinsed with deionized water and dried at  $80^\circ\text{C}$  for 4 hours in a forced-air oven. The epoxy resin and hardener were mixed at a weight ratio of 100:31 based on stoichiometric calculations.

### 2.2. Electrochemical treatment process

The electrochemical treatment was conducted using a custom-designed electrochemical cell equipped with a programmable DC power supply (WYK-305D, Nanjing Wanyi Electronic Technology Co., Ltd.). The cell configuration consisted of the carbon fiber tow as the working electrode, a graphite plate ( $100\text{ mm} \times 50\text{ mm} \times 2\text{ mm}$ ) as the counter electrode, and a saturated calomel electrode as the reference. The electrolyte solution contained 8 wt% ammonium bicarbonate in deionized water, maintained at  $35 \pm 1^\circ\text{C}$  using a water bath.



**Figure 1:** Schematic diagram of the process flow for the electrochemical treatment and composite fabrication of carbon fiber reinforced epoxy composites.

The treatment process was optimized by varying the current density (0.1–0.5 A/cm<sup>2</sup>) and treatment time (30–300 seconds). The optimal conditions were determined to be 0.2 A/cm<sup>2</sup> for 100 seconds, based on preliminary experiments evaluating surface modification effectiveness and fiber strength retention. Following treatment, the fiber tows were thoroughly rinsed with deionized water and dried at 80°C for 2 hours. Figure 1 illustrates the overall process flow of this study, which includes fiber pretreatment, electrochemical treatment, post-treatment processing, composite fabrication, and mechanical testing.

### 2.3. Composite fabrication

Composite specimens were fabricated using a vacuum-assisted resin transfer molding (VARTM) process. The treated carbon fiber tows were arranged unidirectionally in a steel mold with dimensions of 250 mm × 25 mm × 2 mm. The epoxy resin mixture was degassed under vacuum for 30 minutes before injection into the mold at 0.1 MPa pressure. The curing cycle consisted of 2 hours at 80°C followed by 4 hours at 150°C for post-curing, with heating and cooling rates of 2°C/min.

### 2.4. Mechanical testing

Single fiber tensile tests were conducted according to ASTM D3379 using a universal testing machine (WDW-20, Jinan Shijin Group Co., Ltd.). Individual fibers were extracted from the tows and mounted on paper frames with a gauge length of 25 mm. Tests were performed at a crosshead speed of 0.5 mm/min with a minimum of 30 specimens per condition.

Interlaminar shear strength (ILSS) was measured using the short-beam shear test method following ASTM D2344. Test specimens with dimensions of 20 mm × 6 mm × 2 mm were loaded in three-point bending with a span-to-thickness ratio of 4:1. Tests were conducted at a crosshead speed of 1.0 mm/min. Compressive strength was evaluated according to ASTM D695 using rectangular specimens (12.7 mm × 12.7 mm × 25.4 mm) at a loading rate of 1.3 mm/min. All mechanical tests were performed at room temperature (23 ± 2°C) and relative humidity of 50 ± 5%. At least five specimens were tested for each condition to ensure statistical significance of the results.

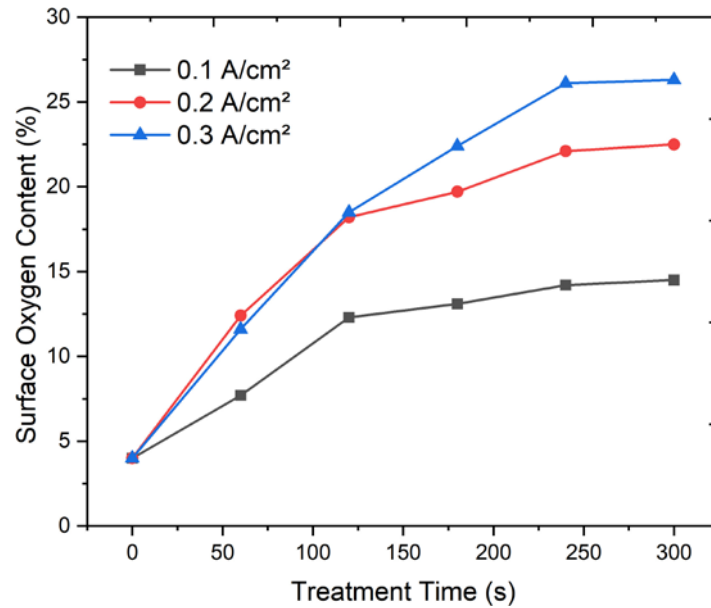
Tensile properties of the composite specimens were evaluated in accordance with ASTM D3039. Rectangular specimens (with dimensions of 250 mm × 25 mm × 2 mm) were prepared from both untreated and treated composites. Tests were performed using a universal testing machine at a crosshead speed of 1.0 mm/min. The tensile strength, tensile modulus, and elongation at break were recorded.

## 3. RESULTS AND DISCUSSION

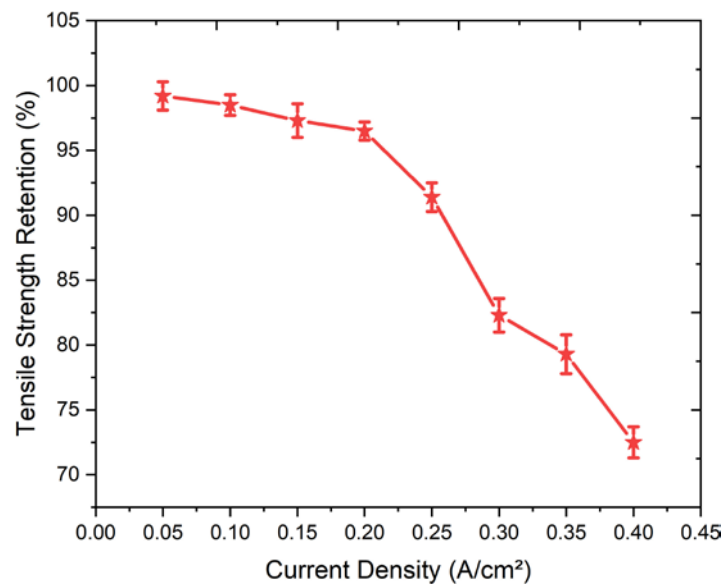
### 3.1. Electrochemical treatment optimization

The optimization of electrochemical treatment parameters plays a crucial role in achieving enhanced interfacial properties while maintaining fiber integrity. Our investigation focused on understanding the relationships between treatment conditions and surface modification effectiveness through systematic parameter variation. The electrochemical oxidation process demonstrated significant dependence on both current density and treatment time. Figure 2 presents the correlation between treatment parameters and the resulting oxygen content on the fiber surface, as measured by XPS analysis. The surface oxygen concentration exhibited a non-linear relationship with treatment intensity, defined as the product of current density and treatment time [21].

