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Mitigating Saline Degradation and Enhancing Tensile Performance in CFRP Laminates Using Graphene-CNT Hybrid Nanoparticles

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Carbon fiber reinforced polymer (CFRP) composites, although frequently preferred in many fields where weight is critical due to their high specific strength, are susceptible to environmental degradation in marine conditions. The primary cause of this degradation is the ingress of moisture into the structure. This leads to significant swelling of the matrix and dimensional mismatches at the fiber-matrix interface. This study investigates the reduction of such saline degradation and the enhancement of tensile performance in CFRP laminates by modifying the epoxy matrix with a 0.5% by weight of graphene doped multi-walled carbon nanotube (MWCNT) hybrid nanoparticles. Nanoparticle modified CFRP laminates were produced and subjected to a 15-day salt spray aging treatment (5% NaCl, 35 °C). Under non-aged conditions, the addition of hybrid nanoparticles (CNT_N-A) significantly improved tensile performance, increasing the average tensile strength by 7.98% compared to the neat reference (REF_N-A) configuration. After salt spray exposure, the modified laminates (CNT_A) exhibited 3.90% moisture absorption, which led to a decrease in tensile strength (6.11%) due to matrix plasticization. Nevertheless, the aged modified composites performed better than the non-aged neat CFRP, exhibiting a 1.38% higher tensile strength. These findings highlight the promising potential of graphene-MWCNT hybrid nanoparticles in enhancing the environmental durability and load-bearing capacity of CFRP structures in harsh saline environments.

1. Introduction

Fiber-reinforced polymer composites are widely used as structural materials in industries such as aerospace, marine, automotive, and construction due to their high specific strength, excellent stiffness, design flexibility, and outstanding resistance to environmental corrosion [1,2]. Among these materials, carbon fiber reinforced polymer (CFRP) composites have attracted particular attention due to their high modulus-to-weight ratio and their potential to replace conventional metallic materials in lightweight structural applications [3]. With advancing technology, the use of CFRP-based composite structures in ocean energy systems, wind turbine blades, ship hulls, and offshore operation platforms has become increasingly important [1].

Despite their superior mechanical performance, composite materials are susceptible to degradation in their mechanical, physical, and chemical properties when exposed to harsh service environments such as marine or hot-wet conditions [3,4]. Environmental factors, including moisture, temperature fluctuations, corrosive media, and ultraviolet radiation, can progressively alter the internal structure of polymer-based composites. In particular, water absorption affects the polymer matrix through mechanisms such as plasticization, swelling, hydrolysis, and microcrack formation [5]. Although carbon fibers themselves are relatively insensitive to moisture, the epoxy matrix is much more vulnerable to the action of water molecules and dissolved ions [6]. Matrix softening and the reduction in glass transition temperature weaken the fiber–matrix interfacial bonding, especially in systems with mismatched thermal expansion behavior, which may ultimately promote delamination and lead to significant reductions in strength and environmental durability [5,7].

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Improving the environmental durability of polymer matrix composites has become one of the major research priorities for ensuring their long-term and reliable use in harsh service environments [4]. To address the environmental limitations of conventional composites, various strategies have been developed, ranging from macroscopic solutions such as fiber metal laminates (FMLs), which combine composite and metallic layers, to surface treatments including acid, plasma, and ozone modification aimed at strengthening the fiber–matrix interface [6]. More recently, one of the most promising approaches has been the modification of the polymer matrix with micro- and nano-scale fillers, which can enhance interfacial performance while also acting as barriers against environmental ingress [8].

