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The impact of seawater aging on basalt/graphene nanoplatelet-epoxy composites: performance evaluating by Dynamic Mechanical Analysis (DMA) and short beam shear (sbs) tests

Deniz suyu yaşlandırmasının bazalt/grafen nanolevha-epoksi kompozitler üzerindeki etkisi: Dinamik Mekanik Analiz (DMA) ve kısa kiriş kayma testleri ile performans değerlendirmesi

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Abstract

Seawater exposure has adverse effects on fiber-reinforced polymer (FRP) composites' mechanical performance and service life because polymer matrix degradation caused by absorbed water can initiate FRP composites' irreversible damage. So, composites' performance under service conditions must be comprehensively evaluated and improved for safe usage in marine structures. For this purpose, the addition of nanoparticles into the polymer matrix can be an effective strategy to reinforce the polymer matrix against the seawater environment's detrimental effects. This paper presents the impact of graphene nanoplatelet (GNP) on basalt/epoxy composites' mechanical performance under long-term seawater aging. Experimental results of short beam shear tests revealed that the addition of 0.5 wt% GNP enhanced interlaminar shear strength by approximately 24 and 14% compared with the neat basalt/epoxy composites for seawater aged and unaged specimens, respectively. The dynamic mechanical analysis has also deduced that the multi-scale composite's transition temperature increased up to 5.7% compared to the neat basalt/epoxy composites.

Keywords: *Basalt fiber, Epoxy, Graphene nanoplate, Dynamic mechanical analysis, Short beam shear test*

Özet

Deniz suyu maruziyeti, fiber takviyeli polimer (FTP) kompozitlerin mekanik performansı ve hizmet ömrü üzerinde olumsuz etkilere sahiptir. Absorbe edilen su polimer matris bozulmasına yol açarak FTP kompozitlerin geri dönüşümsüz hasarını başlatabilmektedir. Bu sebeple, FTP kompozitlerin deniz yapılarında güvenle kullanımı için, servis koşullarındaki performanslarının kapsamlı bir şekilde anlaşılması ve iyileştirilmesi gerekmektedir. Bu amaç doğrultusunda, nanoparçacıkların polimer matris içerisine eklenmesi, deniz suyu ortamının zararlı etkilerine karşı polimer matrisini güçlendirmek için etkili bir stratejidir. Bu makale, grafen nanolevha (GNL) takviyesinin bazalt/epoksi kompozitlerin uzun süreli deniz suyu yaşlanması altında mekanik performansı üzerindeki etkisini sunmaktadır. Kısa kiriş kayma test sonuçları, deniz suyunda yaşlandırılmış ve yaşlandırılmamış numuneler için saf bazalt/epoksi kompozitlere kıyasla ağırlıkça %0.5 GNL eklenmesinin tabakalar-arası arası kayma mukavemetini sırasıyla yaklaşık %24 ve %14 artırdığını ortaya koymuştur. Dinamik mekanik analizlerde, çok-ölçekli kompozitlerin geçiş sıcaklığının, saf bazalt/epoksi kompozitlere kıyasla %5.7'ye kadar yükseldiğini ortaya çıkarmıştır.

Keywords: Bazalt fiber, Epoksi, Grafen nanolevha, Dynamik mekanik test, Kısa kiriş kayma testi

1 Introduction

Fiber-reinforced polymers (FRPs) are widely utilized in aviation, automotive, and naval application thanks to their favorable characteristics (including high performance, lightweight, and reduced lifecycle costs) for engineering [1-7]. FRPs may lose their performance during service due to the harsh environmental effects (temperature, humidity, water, etc.). Especially absorbed water by the composite structure during water exposure of FRPs diffuses through the matrix-fiber interfacial region. It deteriorates the interfacial bonding performance via pitting, hydroxylation hydrolysis, plasticization leaching [8-11]. So, providing the enhancement corrosion resistance is a crucial issue for the durability of FRPs in the seawater environment.