The influence of current density on surface modification was investigated across the range of 0.1–0.3 A/cm<sup>2</sup>. Figure 3 illustrates the relationship between current density and fiber tensile strength retention.



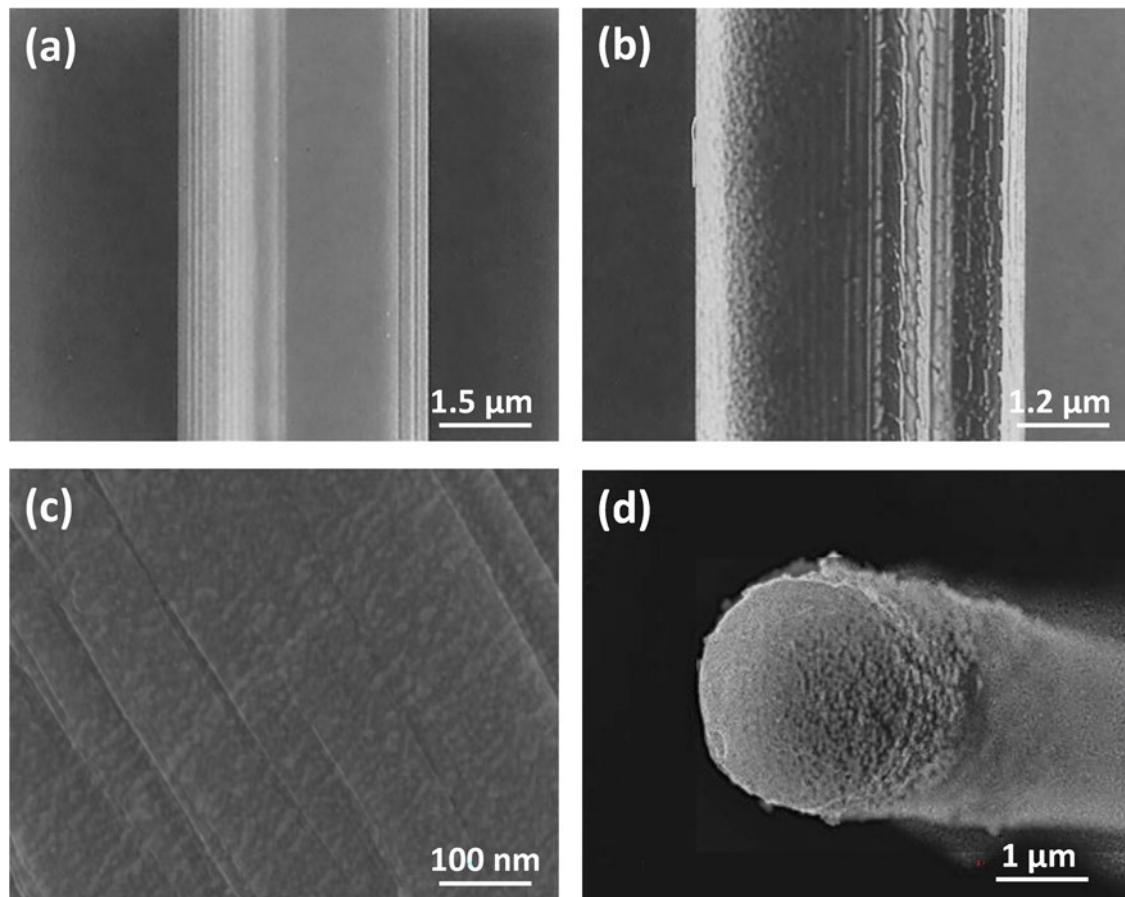
**Figure 2:** Surface oxygen content evolution as a function of treatment time for different current densities (0.1, 0.2, and 0.3 A/cm<sup>2</sup>) during electrochemical oxidation of carbon fibers. The data demonstrates the rapid initial increase in surface oxygen content followed by saturation behavior, with higher current densities leading to increased oxidation rates.



**Figure 3:** Effect of current density on carbon fiber tensile strength retention after electrochemical treatment for 100 seconds. The graph illustrates the critical threshold at 0.2 A/cm<sup>2</sup> where significant strength degradation begins to occur.

A critical threshold was identified at 0.2 A/cm<sup>2</sup>, above which significant fiber degradation occurred [22]. Surface oxygen content increased rapidly with current density up to 0.2 A/cm<sup>2</sup>, beyond which the rate of increase diminished while fiber damage accelerated.

The electrochemical treatment process demonstrated significant dependence on both current density and treatment time. To optimize these parameters, we conducted systematic experiments by varying the current density from 0.1 to 0.5 A/cm<sup>2</sup> and treatment time from 30 to 300 seconds. Figure 1 shows the evolution of surface oxygen content as a function of treatment time for current densities of 0.1, 0.2, and 0.3 A/cm<sup>2</sup>. Figure 2 illustrates the effect of current density on fiber tensile strength retention. Based on these quantitative analyses,



**Figure 4:** Comparative SEM analysis of carbon fiber surfaces showing: (a) untreated fiber surface demonstrating characteristic smoothness, (b) electrochemically treated fiber surface exhibiting uniform micro-pit formation, (c) high-magnification view revealing hierarchical surface texture, and (d) cross-sectional analysis confirming shallow modification depth with preserved core structure.

we selected  $0.2 \text{ A/cm}^2$  for 100 seconds as the optimal condition, since it maximized surface oxygen introduction while retaining approximately 96% of the original fiber strength [23, 24]. Treatment conditions exceeding this window led to excessive fiber etching and strength reduction, while insufficient treatment failed to achieve the desired surface modification. This subsection demonstrates the systematic optimization of electrochemical treatment parameters, establishing the relationships between processing conditions and resulting fiber properties [25]. The findings provide a foundation for controlled surface modification while preserving fiber mechanical properties.