Nanocomposite systems containing very low nanofiller contents, typically below 2 wt.%, can improve the interphase region, promote mechanical interlocking, and enhance toughness by deflecting or hindering crack propagation without causing a significant increase in weight or volume [6]. Among the available nano-reinforcements, carbon-based nanofillers, particularly carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs), have attracted considerable attention because of their high specific surface area, exceptional stiffness, and thermal stability [9,10]. The two-dimensional structure of graphene provides a large contact area and can contribute to barrier formation within the fiber–matrix region, whereas the one-dimensional morphology of CNTs enables them to penetrate the polymer network and act as nanoscale bridges across developing cracks [11]. When these two nanofillers are combined, a synergistic effect may be achieved through the formation of interconnected three-dimensional reinforcement networks, which can improve stress transfer and contribute to enhanced mechanical and environmental performance [11,12].

Under environmental aging conditions, nano-modified composites often exhibit improved resistance to degradation compared with neat resin systems. Nanofillers such as graphene and CNTs can increase the tortuosity of diffusion pathways for water molecules and salt ions, thereby slowing moisture ingress and reducing the extent of epoxy plasticization and hydrolytic damage. Previous studies have shown that graphene-based additives can enhance resistance to long-term hygrothermal or moisture exposure, limit the reduction in glass transition temperature, and mitigate losses in storage modulus. Similarly, CNT-based reinforcements have been reported to suppress interfacial degradation and reduce the deterioration of mechanical properties such as tensile, flexural, and impact-related performance in aged composites [6].

A growing body of literature has examined the performance of composite materials under salt-fog (salt spray) aging, which is commonly used to simulate marine corrosion environments and is generally considered more aggressive than simple moisture exposure. Unlike conventional moisture uptake, salt-fog aging also involves the diffusion of sodium chloride (NaCl) ions into the polymer network and their accumulation within micro-defects in the matrix [13]. Lin et al. reported that, in stitched woven composites, salt-fog penetrated resin cracks, plasticized the matrix, and significantly reduced the glass transition temperature (Tg). They further observed that increasing aging cycles led to matrix softening, reductions in Mode I fracture toughness, and enhanced debonding in resin-rich regions [1]. Similarly, Zhao et al. investigated the bending fatigue behavior of 3D woven composites under salt-fog corrosion and showed that prolonged exposure allowed water and salt species to penetrate the matrix and fiber–matrix interface, contributing to the degradation of resin chemical bonds and a marked reduction in static bending properties and fatigue life [13]. Studies on flax fiber reinforced composites exposed to alternating salt-fog/dry conditions have also shown that salt-fog exposure can induce irreversible microcracking and interfacial deterioration, while subsequent evaporation may leave voids within the structure and further weaken the material [14]. In addition, aggressive saline aging methods such as boiling saltwater exposure have been reported to accelerate fiber–matrix bond degradation, epoxy hydrolysis, and loss of mechanical performance. Taken together, these studies indicate that salt-fog aging is not merely a moisture absorption phenomenon, but a more complex degradation process involving both physical and chemical damage mechanisms that can promote irreversible interfacial cracking and delamination in composites [15].

Although the roles of carbon nanotubes and graphene in improving composite performance and mitigating hygrothermal degradation have been widely investigated, the behavior of carbon fiber reinforced epoxy composites modified with a commercially supplied graphene-doped carbon nanotube nanofiller under salt-fog (salt spray) aging remains insufficiently explored. In particular, limited attention has been given to the tensile behavior of CFRP laminates containing a ready-to-use nanofiller consisting of carbon nanotubes doped with 32 wt.% graphene nanopowder and incorporated into the matrix as received. Therefore, the aim of this study is to modify the epoxy matrix of CFRP laminates with this commercially available

graphene-doped MWCNT nanofiller and to evaluate its effect on tensile performance before and after salt spray aging. For this purpose, an unmodified non-aged reference group, a nanofiller-modified non-aged group, and a nanofiller-modified aged group were comparatively investigated. The findings are expected to provide experimental insight into whether the tensile load-carrying capability improved by nanofiller addition can be maintained after saline exposure, and to clarify the potential of graphene-doped carbon nanotube nanofillers for CFRP composites intended for marine and other harsh service environments.

