

A comparative study of the effect of polyurethane nanofiber and powders filler on the mechanical properties of carbon fiber and glass fiber composites

Poliüretan nanofiber ve toz takviyelerinin karbon fiber ve fiberglas kompozitlerin mekanik özellikleri üzerine etkisinin karşılaştırması

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Abstract

In this study, the polyurethane (PU) nanofibers mat produced by the electrospinning method was used as filler in the polymer composites; fiberglass (FG) and carbon fiber (CF) were employed as reinforcement materials. Moreover, their mechanical properties were compared to sepiolite and SiO₂ powder reinforced composites. The mechanical properties of composites were evaluated using tensile and flexural tests. Moreover, the morphology of the composite was assessed by microscopy techniques. The findings show that the nanofiber-doped FG and CF composite had at least 30% higher tensile strength compared to unreinforced and rival composites; the tensile strength was 135 MPa for FG-Nanofibers and 134 MPa for CF-Nanofibers. On the other hand, the flexural analysis showed that the powders filler had poor flexural stress against untreated composite. However, the flexural strength of FG-Nanofibers and CF-Nanofibers reached 197 MPa and 553 MPa, respectively. The better mechanical properties of nanofiber doped composites were due principally to the random distribution of nanofibers which support all loads from different directions. The cross-section analysis of composites demonstrated that the powders were distributed heterogeneously and some agglomeration was observed at some locations. Generally speaking, it can be concluded that the addition of PU nanofibers increased the mechanical properties of composites, and the tensile strength was at least 3 times higher for CF composites compared to untreated composite.

Keywords: Nanofiber, Polyurethane, Glass fiber, Carbon fiber, Composite.

Öz

Bu çalışmada, elektrospinning yöntemi ile üretilen poliüretan (PU) nanofiber mat, fiberglas (FG) ve karbon fiber (CF) polimer kompozitlerde matrise ilave dolgu maddesi olarak kullanıldı. Ayrıca mekanik özellikleri sepiolit ve SiO₂ toz takviyeli kompozitlerle karşılaştırılmıştır. Kompozitlerin mekanik özellikleri çekme ve eğilme testleri kullanılarak değerlendirildi. Bunun yanında, kompozitin morfolojisi mikroskopi teknikleriyle değerlendirildi. Bulgular, nanofiber katkılı FG ve CF kompozitin, takviyesiz ve rakip kompozitlere kıyasla en az %30 kadar daha yüksek çekme mukavemetine sahip olduğunu göstermektedir; gerilme mukavemeti, FG-Nanofiberler için 135 MPa ve CF-Nanofiberler için 134 MPa bulundu. Öte yandan, eğme analizi, toz dolgu maddesinin işlenmemiş kompozite karşı en az 2.5 kat daha zayıf eğilme gerilimine sahip olduğunu gösterdi. Bununla birlikte, FG-Nanofiberler ve CF-Nanofiberler sırasıyla 197 MPa ve 553 MPa eğme mukavemetine ulaştı. Nanofiber katkılı kompozitlerin daha iyi mekanik özellikleri göstermesi, farklı yönlerden tüm yükleri destekleyen nanofiblerin rastgele dağılımından kaynaklanmaktadır. Kompozitlerin enine kesit analizi sonucunda, tozların heterojen olarak dağıldığını ve bazı yerlerde bir miktar aglomerasyon gözlemlendiğini gösterdi. Genel olarak, PU nanofiblerin eklenmesinin kompozitlerin mekanik özelliklerini arttırdığı ve CF kompozitleri için çekme mukavemetini katkısız kompozite göre en az 3 katı arttırdığı bulunmuştur.

Anahtar kelimeler: Nanofiber, Poliüretan, Cam elyafı, Karbon fiber, Kompozit.