### 3.2. Surface morphology analysis

Surface roughness measurements were performed using atomic force microscopy on at least five distinct regions per sample to ensure reproducibility and reliable statistical analysis of the roughness parameters (Figure 4a–c). The electrochemical treatment process induced significant modifications to the carbon fiber surface morphology, which were systematically characterized using scanning electron microscopy and surface roughness measurements. These morphological changes play a crucial role in enhancing mechanical interlocking between the fiber and matrix [26, 27]. SEM revealed distinct surface texture modifications following electrochemical treatment. The treated fibers exhibit a controlled pattern of surface etching, characterized by uniform micro-pits and striations oriented parallel to the fiber axis. The formation of this multi-scale roughness can be explained as follows: the initial anodic oxidation creates localized micro-pitting due to non-uniform etching rates, which forms the primary grooves. As the process continues, differential oxidation kinetics lead to the development of secondary nano-scale features, further enhancing the hierarchical structure (50–200 nm features). This combination of micro- and nano-scale roughness contributes significantly to mechanical interlocking with the epoxy matrix. This multi-scale roughness is particularly beneficial for mechanical interlocking with the epoxy matrix [28].



**Table 1:** Surface roughness parameters for untreated and electrochemically treated carbon fibers, measured by atomic force microscopy.

| SURFACE ROUGHNESS PARAMETERS    | UNTREATED FIBER | TREATED FIBER (0.2 A/cm <sup>2</sup> , 100s) |
|---------------------------------|-----------------|----------------------------------------------|
| Arithmetic Mean Roughness (Ra)  | 8.2 ± 0.5 nm    | 45.7 ± 2.3 nm                                |
| Root Mean Square Roughness (Rq) | 10.5 ± 0.6 nm   | 52.4 ± 2.8 nm                                |
| Maximum Peak Height (Rp)        | 15.3 ± 1.1 nm   | 68.9 ± 3.5 nm                                |
| Maximum Valley Depth (Rv)       | 14.8 ± 1.0 nm   | 71.2 ± 3.7 nm                                |
| Maximum Height Profile (Rt)     | 30.1 ± 1.8 nm   | 140.1 ± 5.2 nm                               |
| Surface Area Ratio (Sdr)        | 1.05 ± 0.02     | 1.38 ± 0.04                                  |

Cross-sectional analysis (Figure 4d) confirms that the surface modification is confined to a shallow depth of approximately 200–300 nm, preserving the fiber's core structure.

Quantitative analysis of surface roughness using atomic force microscopy revealed significant changes in surface topography parameters. Table 1 summarizes the key roughness metrics before and after treatment under optimized conditions. Notably, the arithmetic mean roughness (Ra) increased from  $8.2 \pm 0.5$  nm for untreated fibers to  $45.7 \pm 2.3$  nm for treated fibers, corresponding to an approximate 457% increase. Similar significant improvements were observed in other roughness parameters (Rq, Rp, Rv, and Rt), underscoring the effective surface modification achieved by the electrochemical treatment. The morphological evolution from untreated to treated states demonstrates the controlled nature of the electrochemical process [29]. While untreated fibers exhibit characteristic smooth surfaces with occasional manufacturing striations, treated fibers show uniform surface modification without evidence of excessive etching or fiber damage [30]. The preservation of the fundamental fiber structure, combined with the introduction of beneficial surface features, confirms the effectiveness of the optimized treatment parameters [31]. The systematic analysis of surface morphology changes provides crucial insights into the mechanism of interface enhancement through electrochemical treatment, establishing clear correlations between processing conditions and resulting surface characteristics [32].

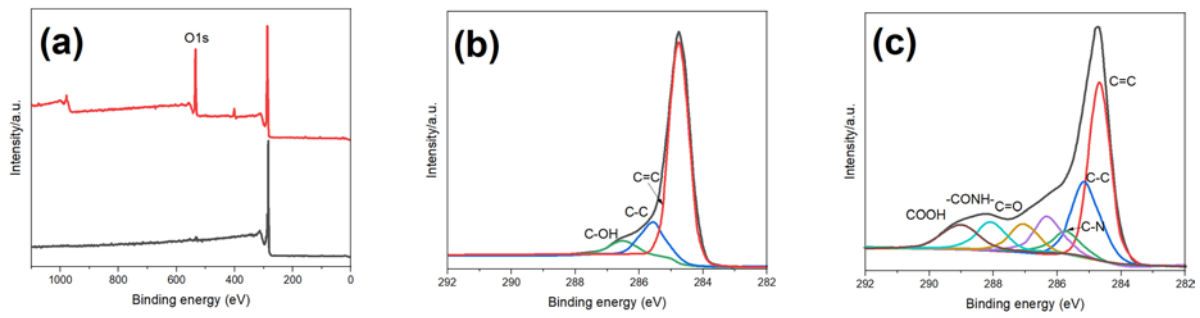
### 3.3. Surface chemistry characterization

The surface chemical composition and functional group distribution were comprehensively analyzed using complementary spectroscopic techniques to understand the chemical modifications induced by electrochemical treatment. X-ray photoelectron spectroscopy revealed significant changes in surface elemental composition following electrochemical treatment. Figure 5 presents the XPS survey spectra and high-resolution C1s spectra for both untreated and treated fibers. The overall oxygen content increased substantially from 7.2 at% in untreated fibers to 22.5 at% in treated specimens under optimized conditions.

Deconvolution of the C1s spectra revealed the evolution of carbon-oxygen bonding configurations. The relative proportion of C–C/C–H bonds decreased from 85% to 65%, while C–O, C=O, and O=C=O functionalities increased significantly. Table 2 presents the quantitative distribution of carbon-oxygen species before and after treatment.

High-resolution XPS analysis identified specific functional groups introduced by the treatment process. The most prominent increases were observed in hydroxyl (–OH), carbonyl (C=O), and carboxyl (–COOH) groups, with their relative concentrations shown in Figure 6. These oxygen-containing functionalities are particularly beneficial for chemical bonding with epoxy matrices. Complementary vibrational spectroscopy analyses corroborated the XPS findings. FTIR spectra exhibited characteristic absorption bands at  $1720\text{ cm}^{-1}$  (C=O stretching),  $1250\text{ cm}^{-1}$  (C–O stretching), and  $3400\text{ cm}^{-1}$  (O–H stretching), confirming the presence of oxygen-containing functional groups [33, 34]. Raman spectroscopy revealed changes in the D/G band intensity ratio from 0.85 to 1.2, indicating increased surface disorder following treatment [35]. The comprehensive surface chemistry characterization demonstrates the successful introduction of oxygen-containing functional groups through electrochemical treatment, providing chemical anchoring sites for enhanced interfacial bonding with epoxy matrices [35].