2. Results and Discussion

The individual tensile force–extension curves of the three experimental groups are presented in Figures 1–3, while their overall comparative behavior is illustrated in Figure 4. The reference non-aged specimens (REF_N-A) exhibited a relatively linear increase in load up to failure, followed by an abrupt drop in the post-peak region, which is characteristic of brittle fracture-dominated behavior in fiber-reinforced epoxy laminates. Some scatter was observed among the three reference specimens, particularly in terms of peak load and extension at failure, indicating specimen-to-specimen variability within the neat laminate group.

For the nanofiller-modified non-aged group (CNT_N-A), the force–extension curves showed a similarly increasing load response, but with generally higher peak load levels than those of the reference specimens. The curves in this group were also closely spaced prior to failure, suggesting a more stable tensile response under non-aged conditions. In comparison with the neat reference group, the modified laminates sustained higher loads over a comparable extension range, indicating that the addition of graphene-doped MWCNT nanofiller improved the tensile load-carrying behavior of the CFRP laminates.

After salt spray aging, the nanofiller-modified aged group (CNT_A) still exhibited force–extension curves with peak loads above those of the neat reference group, although a reduction relative to the non-aged modified group was observed. The overall shape of the curves remained similar, but the peak force level decreased after aging, which is consistent with the expected deterioration induced by saline environmental exposure [16].

As clearly depicted in the comparative curves (Figure 4), the nanoparticle-modified laminates maintain a superior load-carrying capacity and structural stability compared to the neat reference, even after saline exposure. The numerical results obtained from the tensile tests are summarized in Table 1. As seen in Table 1, the average maximum load values obtained from the tensile tests were approximately 25.9 kN for REF_N-A, 28.4 kN for CNT_N-A, and 27 kN for CNT_A. These results indicate that the addition of 0.5 wt.% graphene-doped MWCNT nanofiller increased the maximum load of the non-aged CFRP laminates by approximately 9.75% compared with the neat reference group. This finding suggests that the nanofiller contributed positively to the load-bearing capacity of the composite system under unaged conditions. The findings are consistent with similar improvements reported in the literature [17,18].

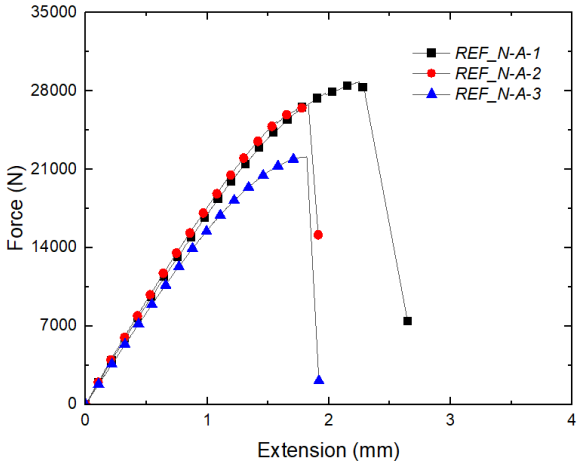

Figure 1. Tensile force–extension curves of the neat non-aged reference specimens.

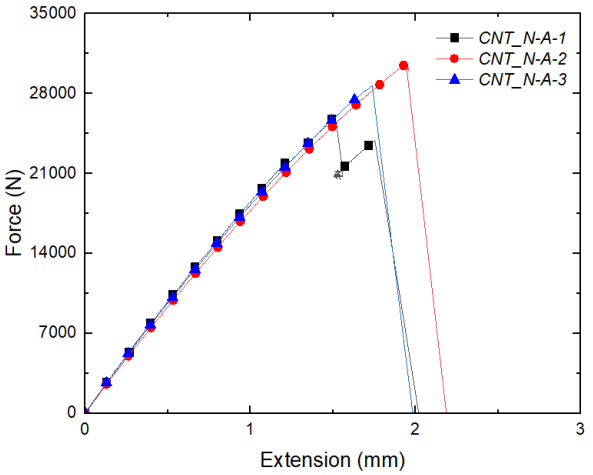

Figure 2. Tensile force–extension curves of the nanoparticle modified non-aged specimens.