1 Introduction

Polymer matrix composites (PMCs) were the most favored material in such industries as automotive, aerospace, sports equipment, and navy due to these materials' being lightweight, corrosion-resistant as well as due to their higher mechanical properties [1]-[6]. The PMCs were composed usually of epoxy resin such as polymer matrix and were often reinforced with glass, carbon, aramid, and polyethylene fibers. The demand for advanced PMC materials by the industry drove researchers to modify the properties of the polymer matrix by the addition of micro- and nano-scaled materials [7]-[9]. Different types of

fillers were considered to enhance the mechanical properties of polymer composites such as rubber particles, nano-scaled clays, Al₂O₃, SiO₂, graphene oxide, and nanofibers. [10]-[17]. Nanofibers had advantages compared to other fillers because they had better dispersion and because they do not increase the viscosity in the resin [18]. Very few studies were realized on the effect of polyurethane nanofibers on the mechanical properties of fiberglass composite, [19] and their influence on the carbon fibers composite was still not investigated properly. Different nanofibers made of poly (ε-caprolactone) (PCL), poly (vinylidene fluoride) (PVDF), polyacrylonitrile (PAN), and Polyamide 6 (PA6) were examined in the composite as filler [8],

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[14],[20]-[23]. For example, Zhou et al. [16] attempted to improve the carbon fabric-epoxy composite using 1 wt.%-3 wt.% carbon nanofibers (the fiber diameter was between 60-200 nm). The maximum strength of composite reached 68.98 MPa while the young modulus was 3.17 GPa with the addition of 2 wt.% carbon nanofibers after the tensile test. Moreover, the flexural test showed an increase of 22.3% of the strength of 2 wt. % nanofibers-doped composite compared to the unreinforced one. The use of thermoplastic nanofiber-based material as interlay was studied by De Schoenmaker et al. [8] on glass fiber/ epoxy. The PA6 deposited directly by the electrospinning process to glass fiber composite had better strength with 611 MPa with the young modulus of 27 GPa compared to interlayered composite. Also, delamination and cracks were decreased because of the formation of the barrier by the addition of nanofibers. On the other hand, the toughness properties of carbon/epoxy reinforced with different nanofibers as an interlayer in the resin were studied by Zhang et al. [20]. Findings showed that the polymerization-induced phase partition was decisive on the toughness of the composite. PCL nanofibers provided better toughening compared to other thermoplastic nanofibers. Despite heterogeneous dispersion of nanoclays and nanopowders, some works showed enhancement of mechanical properties of the composite [5], [11]-[13],[24]-[27]. The SiO₂ filler was used in a different industry due to nontoxic, biocompatible and highly thermal resistance properties; for example, they were used in the naval composite to enhance impact damage. Landowski et al. [5] inserted in epoxy resin matrix between 1- 8 wt.% SiO₂ nanoparticles; they reported that with the addition of 8 wt.% SiO₂ the impact damage size decreased by ~ 28 % while flexural strength decreased after the addition of 5 wt. % SiO₂. Tsai et al. [27] showed that 20 wt. % SiO₂ nanoparticles doped in the glass/epoxy exhibited higher compressive strength than unreinforced glass/epoxy. On the other hand, the mixture of nano and micro filler was studied by Manjunath et al. [7]. They reported that reinforced glass/epoxy with nano-sized alumina (3 wt.%) and silica (7 wt.%) slightly increased the strength, but the addition of micro alumina trihydrate (5 wt.%) in the nano-doped composite increased the strength by 9 %. Higher flexural strength and modulus were obtained with the hybrid combination of 3Al₂O₃+ 2SiO₂+ 5 alumina trihydrate +glass/epoxy). Regarding nano clay fillers, montmorillonite clay was the most frequently used in fabric nanocomposite [28]. Before using them in the composite, clays were modified into an organophilic structure by ammonium iron or phosphonium ion. The nano-sized organo-montmorillonite (OMMT) with different ratios from 0 to 10 wt.% was added to epoxy with variant mixture sequence by Yap and Chow [29]. The flexural strength and modulus were higher when applying method 3 (diglycidyl ether-bisphenol A (DGEBA) and curing agent were mixed, followed by the addition of OMMT) compared to methods 1 and 2. Moreover, different clays as reinforcement such as sepiolite, chlorite, and kaolinite were also under investigation [24],[30],[31].