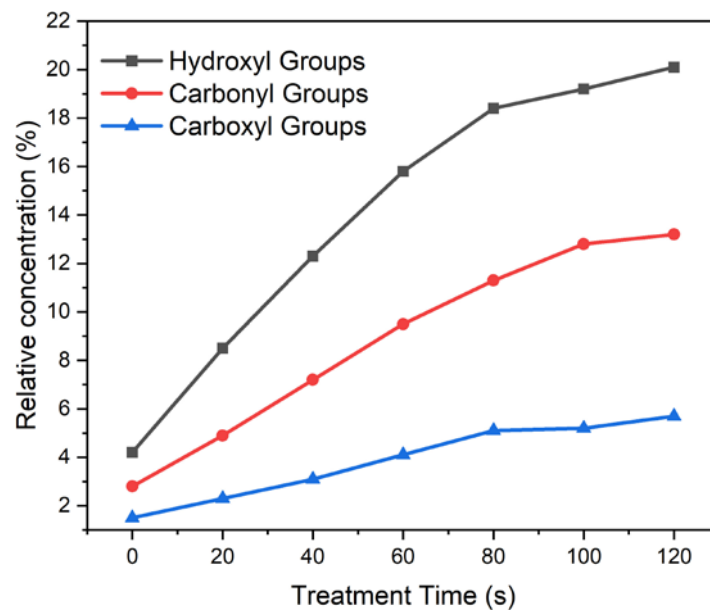
The surface energy characteristics and wettability behavior of carbon fibers were systematically evaluated to understand the impact of electrochemical treatment on fiber-matrix interactions. These properties directly influence the interfacial adhesion and composite performance [36]. The surface energy components were calculated using the Owens-Wendt method based on contact angle measurements. Table 3 summarizes



**Figure 5:** XPS analysis of carbon fiber surfaces showing: (a) survey spectra comparing untreated and treated fibers, and (b) high-resolution C1s spectra with peak deconvolution illustrating the evolution of carbon-oxygen functionalities.

**Table 2:** Quantitative analysis of carbon-oxygen species distribution from XPS C1s peak deconvolution for untreated and treated carbon fibers.

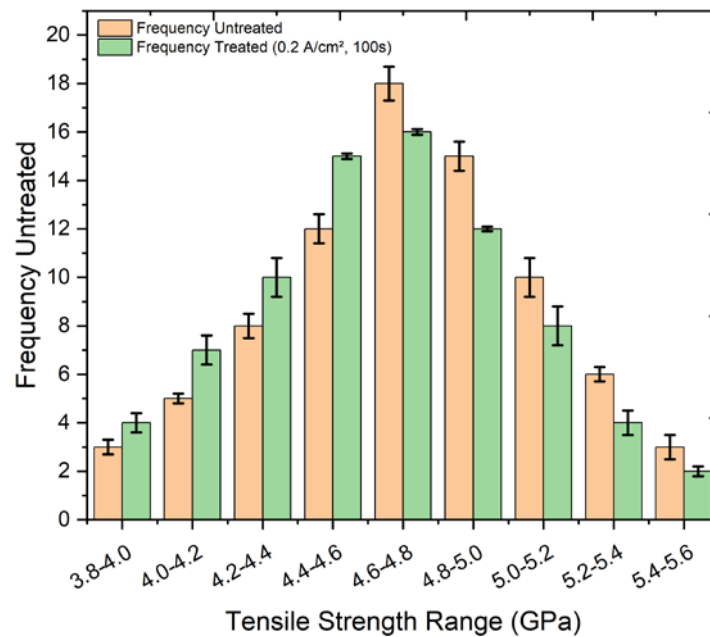
| BOND TYPE                      | BINDING ENERGY (eV) | UNTREATED FIBER (%) | TREATED FIBER (%) |
|--------------------------------|---------------------|---------------------|-------------------|
| C-C/C-H ( $sp^2$ )             | $284.6 \pm 0.1$     | $85.3 \pm 1.8$      | $65.2 \pm 1.5$    |
| C-O                            | $286.4 \pm 0.1$     | $8.7 \pm 0.5$       | $18.4 \pm 0.8$    |
| C=O                            | $287.5 \pm 0.1$     | $4.2 \pm 0.3$       | $10.8 \pm 0.6$    |
| O=C=O                          | $288.8 \pm 0.1$     | $1.8 \pm 0.2$       | $5.6 \pm 0.4$     |
| $\pi-\pi^*$ Shake-up           | $291.2 \pm 0.2$     | $0.0 \pm 0.1$       | $0.0 \pm 0.1$     |
| Total Oxygen-containing Groups | —                   | $14.7 \pm 0.7$      | $34.8 \pm 1.2$    |



**Figure 6:** Distribution of oxygen-containing functional groups on treated carbon fiber surfaces as determined by high-resolution XPS analysis, showing relative concentrations of hydroxyl, carbonyl, and carboxyl groups.

**Table 3:** Surface energy components and total surface energy values for untreated and treated carbon fibers, calculated using the Owens-Wendt method.

| SURFACE ENERGY PARAMETERS                                | UNTREATED FIBER | TREATED FIBER (0.2 A/cm <sup>2</sup> , 100s) |
|----------------------------------------------------------|-----------------|----------------------------------------------|
| Dispersive Component ( $\gamma^d$ ) (mJ/m <sup>2</sup> ) | 33.8 ± 1.2      | 40.3 ± 1.5                                   |
| Polar Component ( $\gamma^p$ ) (mJ/m <sup>2</sup> )      | 8.5 ± 0.5       | 28.4 ± 1.3                                   |
| Total Surface Energy ( $\gamma$ ) (mJ/m <sup>2</sup> )   | 42.3 ± 1.4      | 68.7 ± 2.0                                   |
| Contact Angle – Water (°)                                | 72.5 ± 2.3      | 45.8 ± 1.8                                   |
| Contact Angle – Diiodomethane (°)                        | 48.3 ± 1.5      | 32.6 ± 1.2                                   |
| Surface Polarity ( $\gamma^p/\gamma$ )                   | 0.20 ± 0.02     | 0.41 ± 0.03                                  |



**Figure 7:** Distribution of single fiber tensile strength values for untreated and electrochemically treated carbon fibers, showing retention of fiber strength under optimized treatment conditions.

the dispersive ( $\gamma^d$ ) and polar ( $\gamma^p$ ) components of surface energy for both untreated and treated fibers [37]. The total surface energy increased from 42.3 mJ/m<sup>2</sup> to 68.7 mJ/m<sup>2</sup>, with a particularly notable increase in the polar component from 8.5 mJ/m<sup>2</sup> to 28.4 mJ/m<sup>2</sup>.