When the modified laminates were subjected to salt spray aging, the average maximum load decreased from 28.4 kN to 27 kN, corresponding to a reduction of approximately 4.89%. This reduction was accompanied by a 3.90% water intake in the aged nanofiller-modified specimens, supporting the interpretation that moisture absorption during salt spray exposure contributed to the observed loss in

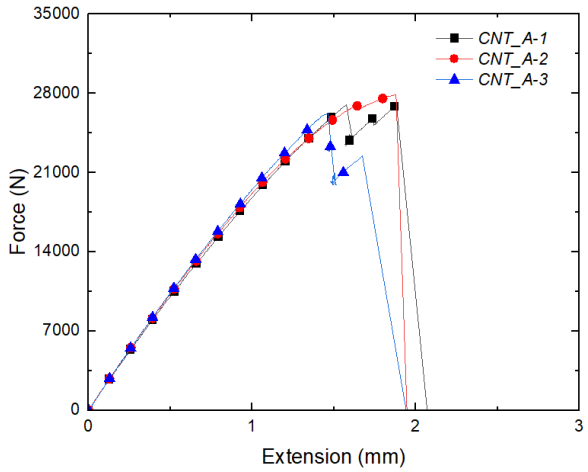

Figure 3. Tensile force–extension curves of the nanoparticle modified aged specimens.

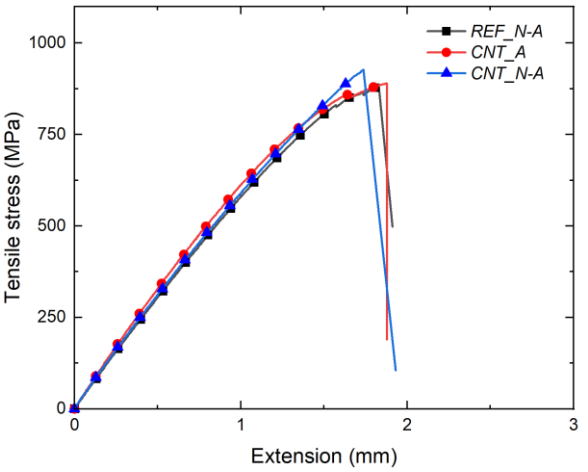

Figure 4. Comparative tensile stress–extension curves of the neat and nanoparticle-modified CFRP configurations.

tensile performance. This decrease is consistent with the expected adverse effect of saline environmental exposure on epoxy-based composite systems. Fang and Guo indicated that this type of degradation is fundamentally caused by the severe swelling of the epoxy matrix due to moisture ingress, which creates a critical dimensional mismatch with the non-absorbent carbon fibers. This internal expansion mismatch generates stress that induces irreversible interfacial debonding and micro-cracks [19]. Nevertheless, the aged nanofiller-modified group still exhibited a higher average maximum load than the unaged neat reference group, with an improvement of approximately 4.38%. It indicates that even after environmental aging, the nanofiller-modified laminates maintained a load-carrying capacity above that of the baseline neat composite.