This study aims to investigate the PU nanofiber mat reinforcement effect on the mechanical properties of CF and FG composite. Moreover, the performance of PU nanofibers was analysed against different-sized powders. To this end, PU nanofibers mat, sepiolite powder, and SiO₂ nanopowder were used as filler materials. The first part of this study focused on the morphology of the composites that were examined by optic

microscope (OM) and scanning electron microscopy (SEM). In addition, an X-Ray diffractometer (XRD) was employed to determine the phase of the composite. The second part of the study concentrated on evaluating the strength of the composite using tensile and flexural tests.

2 Materials and methods

2.1 Preparation of materials

Commercial fiberglass fabric plain of 300 g/m² (Product code: EE300, warp: 68, weft: 68, width: 0,19 mm ± 15%) with fiber orientation 0°/90°, carbon fiber plain of 90 g/m² (Product code: CC90, warp: HS1K , weft: HS1K, width: 0,12 mm ± 15%) with fiber orientation 0°/90°, Polipol™ 3401-TAB polyester resin, Cobalt oktoate 6%, Metil etil keton peroksit (MEK-P) hardener, EPIKOTETM Resin MGS® L 160, EPIKURETM Curing Agent MGS® H 160 were purchased from DOST KIMYA AŞ. Figure 1.

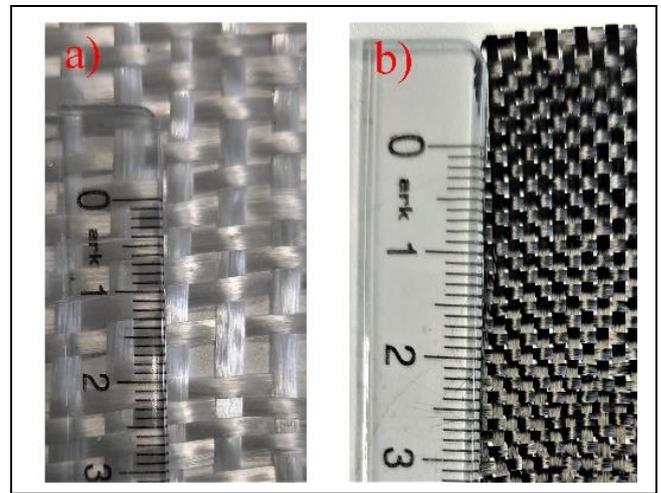


Figure 1. Macro image of fiberglass fabric. (a): and carbon fiber. (b): Plain.

Of the filler materials, nanopowder Silica (SiO₂) having 10-20 nm particle size and powder sepiolite (Mg₄Si₆O₁₅(OH)₂·6H₂O) were acquired from Sigma-Aldrich. The PU nanofibers mat was produced randomly using the electrospinning technique Figure 2. The method of production of PU nanofibers was explained elsewhere [32]-[34].

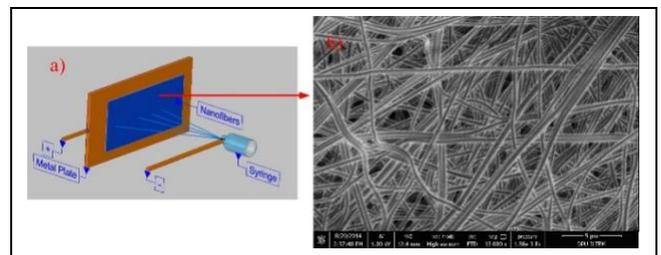


Figure 2(a): Electrospinning technique. (b): The SEM image of PU nanofibers at a magnification of 10 000x.