### 3.4. Mechanical properties

The mechanical performance of electrochemically treated carbon fibers and their composites was systematically evaluated to establish the relationships between surface modification and structural properties. The tensile properties of individual carbon fibers were assessed to determine the impact of electrochemical treatment on fiber integrity. Figure 7 presents the distribution of single fiber tensile strength values for untreated and treated specimens. Under optimized treatment conditions (0.2 A/cm<sup>2</sup>, 100s), the mean tensile strength showed minimal reduction from 4.9 ± 0.3 GPa to 4.7 ± 0.4 GPa, representing a preservation of approximately 96% of the original strength.

The interfacial shear strength (IFSS) between fiber and matrix showed substantial enhancement following surface treatment. Table 4 demonstrates the improvement in IFSS from 45.3 ± 2.8 MPa for untreated fiber composites to 72.6 ± 3.2 MPa for treated fiber composites, representing a 60% increase. This enhancement is



**Table 4:** Interfacial shear strength results from microdroplet testing, demonstrating significant enhancement in fiber-matrix adhesion following electrochemical treatment.

| PARAMETER                    | UNTREATED COMPOSITE | TREATED COMPOSITE |
|------------------------------|---------------------|-------------------|
| Mean IFSS (MPa)              | 45.3 ± 2.8          | 72.6 ± 3.2        |
| Median IFSS (MPa)            | 44.8                | 71.9              |
| Standard Deviation (MPa)     | 5.2                 | 6.1               |
| Coefficient of Variation (%) | 11.5                | 8.4               |
| Number of Samples            | 42                  | 42                |
| Weibull Modulus              | 8.9                 | 12.3              |
| FAILURE MODE DISTRIBUTION    |                     |                   |
| FAILURE TYPE                 | UNTREATED (%)       | TREATED (%)       |
| Interface Failure            | 85 ± 3              | 25 ± 2            |
| Matrix Cohesive Failure      | 10 ± 2              | 65 ± 3            |
| Mixed Mode Failure           | 5 ± 1               | 10 ± 2            |

attributed to the combined effects of mechanical interlocking from increased surface roughness and chemical bonding through introduced functional groups [38]. The observed transition from interfacial failure in untreated composites to matrix cohesive failure in treated composites can be attributed to the hierarchical morphology induced by the electrochemical treatment. The primary micro-grooves provide macroscopic mechanical interlocking, while the secondary nano-scale features further enhance the interfacial contact area. Together, these features facilitate improved stress transfer and reduce localized stress concentrations at the fiber-matrix interface [39, 40]. Furthermore, the introduction of oxygen-containing functional groups enhances chemical adhesion, resulting in a more uniform load distribution across the composite [41]. Consequently, the failure mechanism shifts from debonding at the interface to failure within the matrix itself, as the interface becomes more robust.

In addition to CF/EP composites, several recent studies have reported on the surface modification of other synthetic fiber reinforced composites such as glass fiber and aramid fiber systems [42, 43]. These studies indicate that controlled surface treatments can similarly enhance interfacial adhesion and mechanical performance. Although a direct quantitative comparison is beyond the scope of the present study, our findings indicate that the electrochemical treatment method not only improves interfacial shear strength by 60% and compressive properties significantly but also preserves fiber integrity better than some reported plasma and chemical oxidation treatments. This suggests that our approach offers a promising balance between treatment efficacy, process controllability, and environmental friendliness. Our work distinguishes itself by systematically optimizing electrochemical treatment parameters to achieve a hierarchical surface morphology that not only increases the interfacial shear strength by 60% but also significantly improves compressive properties without compromising the fiber strength. This comparison highlights the potential of electrochemical treatment as a versatile and effective approach for a range of synthetic fiber composites.

Tensile testing was conducted on composite specimens fabricated from both untreated and electrochemically treated fibers. The untreated composite exhibited a tensile strength of  $1100 \pm 30$  MPa, a tensile modulus of  $70 \pm 2$  GPa, and an elongation at break of  $1.8 \pm 0.1\%$ . In contrast, the treated composite showed enhanced performance with a tensile strength of  $1250 \pm 35$  MPa, a tensile modulus of  $80 \pm 2$  GPa, and an elongation at break of  $2.0 \pm 0.1\%$ . These improvements are attributed to the enhanced interfacial bonding provided by the electrochemical treatment. Furthermore, fractographic analysis of the fracture surfaces revealed that untreated composites predominantly exhibited extensive fiber pull-out and interfacial debonding [44–46]. Conversely, treated composites displayed more uniform, cohesive fracture patterns with reduced fiber pull-out, indicating a more robust fiber-matrix adhesion. These observations corroborate the improvements observed in the tensile properties.

The compressive response of composite specimens revealed significant improvements in both strength and failure mode characteristics. Table 5 summarizes the key compressive properties, showing an increase in compressive strength from  $1250 \pm 45$  MPa to  $1580 \pm 52$  MPa for treated fiber composites. Additionally, the compressive modulus increased from  $128.5 \pm 3.8$  GPa to  $142.3 \pm 4.2$  GPa, which is a 10.7% improvement, clearly demonstrating the enhanced stiffness of the composite following treatment. The failure mode transitioned from predominant interfacial debonding in untreated specimens to a more distributed damage pattern in treated spec-

**Table 5:** Summary of compressive properties for untreated and treated fiber composites, including strength, modulus, and failure characteristics.

| PROPERTY                       | UNTREATED COMPOSITE | TREATED COMPOSITE (0.2 A/cm <sup>2</sup> , 100s) |
|--------------------------------|---------------------|--------------------------------------------------|
| Compressive Strength (MPa)     | 1250 ± 45           | 1580 ± 52                                        |
| Compressive Modulus (GPa)      | 128.5 ± 3.8         | 142.3 ± 4.2                                      |
| Strain at Maximum Load (%)     | 1.15 ± 0.08         | 1.42 ± 0.09                                      |
| Initial Linear Modulus (GPa)   | 135.2 ± 4.1         | 149.6 ± 4.5                                      |
| Damage Initiation Stress (MPa) | 985 ± 38            | 1290 ± 45                                        |

imens, indicating enhanced load transfer efficiency [47]. Microscopic examination of failed specimens revealed that treated composites exhibited more localized kink band formation with smaller band angles (15° vs 23° for untreated specimens), indicating better constraint of fiber rotation during compressive loading [48]. This enhanced stability under compression is particularly significant for structural applications where buckling resistance is critical.