The same trend was observed in the tensile strength values calculated from the cross-sectional areas of the specimens. The average tensile strength was 851.94 MPa for REF_N-A, 919.89 MPa for CNT_N-A, and 863.69 MPa for CNT_A. Compared with the neat reference laminate, the non-aged nanofiller-modified group showed an increase of approximately 7.98% in tensile strength. After salt spray aging, the tensile strength of the modified laminates decreased by approximately 6.11% relative to CNT_N-A. However, the aged modified group remained slightly above the neat reference group, showing an advantage of approximately 1.38%. These results indicate that the nanofiller not only improved the initial tensile performance of the CFRP laminates but also allowed the aged laminates to retain a tensile strength comparable to, and slightly higher than, that of the neat reference system. The improvement observed in the non-aged condition can be attributed to the contribution of the carbon-based nanofiller to matrix reinforcement and stress transfer efficiency within the composite structure. In epoxy-based systems, the synergistic combination of one-dimensional carbon nanotubes and two-dimensional graphene constructs a multi-dimensional interconnected network that efficiently transfers stress. As demonstrated in recent studies, these hybrid fillers may act as nanoscale stress-transfer elements that significantly delay the local development of matrix-dominated damage under tensile loading through mechanisms such as interfacial mechanical interlocking, crack deflection, and crack bridging [20–22].

The increase in both maximum load and tensile strength is in agreement with the general trend reported in the literature for carbon-based nano-reinforced polymer composites, where graphene- and CNT-containing fillers often improve load transfer, crack deflection resistance, and the overall stiffness-strength response of the matrix-dominated regions. In the present case, a commercially supplied graphene-doped MWCNT nanofiller at a relatively low loading of 0.5 wt.% was sufficient to produce a measurable and significant increase in tensile performance.

The comparison between CNT_N-A and CNT_A demonstrates that salt spray aging caused a clear reduction in tensile performance, as expected for CFRP laminates exposed to a saline environment. Both maximum load and tensile strength decreased after 15 days of salt spray exposure at 35 °C and 5% salinity. In addition, the aged nanofiller-modified group exhibited a water intake of 3.90%, indicating that moisture uptake occurred during environmental exposure. This absorbed moisture is likely to have contributed to matrix plasticization

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Table 1. Maximum load and tensile strength values of the tested CFRP laminate groups.

Tensile test results	REF_N-A	CNT_N-A	CNT_A
	28,779.15	25,990.68	26,972.06
P_{max} (N)	26,803.79	30,627.61	27,849.77
	22,114.04	28,655.74	26,278.78
Aveg. P_{max} (N)	25,898.99	28,424.68	27,033.54
Tensile strength (MPa)	851.94	919.89	863.69

and to the gradual weakening of the fiber–matrix interphase, which are widely recognized as major degradation mechanisms in epoxy-based composite systems under saline or hygrothermal conditions [15].

3. Conclusion

This study evaluated the tensile performance of neat and graphene-doped MWCNT modified CFRP laminates before and after salt spray aging. The incorporation of 0.5 wt.% commercially supplied graphene-doped MWCNT nanofiller improved the tensile response of the laminates under non-aged conditions. Compared with the neat non-aged reference (REF_N-A), the nanofiller-modified non-aged laminates (CNT_N-A) exhibited a 9.75% increase in average maximum load and a 7.98% increase in tensile strength.

Salt spray aging, performed for 15 days at 35 °C in a 5% saline environment, reduced the tensile performance of the modified laminates. Relative to CNT_N-A, the aged modified group (CNT_A) showed a 4.89% decrease in average maximum load and a 6.11% decrease in tensile strength. This reduction was accompanied by a 3.90% water intake, indicating moisture absorption during environmental exposure and supporting the observed degradation in tensile response.

Despite this decrease, the aged nanofiller-modified laminates remained mechanically competitive with the neat reference system. In particular, CNT_A exhibited a 4.38% higher average maximum load and a 1.38% higher tensile strength than REF_N-A. These results indicate that the graphene-doped MWCNT nanofiller not only enhanced the initial tensile load-carrying capability of the CFRP laminates, but also enabled the modified system to retain a practically useful performance level after salt spray aging.

One limitation of the present study is the absence of an aged neat CFRP control group, which restricts the direct separation of intrinsic nanofiller reinforcement effects from environmental durability effects. Future studies should include an aged neat reference group for a more comprehensive evaluation of degradation and retention behavior.