2.2 Methods of composite fabrication

The fiberglass and carbon fiber fabric was cut 500X500 mm before the application of resin and hardener. The hand lay-up method was used, and the stacking sequence was [0°, 90°] 2S meaning 2 plies were employed to fabric composite. For the preparation of fiberglass composite matrice; Polipol™ 3401-TAB polyester resin, 0.2% cobalt octoate 6 %, and 1% Metil

ethyl ketone peroksit (MEK-P) hardener was employed. Carbon fiber composite matrix was prepared using EPIKOTETM Resin MGS® L 160 and EPIKURETM Curing Agent MGS® H 160 with a ratio of the weight of 100/28, respectively. A 10 wt. % powder filler was added to the mixture of resin with a hardener. The PU nanofiber mats having a thickness of 30 µm were deposited by lay-up on the impregnated surface. Unreinforced and reinforced fiberglass composite was cured at room temperature for 24 h at the pressure of 14.2 psi using vacuum bagging method; then were post-cured at 80 °C for 3 h, as prescribed by the supplier. However, unreinforced and reinforced carbon fiber composite was cured at room temperature for 24 h at the pressure of 14.2 psi using the vacuum bagging method and no post-cure was performed, as prescribed by the supplier.

2.3 Characterization of composite

The surface and cross-section of the composite were imaged using a Nikon Eclipse L150 optic microscope (OM) and field emission scanning electron microscopy (FESEM) with an energy dispersive detector (EDS). The tensile and flexural test was realized using Shimadzu AG-IS 250 kN. The tensile test was realized according to the standard ASTM D3039 at a speed rate of 2 mm/min while the three-point-bending flexural test was realized according to ASTM D790 at a speed rate of 2 mm/min Figure 3. At least 5 tests were run, and their average was taken to be used in data.

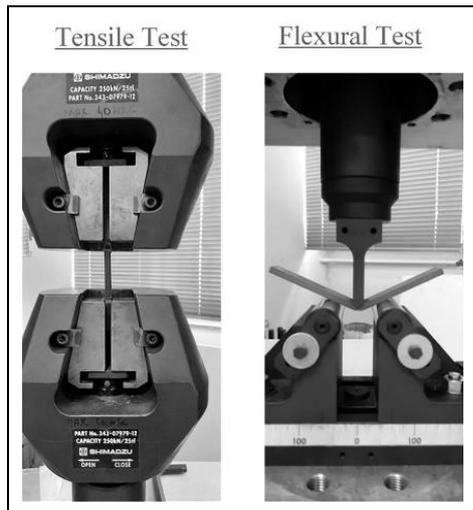


Figure 3. Image of during tensile and flexural test.

3 Results

3.1 Morphological and phase characterization

The used PU nanofiber mat was randomly distributed with high porosity and had an average diameter of ~450 nm with some beads Figure 2. Specifically, it was observed in the literature that the higher porosity and surface area of nanofibers allow more resin absorption which increases the strength of the composite [18],[25]. The morphology of the surface, the matrix, and the cross-section of the composite were performed using OM and FESEM-EDS Figure 4-6. The analysis of the surface of unreinforced fiberglass showed aligned and coherent fiber with an average diameter of ~290 µm. Moreover, the surface of the fiber was composed of various craters, probably due to the manufacturing process of fiberglass Figure 4(a). The cross-section of sepiolite powders doped in the matrix was presented

in Figure 4(b). There were sepiolite powders (yellow arrow), especially around perpendicular and diagonal fibers in the matrix.

Regarding nanopowder SiO₂-doped composite, the OM exhibited that some agglomeration at a different location was observed in the matrix (yellow arrow) Figure 4(c). These coalescences can affect the mechanical properties of composites due to the formation of stress at these locations. On the other hand, the surface of unreinforced carbon fibers had a diameter of ~ 125-200 µm with some carbon dots Figure 4(d).