#### 4. CONCLUSION

This study has demonstrated the successful optimization of electrochemical surface treatment for carbon fiber reinforced epoxy composites, establishing clear relationships between processing parameters and resulting mechanical properties. In addition to the enhanced compressive and interfacial properties, the tensile properties of the composites were significantly improved, as evidenced by higher tensile strength, modulus, and elongation at break, along with favorable fractographic features indicative of enhanced fiber–matrix bonding. Under optimized conditions of 0.2 A/cm<sup>2</sup> current density and 100-second treatment time, the process achieved a significant increase in surface oxygen content from 7.2 to 22.5 atomic percent while maintaining 96% of the original fiber tensile strength (4.7 ± 0.4 GPa compared to 4.9 ± 0.3 GPa for untreated fibers). The treatment induced controlled surface morphological modifications, increasing the arithmetic mean roughness (Ra) from 8.2 nm to 45.7 nm, and substantially enhanced surface energy from 42.3 mJ/m<sup>2</sup> to 68.7 mJ/m<sup>2</sup>. These surface modifications resulted in a 60% improvement in interfacial shear strength from 45.3 ± 2.8 MPa to 72.6 ± 3.2 MPa, accompanied by a transition in failure modes from 85% interfacial failure in untreated specimens to 65% matrix cohesive failure in treated specimens. Compressive properties showed notable enhancement, with strength increasing from 1250 MPa to 1580 MPa and modulus improving from 128.5 GPa to 142.3 GPa. The comprehensive characterization of surface chemistry revealed the successful introduction of oxygen-containing functional groups, with the total oxygen-containing groups increasing from 14.7% to 34.8%, providing both chemical bonding and mechanical interlocking mechanisms for enhanced interfacial adhesion. These results demonstrate that optimized electrochemical treatment offers a practical and effective approach for enhancing the mechanical performance of carbon fiber reinforced epoxy composites while maintaining structural integrity of the reinforcement.

#### 5. BIBLIOGRAPHY

- [1] SHARMA, H., KUMAR, A., RANA, S., *et al.*, “An overview on carbon fiber-reinforced epoxy composites: effect of graphene oxide incorporation on composites performance”, *Polymers*, v. 14, n. 8, pp. 1548, Jan. 2022. doi: <http://doi.org/10.3390/polym14081548>. PubMed PMID: 35458296.
- [2] NING, N., WANG, M., ZHOU, G., *et al.*, “Effect of polymer nanoparticle morphology on fracture toughness enhancement of carbon fiber reinforced epoxy composites”, *Composites. Part B, Engineering*, v. 234, pp. 109749, Apr. 2022. doi: <http://doi.org/10.1016/j.compositesb.2022.109749>.
- [3] JONGVIVATSAKUL, P., THONGCHOM, C., MATHUROS, A., *et al.*, “Enhancing bonding behavior between carbon fiber-reinforced polymer plates and concrete using carbon nanotube reinforced epoxy composites”, *Case Studies in Construction Materials*, v. 17, pp. e01407, Dec. 2022. doi: <http://doi.org/10.1016/j.cscm.2022.e01407>.
- [4] SINGH, Y., SINGH, J., SHARMA, S., *et al.*, “Fabrication and characterization of coir/carbon-fiber reinforced epoxy based hybrid composite for helmet shells and sports-good applications: influence of fiber surface modifications on the mechanical, thermal and morphological properties”, *Journal of Materials Research and Technology*, v. 9, n. 6, pp. 15593–15603, Nov. 2020. doi: <http://doi.org/10.1016/j.jmrt.2020.11.023>.