Overall, the findings highlight the potential of commercially supplied graphene-doped carbon nanotube nanofillers as an effective matrix modification route for CFRP laminates intended for marine and other harsh environmental applications. Since the present work was limited to tensile testing and did not include direct microstructural characterization, future studies should focus on correlating the retained mechanical response with interfacial and morphological changes induced by saline aging.

Method

Unidirectional carbon fiber fabric with an areal weight of 300 g/m^2 was used as the reinforcement material in the production of the composite laminates. According to the supplier data (Dost Kimya, Inc), the carbon fiber has a density of 1.79 g/cm^3, a tensile strength of 4900 MPa, a tensile modulus of 240 GPa, and a tensile strain of 2% [23].

An epoxy resin system consisting of MGS L160 epoxy resin and H160 hardener was used as the polymer matrix. The resin system was selected due to its suitability for room-temperature curing and composite laminate manufacturing [23]. A commercially supplied carbon nanotube-based nanofiller doped with 32 wt.% graphene nanopowder (Nanografi Nanotechnology, product no. NG01SC0606) was used as received and was incorporated into the epoxy matrix at a loading level of 0.5 wt.% for the preparation of the nanofiller-modified composite groups [24].

The composite laminates were manufactured by the hand lay-up and vacuum bagging method. Initially, the epoxy resin system was prepared. For the modified group, the graphene-doped MWCNT nanofiller was added to the epoxy at a concentration of 0.5 wt.%. The ultrasonication technique was applied to homogenize the epoxy resin with the nanoparticles. To prevent the resin from overheating during ultrasonication, the process was carried out whilst the mixture was in an ice bath. The final stage of resin preparation involved the mechanical mixing of the nanoparticle-containing epoxy with the hardener. In accordance with the manufacturer's recommendation, the mixture was prepared at a weight ratio of 4:1 epoxy to hardener

[23]. The resulting mixture was then used to impregnate the unidirectional carbon fiber plies by hand lay-up. The laminate consists of a total of 14 layers, arranged in the stacking sequence of $[0°, 90°, 0°, 90°, 0°, 90°, 0°]_s$.

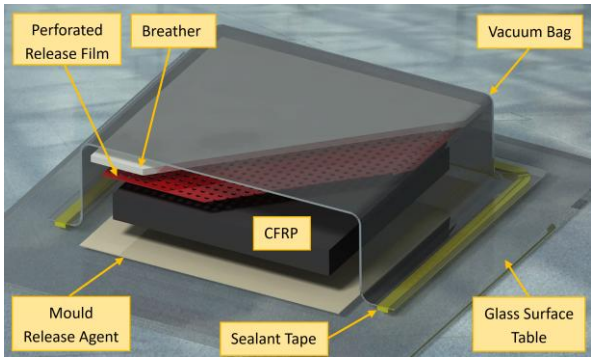

Figure 5. A schematic representation of the vacuum bagging method applied in this study.

After lay-up, a perforated release film and a breather layer were placed over the laminate stack, and a vacuum bagging film was used to cover the lamination. Airtight sealing was achieved using sealant tape along the mold perimeter. A vacuum was applied using a vacuum pump to an absolute pressure of 1 atm, and the laminates were consolidated under vacuum throughout the curing stage. A production schematic showing the components used in the vacuum bagging method is shown in Figure 5. All laminates were cured at room temperature. To investigate the influence of nanofiller addition and the retained tensile performance after salt spray aging, neat CFRP laminates and nanofiller-modified CFRP laminates were produced under the same manufacturing conditions.

After the fabrication of composite plates was completed, the tensile specimens were prepared. The specimens were classified into three categories in the scope of this study. The designations were made as follows:

REF_N-A: neat CFRP laminate, non-aged configuration.

CNT_N-A: 0.5 wt.% graphene-doped MWCNT modified CFRP laminate, non-aged configuration.

CNT_A: 0.5 wt.% graphene-doped MWCNT modified CFRP laminate, salt spray aged configuration.