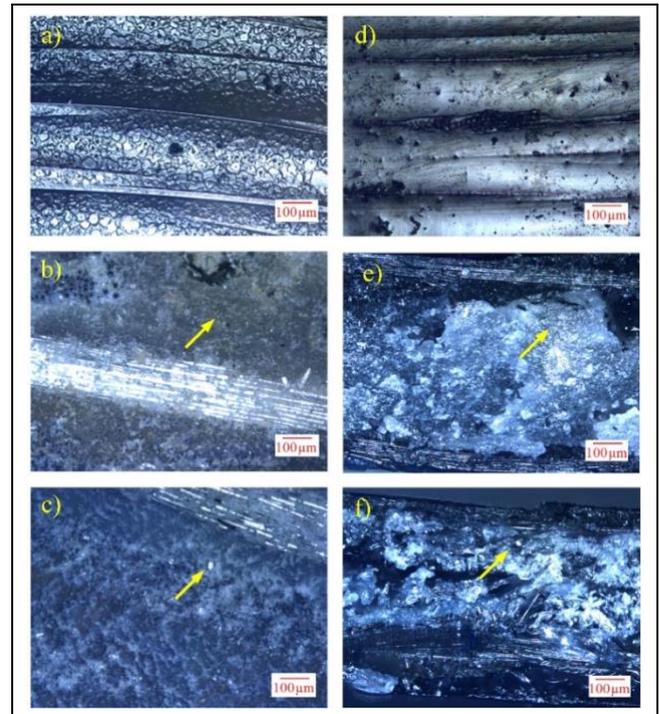


Figure 4. Optic microscope images of unreinforced and reinforced FG and CF composites (100x); a) surface of fiberglass plain. (b): Cross-section of sepiolite reinforced fiberglass composite. (c): cross-section of SiO₂ reinforced fiberglass composite. (d): the surface of carbon fiber plain. (e): Cross-section of sepiolite reinforced carbon fiber composite. (f): Cross-section of SiO₂ doped carbon fiber composite.

The fragmentation of sepiolite powder and SiO₂ nanoparticles that were in the matrix of carbon fiber was similar to fiberglass composite Figure (4e-4f). FESEM-EDS analysis also confirmed the presence of nanofibers and powders in the matrix Figure 5 and Figure 6. As expected, C, O, and N elements were found in the matrix of unreinforced carbon fiber composite. The cross-section view of the composite shows that the resin and fibers were well-compromised Figure (5a). Eventually, the diameter of nanofibers reached from ~450 nm to ~1500 nm Figure 5(b). Here, it can be stated that the nanofibers can play a good adhesion bridge between fibers and resin. On the other hand, sepiolite powder is composed of magnesium silicate (Mg₄Si₆O₁₅(OH)₂·6H₂O), and the high surface area of this clay makes it an excellent absorbent of H₂O [17]. When looking at the chemical composition of the resin-filled with sepiolite, the Mg and Si elements were detected with 1.79 wt.% and 2.23 wt.%, respectively Figure 6(a). Otherwise, the composite with doped nanopowder SiO₂ had 0.66 wt.% of Si, meaning a fair distribution of nanopowders Figure 6(b). The findings on the

cross-section analysis of fiberglass composite were similar to that of the carbon fiber composite.

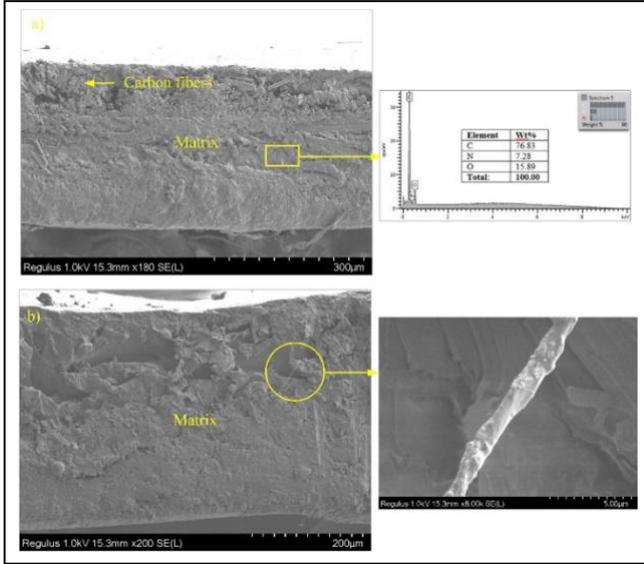


Figure 5. FE-SEM analysis of carbon fiber composite (2500x). (a): Cross-section of reinforced CF with PU nanofibers with EDS analysis of matrix. (b): Cross-section of CF composite showing PU nanofiber in the matrix.