- [5] YANG, L., HAN, P., GU, Z., “Grafting of a novel hyperbranched polymer onto carbon fiber for interfacial enhancement of carbon fiber reinforced epoxy composites”, *Materials & Design*, v. 200, pp. 109456, Feb. 2021. doi: <http://doi.org/10.1016/j.matdes.2021.109456>.
- [6] WANG, J., CHEN, X., WANG, J., *et al.*, “High-performance, intrinsically fire-safe, single-component epoxy resins and carbon fiber reinforced epoxy composites based on two phosphorus-derived imidazoliums”, *Polymer Degradation & Stability*, v. 208, pp. 110261, Feb. 2023. doi: <http://doi.org/10.1016/j.polymdegradstab.2023.110261>.
- [7] TIAN, Z., WANG, Y., HOU, X., “Review of chemical recycling and reuse of carbon fiber reinforced epoxy resin composites”, *New Carbon Materials*, v. 37, n. 6, pp. 1021–1041, Dec. 2022. doi: [http://doi.org/10.1016/S1872-5805\(22\)60652-8](http://doi.org/10.1016/S1872-5805(22)60652-8).
- [8] HMEIDAT, N.S., ELKINS, D.S., PETER, H.R., *et al.*, “Processing and mechanical characterization of short carbon fiber-reinforced epoxy composites for material extrusion additive manufacturing”, *Composites. Part B, Engineering*, v. 223, pp. 109122, Oct. 2021. doi: <http://doi.org/10.1016/j.compositesb.2021.109122>.
- [9] YAO, S.-S., JIN, F.-L., RHEE, K.Y., *et al.*, “Recent advances in carbon-fiber-reinforced thermoplastic composites: A review”, *Composites. Part B, Engineering*, v. 142, pp. 241–250, Jun. 2018. doi: <http://doi.org/10.1016/j.compositesb.2017.12.007>.
- [10] LI, S., ZHAO, Y., ZHANG, Z., *et al.*, “Electrochemical and mechanical effects of acid and thermal treatments of carbon fiber”, *Advanced Engineering Materials*, v. 17, n. 1, pp. 52–57, Jan. 2015. doi: <http://doi.org/10.1002/adem.201400033>.
- [11] LIU, L., JIA, C., HE, J., *et al.*, “Interfacial characterization, control and modification of carbon fiber reinforced polymer composites”, *Composites Science and Technology*, v. 121, pp. 56–72, Dec. 2015. doi: <http://doi.org/10.1016/j.compscitech.2015.08.002>.
- [12] WANG, S., YANG, Y., MU, Y., *et al.*, “Synergy of electrochemical grafting and crosslinkable crystalline sizing agent to enhance the interfacial strength of carbon fiber/PEEK composites”, *Composites Science and Technology*, v. 203, pp. 108562, Feb. 2021. doi: <http://doi.org/10.1016/j.compscitech.2020.108562>.
- [13] CHEN, S., QIU, L., CHENG, H.-M., “Carbon-based fibers for advanced electrochemical energy storage devices”, *Chemical Reviews*, v. 120, n. 5, pp. 2811–2878, Mar. 2020. doi: <http://doi.org/10.1021/acs.chemrev.9b00466>. PubMed PMID: 32073258.
- [14] XIE, Y., “Electrochemical and hydrothermal activation of carbon fiber supercapacitor electrode”, *Fibers and Polymers*, v. 23, n. 1, pp. 10–17, Jan. 2022.
- [15] TRAN, T.-K., CHIU, K.-F., LIN, C.-Y., *et al.*, “Electrochemical treatment of wastewater: selectivity of the heavy metals removal process”, *International Journal of Hydrogen Energy*, v. 42, n. 45, pp. 27741–27748, Nov. 2017.
- [16] SUBBIAH, A., CHOCKALINGAM, P., MUNIMATHAN, A., *et al.*, “Effect of fiber orientation on interlaminar shear stresses and thermal property of sisal fiber reinforced epoxy composites”, *Matéria (Rio de Janeiro)*, v. 29, pp. e20240491, Oct. 2024.
- [17] SOROKIN, A.E., PETROVA, G.N., DONSIKIKH, I.N., “Use of chemical and electrochemical treatment methods in the liquid-phase modification of carbon fiber and fiberglass surfaces in the production of construction materials: a review”, *Theoretical Foundations of Chemical Engineering*, v. 54, n. 5, pp. 1061–1067, Sep. 2020.
- [18] YUAN, X., ZHU, B., CAI, X., *et al.*, “Influence of different surface treatments on the interfacial adhesion of graphene oxide/carbon fiber/epoxy composites”, *Applied Surface Science*, v. 458, pp. 996–1005, Nov. 2018.
- [19] CAKICI, M., KAKARLA, R.R., ALONSO-MARROQUIN, F., “Advanced electrochemical energy storage supercapacitors based on the flexible carbon fiber fabric-coated with uniform coral-like MnO<sub>2</sub> structured electrodes”, *Chemical Engineering Journal*, v. 309, pp. 151–158, Feb. 2017. doi: <http://doi.org/10.1016/j.cej.2016.10.012>.
- [20] TANG, W., ZHANG, Y., ZHONG, Y., *et al.*, “Natural biomass-derived carbons for electrochemical energy storage”, *Materials Research Bulletin*, v. 88, pp. 234–241, Apr. 2017.
- [21] RANDALL, J.D., EYCKENS, D.J., SERVINIS, L., *et al.*, “Designing carbon fiber composite interfaces using a ‘graft-to’ approach: Surface grafting density versus interphase penetration”, *Carbon*, v. 146, pp. 88–96, May. 2019. doi: <http://doi.org/10.1016/j.carbon.2019.01.064>.

- [22] CYSNE BARBOSA, A.P., FULCO, A.P., GUERRA, E., *et al.*, “Accelerated aging effects on carbon fiber/epoxy composites”, *Composites. Part B, Engineering*, v. 110, pp. 298–306, Feb. 2017. doi: <http://doi.org/10.1016/j.compositesb.2016.11.004>.
- [23] PARK, J., OH, H., HA, T., *et al.*, “A review of the gas diffusion layer in proton exchange membrane fuel cells: Durability and degradation”, *Applied Energy*, v. 155, pp. 866–880, Oct. 2015.
- [24] ALAM, P., MAMALIS, D., ROBERT, C., *et al.*, “The fatigue of carbon fibre reinforced plastics – A review”, *Composites. Part B, Engineering*, v. 166, pp. 555–579, Jun. 2019.
- [25] GONG, Y., LI, J., ZHANG, Y., *et al.*, “Partial degradation of levofloxacin for biodegradability improvement by electro-Fenton process using an activated carbon fiber felt cathode”, *Journal of Hazardous Materials*, v. 304, pp. 320–328, Mar. 2016. doi: <http://doi.org/10.1016/j.jhazmat.2015.10.064>. PubMed PMID: 26561756.
- [26] ARUMUGAM, V., KAVIPRIYA, S., TAJ, M.N.A.G., *et al.*, “Numerical simulation of confinement effect of CFRP and GFRP strengthened concrete specimens”, *Matéria (Rio de Janeiro)*, v. 29, pp. e20240010, Apr. 2024.
- [27] XIE, J.-M., ZHOU, X., LIU, Y., *et al.*, “Process parameters optimization for 3d printing of continuous carbon fiber reinforced composite”, *Matéria (Rio de Janeiro)*, v. 29, pp. e20240201, Jul. 2024.
- [28] ANDIDEH, M., ESFANDEH, M., “Statistical optimization of treatment conditions for the electrochemical oxidation of PAN-based carbon fiber by response surface methodology: application to carbon fiber/epoxy composite”, *Composites Science and Technology*, v. 134, pp. 132–143, Oct. 2016.
- [29] ZHANG, M., QIAN, X., MA, K., *et al.*, “Enhanced interfacial properties of high-modulus carbon fiber reinforced PEKK composites by a two-step surface treatment: electrochemical oxidation followed by thermoplastic sizing”, *Applied Composite Materials*, v. 29, n. 2, pp. 745–764, Apr. 2022.
- [30] CHEN, J., LI, G., LIU, Z., *et al.*, “Influence of the electrochemical treatment level of carbon fibers on the compression after impact property of carbon fiber/epoxy composites”, *Polymer Composites*, v. 44, n. 2, pp. 1213–1227, Feb. 2023.
- [31] HUANG, L., HE, Y., GAO, Z., *et al.*, “Hybrid-structured carbon fiber fabric/silk fiber non-woven fabric/carbonyl iron powder/epoxy composites with highly efficient electromagnetic interference shielding and mechanical properties”, *Composites Science and Technology*, v. 258, pp. 110868, Nov. 2024.
- [32] LI, H., LIEBSCHER, M., YANG, J., *et al.*, “Electrochemical oxidation of recycled carbon fibers for an improved interaction toward alkali-activated composites”, *Journal of Cleaner Production*, v. 368, pp. 133093, Sep. 2022. <http://doi.org/10.1016/j.jclepro.2022.133093>.
- [33] ZHANG, L., ZHOU, M., HE, Y., *et al.*, “Flexible porous non-woven silk fabric based conductive composite for efficient multimodal sensing”, *Chemical Engineering Journal*, v. 497, pp. 154445, Oct. 2024. doi: <http://doi.org/10.1016/j.cej.2024.154445>.
- [34] SUNDARESAN, R., MARIYAPPAN, V., CHEN, T.-W., *et al.*, “One-dimensional rare-earth tungstate nanostructure encapsulated reduced graphene oxide electrocatalyst-based electrochemical sensor for the detection of organophosphorus pesticide”, *Journal of Nanostructure in Chemistry*, v. 14, n. 5, pp. 355–368, Oct. 2024. doi: <http://doi.org/10.1007/s40097-023-00524-6>.
- [35] ANDIDEH, M., ESFANDEH, M., “Effect of surface modification of electrochemically oxidized carbon fibers by grafting hydroxyl and amine functionalized hyperbranched polyurethanes on interlaminar shear strength of epoxy composites”, *Carbon*, v. 123, pp. 233–242, Oct. 2017. doi: <http://doi.org/10.1016/j.carbon.2017.07.035>.
- [36] UNTERWEGER, C., DUCHOSLAV, J., STIFTER, D., *et al.*, “Characterization of carbon fiber surfaces and their impact on the mechanical properties of short carbon fiber reinforced polypropylene composites”, *Composites Science and Technology*, v. 108, pp. 41–47, Feb. 2015. doi: <http://doi.org/10.1016/j.compscitech.2015.01.004>.
- [37] GAO, B., ZHANG, R., HE, M., *et al.*, “Effect of a multiscale reinforcement by carbon fiber surface treatment with graphene oxide/carbon nanotubes on the mechanical properties of reinforced carbon/carbon composites”, *Composites. Part A, Applied Science and Manufacturing*, v. 90, pp. 433–440, Nov. 2016. doi: <http://doi.org/10.1016/j.compositesa.2016.08.012>.
- [38] STOJCEVSKI, F., HILDITCH, T.B., HENDERSON, L.C., “A comparison of interfacial testing methods and sensitivities to carbon fiber surface treatment conditions”, *Composites. Part A, Applied Science and Manufacturing*, v. 118, pp. 293–301, Mar. 2019. doi: <http://doi.org/10.1016/j.compositesa.2019.01.005>.