This grouping was established to evaluate the influence of nanofiller addition under non-aged conditions and to determine the extent to which the tensile response of the nanofiller-modified laminates was affected by salt spray aging.

Salt spray aging was applied only to the nanofiller-modified specimens designated for environmental exposure. Aging was carried out in a salt spray chamber for 15 days at 35 °C in a saline environment at 5% concentration. The specimens subjected to aging and the aging parameters set via the device control panel are shown in Figure 6. At the end of the exposure period, the aged specimens were removed from the chamber and prepared for tensile testing. The non-aged specimens were kept under laboratory conditions and used as reference groups for comparative evaluation.

(a)

(b)

Figure 6. (a) Testing specimens exposed to salt spray aging, (b) control panel showing aging environment.

Tensile tests were performed in accordance with ASTM D638 using an Instron 8801 universal testing machine in order to evaluate the effect of nanofiller addition and salt spray aging on the tensile response of the CFRP laminates. For each experimental group, three specimens were tested under the same conditions. The specimens were 165 mm in length, 19 mm in width, remaining inside the crosshead grippers, and the gauge length section was 6 mm wide. Tested specimens are shown in Figure 7. During testing, force–extension data were continuously recorded. Since no extensometer was used, the displacement values correspond to the crosshead displacement of the testing machine. Therefore, the mechanical evaluation in the present study was based primarily on comparisons of maximum load, force–extension response, and cross-section-normalized tensile strength among the groups.

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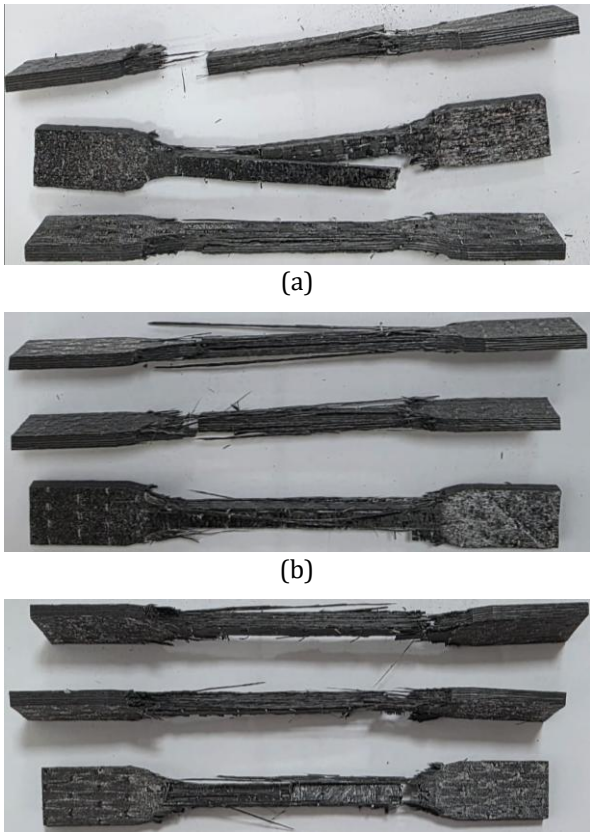
The obtained force–extension curves were used to determine the maximum load values and to compare the tensile behavior of the neat and nanofiller-modified composites under non-aged and aged conditions. After determining the specimen cross-sectional areas from width and thickness measurements, the maximum load values were converted into tensile strength values for each specimen.



(a) (b) (c)

Figure 7. Specimens after tensile testing; a) REF_N-A, b) CNT_N-A, c) CNT_A.

Authors' Contributions

DCA: Methodology, data collection, data analysis, and interpretation, writing of the original draft. **YY:** Methodology, data analysis. **ÇU:** Methodology, data collection, data analysis, and writing – review and editing.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this studydonot require ethical committee permission and/or legal-special permission.

Conflict of Interest

There is no conflict of interest in this study.

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