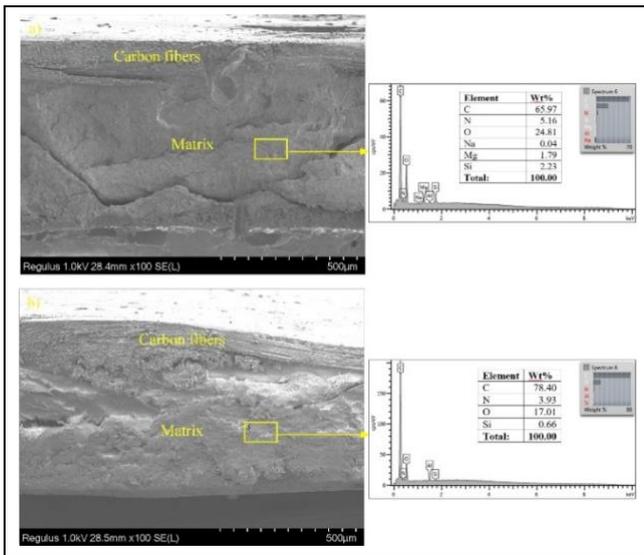


Figure 6. FE-SEM analysis of carbon fiber composite (2500x). (a): Cross-section of reinforced CF with sepiolite powder with EDS analysis of matrix. (b): Cross-section of reinforced CF with SiO₂ powder with EDS analysis of matrix.

3.2 Mechanical analysis of composite

Tensile and flexural tests were performed to determine the maximum load that the composite materials can support. The unreinforced FG composite registered maximum strength of 72 MPa with an elastic modulus of 0.09 GPa Figure 7. The maximum strength was calculated at the breakage. The maximum strength value of composite was calculated at the breakage of the composite while elastic modulus was determined from the linear region by taking the slope. Adding PU nanofiber mat in the resin roughly doubled the strength of composite against failure with 135 MPa. It was indicated that the main reason for this increase is due to the higher porosity

of nanofibers solidly bonding with the matrix [18]. The result found was ~ 5 times lower than the work realized by De Schoemaker et al. [8] due to deposited PA6 nanofibers versus interlayered PA6 nanofibers. On the other hand, the maximum strength of powder-filled composites was below that of FG-Nanofiber but higher than that of unreinforced composite Figure 7. Moreover, the stiffness of the micro and nano-filled composites was higher than the unreinforced composite. It should be noted here that adding powders or nanofibers in the resin effectively helped to enhance the strength of the fiberglass composite, and it was more pronounced for nanofiber doped composites.

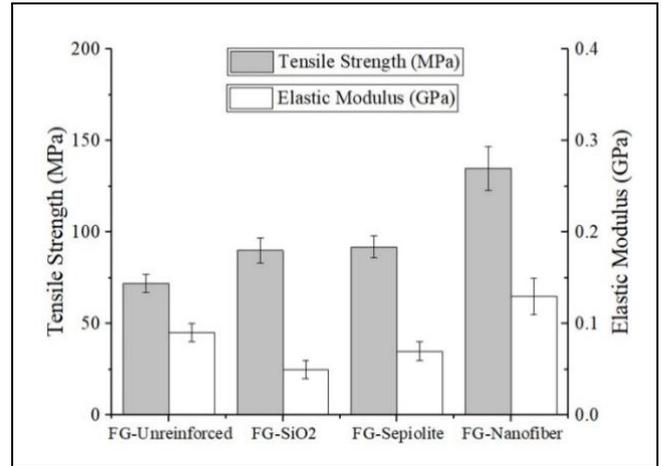


Figure 7. Data extracted from stress-strain curves of unreinforced and reinforced FG composites.