- [39] HE, Y., CHEN, Q., WU, D., *et al.*, “Effect of multiscale reinforcement by fiber surface treatment with polyvinyl alcohol/graphene oxide/oxidized carbon nanotubes on the mechanical properties of reinforced hybrid fiber composites”, *Composites Science and Technology*, v. 204, pp. 108634, Mar. 2021. doi: <http://doi.org/10.1016/j.compscitech.2020.108634>.
- [40] MIRZAEI RAD, F., TAFVIZI, F., NOORBAZARGAN, H., *et al.*, “Ag-doped ZnO nanoparticles synthesized through green method using Artemisia turcomanica extract induce cytotoxicity and apoptotic activities against AGS cancer cells: an in vitro study”, *Journal of Nanostructure in Chemistry*, v. 14, n. 6, pp. 403–418, Dec. 2024. doi: <http://doi.org/10.1007/s40097-023-00528-2>.
- [41] SUN, J., ZHOU, X., WEI, X., *et al.*, “Study on bending properties and damage mechanism of carbon fiber reinforced aluminum laminates”, *Matéria (Rio de Janeiro)*, v. 29, n. 3, pp. e20240310, Jul. 2024. doi: <http://doi.org/10.1590/1517-7076-rmat-2024-0310>.
- [42] LIU, Z., ZHANG, L., YU, E., *et al.*, “Modification of glass fiber surface and glass fiber reinforced polymer composites challenges and opportunities: from organic chemistry perspective”, *Current Organic Chemistry*, v. 19, n. 11, pp. 991–1010, Jun. 2015. doi: <http://doi.org/10.2174/138527281911150610100914>.
- [43] CHEN, J., ZHU, Y., NI, Q., *et al.*, “Surface modification and characterization of aramid fibers with hybrid coating”, *Applied Surface Science*, v. 321, pp. 103–108, Dec. 2014. doi: <http://doi.org/10.1016/j.apsusc.2014.09.196>.
- [44] SONG, H., HE, G., HE, Y., *et al.*, “Effects of surface modification on mechanical properties and electromagnetic interference shielding properties of carbon fiber/silk fiber hybrid composites”, *Polymer Composites*, v. 45, n. 11, pp. 10276–10289, 2024. doi: <http://doi.org/10.1002/pc.28472>.
- [45] ZARE, N., KARIMI-MALEH, H., ZHANG, Z., *et al.*, “Enhancing cancer biomarker identification: precise monitoring of MUC1 using V2C/Au nanocomposite-amplified electrochemical biosensor”, *Carbon Letters*, Mar. 2025.
- [46] HIEU, N.H., DUYEN, D.T.M., THANG, T.Q., *et al.*, “Fabrication of reduced graphene oxide-doped carbon aerogels from water hyacinth for removal of methylene blue in water and energy storage”, *Journal of Nanostructure in Chemistry*, v. 14, n. 6, pp. 383–401, Dec. 2024. doi: <http://doi.org/10.1007/s40097-023-00526-4>.
- [47] CAUICH-CUPUL, J.I., HERRERA-FRANCO, P.J., GARCÍA-HERNÁNDEZ, E., *et al.*, “Factorial design approach to assess the effect of fiber-matrix adhesion on the IFSS and work of adhesion of carbon fiber/polysulfone-modified epoxy composites”, *Carbon Letters*, v. 29, n. 4, pp. 345–358, Aug. 2019. doi: <http://doi.org/10.1007/s42823-019-00039-7>.
- [48] BEGGS, K.M., RANDALL, J.D., SERVINIS, L., *et al.*, “Increasing the resistivity and IFSS of unsized carbon fibre by covalent surface modification.”, *Reactive & Functional Polymers*, v. 129, pp. 123–128, 2018. doi: <http://doi.org/10.1016/j.reactfunctpolym.2017.06.016>.