When looking at the strength of CF composites, the addition of powders and nanofibers was beneficial to increase the strength of composites (Figure 8). The maximum strength was obtained with nanofiber-doped composite followed by CF-SiO₂ and CF-Sepiolite. It was evident that PU nanofibers did strengthen the carbon fiber/epoxy laminate with 134 MPa Figure 8. This enhancement is approximately double than of the study by Zhou et al [16] because, as explained above, nanofibers, due to their high volume-area ratio, form a higher adhesion with the resin. The nanofibers have encapsulated the resin due to the high volume area ratio permitting higher adhesion.

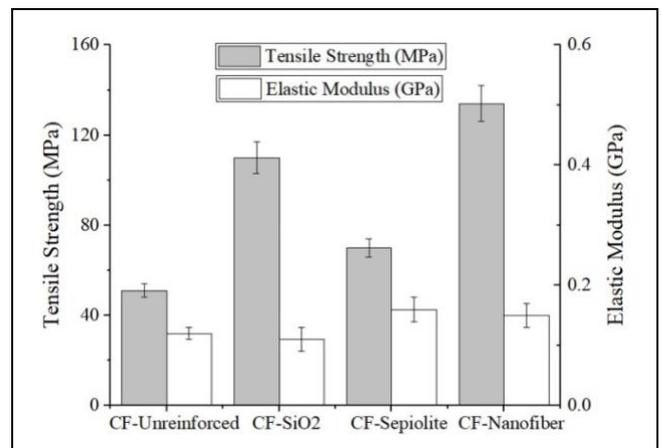


Figure 8. Data extracted from stress-strain curves of unreinforced and reinforced CF composites.

In the case of the study by Zhou et al., [16] carbon nanofibers were agglomerated into each other and did not supporting the

load applied. However, the tensile strength value of the CF-Nanofiber composite was no higher than the nanofiber FG-Nanofiber composite. This is due presumably to the high density of fiberglass plain compared to carbon fiber plain affecting the mechanical properties. Moreover, the carbon fiber composite was rigid compared to fiberglass composite. The weak strength performance of the CF-Sepiolite composite was observed compared to the rival composite.

During the flexural test, breakage of filament and inter-layer delamination was observed, and immediately the composites failed after they arrived at maximum stress. Figure 9 reveals that by impregnating nanofibers in the matrix with epoxy FG composite, flexural strength was significantly enhanced, and it reached 197 MPa. Furthermore, the rigidity of the composite increased compared to that of the rivals. This improvement was explained by [35] citing that the bridging effect plays a significant role to crack propagation. Besides, the dense matrix and high surface area created by nanofibers allow more adherence [22]. Nonetheless, Liu et al. [19] showed that increasing the thickness of laminates using thermoplastic TPU nanofibers (higher than 0.6 mm) decreases flexural strength. They obtained flexural strength of 676 MPa for no-filler laminate, whereas ~709 MPa was recorded for 0.2 μm thickness of TPU nanofiber-doped laminate. The higher flexural strength found in their study compared to our findings was because they used more plies. The worst flexural stress was obtained with the addition of sepiolite powder. The main reason for the decrease of strength can be explained by the lower stiffness of sepiolite powder, causing a softer matrix. Otherwise, the SiO_2 nanoparticle behavior as described in the morphology analysis was due to nanoparticle agglomeration causing stress accumulation and ending with a reduction in strength and rigidity. It can be summarized here that the powders did not strengthen the composite, but the nanofibers were beneficial for FG composite.

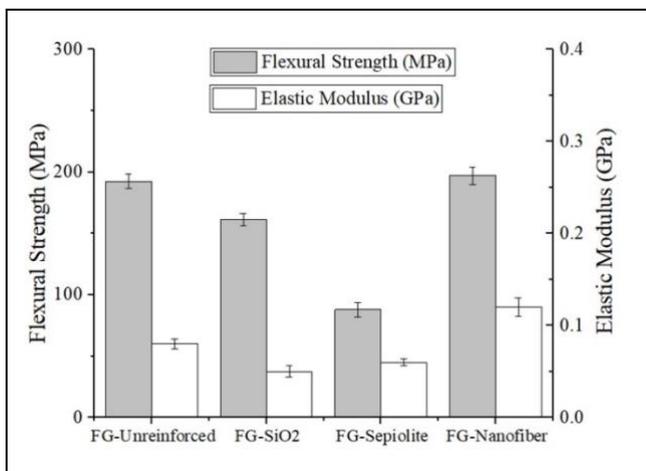


Figure 9. Data extracted from flexural stress curves of unreinforced and reinforced FG composites.

The CF composite sample with nanofibers electrospun performed better flexural stress and rigidity compared to its alternative Figure 10. On the other hand, the flexural stress of CF-Nanofiber was 356 MPa higher than FG-Nanofiber. Regarding the effect of powders, they exhibited lower flexural stress against unreinforced composite such as FG composite. The addition of SiO_2 and Sepiolite powders decreased strength to 384 MPa and 203 MPa, respectively. The lower strength can be associated with the nanoparticle gathering which probably

accumulates more stress. Moreover, nanoparticles may create a rougher surface interlayer with lower bonding which provokes dissociation from the matrix during loading. It should be noted here that PU nanofiber filler outperformed during flexural test compared to powders fillers such as tensile test.

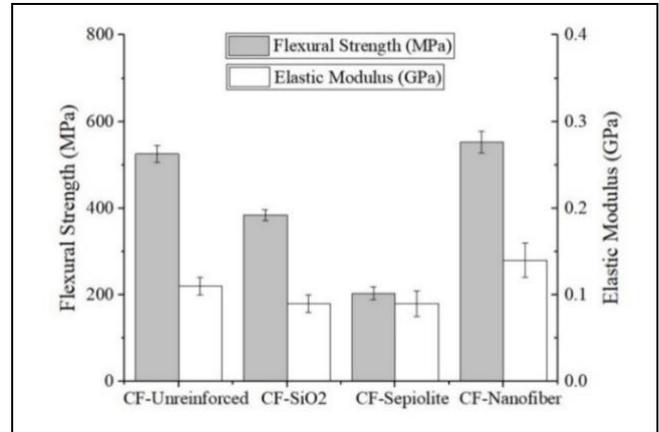


Figure 10. Data extracted from flexural stress curves of unreinforced and reinforced CF composites.

4 Conclusions

In this work, mechanical tests were conducted on the FG and carbon fiber composites reinforced with PU nanofibers and powders. The following findings can be drawn:

-Morphological analysis confirmed that nanofibers were impregnated in the matrix and played a strong barrier against fracture, while SiO_2 and sepiolite powder were found agglomerated in the matrix.

- The PU nanofiber mat filler increased at least 2 times the tensile strength and at least 10% of the rigidity of FG and CF composite compared to unreinforced composite due to higher porosity and surface area of nanofibers allowing more bonding with resin. On the other hand, SiO_2 and sepiolite powders enhanced at least 15% the tensile strength compared to unreinforced composite but it was at least 30% lesser than PU nanofiber mat,
- Regarding flexural strength, nanofiber mat filler was at least 5% better while SiO_2 and sepiolite powders exhibited at least 10% lower flexural strength compared to unreinforced composite. The better performance of nanofiber mat filler against the applied loads can be explained by the dense matrix and surface structure,
- The study also showed that one plie of 30 μm of PU nanofibers mat filler can be used in FG and CF composite to increase the mechanical properties for manufacturing high-performance parts. On the other hand, the addition of 10 wt.% of powder in the matrix exhibited better tensile strength behavior in adverse decreased the flexural strength for FG and CF composite.

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6 Author contribution statement

In this work, the Mustafa Özgür ÖTEYAKA contributed by his idea, designing and providing the materials and literature review; the Kerem AYBAR worked on the evaluation of the results obtained, literature review; the last Hasan Candan

ÖTEYAKA contributed by spell check, evaluation of results, and literature review.

7 Ethics committee approval and conflict of interest statement

The article does not require permission from the ethics committee and there is no conflict of interest with any person/institution.

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