On the other hand, composite structures' corrosion resistance is mainly related to the matrix's water absorption resistance property that holds the primary load-bearing reinforcement fibers together. The corrosion effect on the composite structures is related to the corrosive medium's absorbability by the matrix. Although epoxy resins are preferred in engineering applications with their relatively good corrosion resistance and high mechanical performance properties, epoxy's functional properties are intensely influenced by the humid environment in-service conditions. Further, the environmental effects cause the degradation of epoxy and consequent deterioration of its stability. This effect generates a substantial disadvantage in many engineering structures [12].

When the matrix or fiber-matrix interface is damaged by water aging, there is the possibility of decreasing ILSS and thermomechanical performances of the composites [13, 14]. The reason for this is that ILSS is the measure of fiber-matrix interface bonding strength. Similarly, the dynamic mechanical analysis (DMA) technique also enables us to evaluate the fiber-matrix interfacial adhesion or interfacial degradation [15].

Matrix modification with adding nanoparticles enables to enhance interface bond between polymer matrix and fibers. Additionally, adding nanoparticles in a polymer matrix provides another significant advantage: a substantial

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decrease of permeability due to nanoparticles' tortuous path with a high aspect ratio. On the other hand, matrix modification by adding rigid nanoparticles improves the stiffness and thermal glass transition temperature (Tg) of the composites [16]. Therefore, matrix modification is an effective method to ameliorate the mechanical, dynamic mechanical, and barrier performances of FRPs [17]. Graphene nanoplatelets (GNPs) have excellent potential performance/cost balance, and it attracts researchers' interest in creating enhanced hybrid composites due to their excellent mechanical properties [18, 19]. GNPs have a high aspect ratio and surface area, improving the load transfer capability [20].

Expanding awareness of eco-friendly materials has recently induced investigators to use an alternate natural fiber to replace inorganic fibers in recent years [19]. In this regard, basalt fibers (BFs) extruded from the basalt rock are preferred for engineering applications as natural mineral fiber. BFs have exceptional characteristic properties at least 16% higher elastic modulus, excellent alkaline durability, good chemical resistance, and low-cost relative to E-glass fiber [21].

Potential behaviors of basalt-FRPs in the marine application have previously been discussed in the open literature [22]. A few researchers have studied the endurance of basalt-FRPs under a seawater environment [23]. Additionally, there is still no research study evaluating the seawater-aging performance of basalt-FRPs after modifying the matrix with GNPs. However, the matrix modification's advantages to the composite structure are open to discussion. The investigation of the reliability and durability of GNP modified basalt-FRPs in a seawater environment is necessary. This study aims to evaluate the durability of seawater immersed basalt/GNP-epoxy multi-scale composites by SBS and DMA techniques. The current investigation reports the influence of GNP and seawater aging on the endurance of the basalt/epoxy multi-scale composites.

2 Materials and methods

2.1 Materials

The laminated multi-scale composites were prepared using basalt fiber woven fabric (Tila Kompozit Co., Turkey), MGS L160/H160 epoxy system (Dost Kimya Co., Turkey), and GNP nanoreinforcements (Nanografi Co. Ltd., Turkey). The technical specifications of the materials by the supplier company are as follows. The fabric has 300 g/m^2 areal density and 0.45 mm average thickness. GNPs have about 6 nm thickness, 5 µm diameter, and 150 m^2/g specific surface area.

2.2 Methods

To perform epoxy matrix modification, the 0.1, 0.3, 0.5, and 0.7 %wt GNPs were first dispersed via probe ultrasonicator in acetone (for 10 min at 20 kHz) and then added into the epoxy resin (for 10 min at 20 kHz). After evaporation of acetone in a vacuum oven, the curing agent was added (accordingly manufacturer's recommendations) and stirred (mechanically for 5 min). The basalt/epoxy laminates were manufactured as two steps to minimize the filtering effect of nanoparticles. Firstly, prepared epoxy resin mixtures were applied on twelve pieces of basalt woven fabrics using a roller. These resin-impregnated layers were stacked and sealed with a vacuum bag to remove the excessive amount of resin. The pressure was maintained at 0.8 bar assisted with a vacuum pump. Finally, the composite laminates were cured at 60 °C for 1 h and then post-cured at 120 °C for 4 h. [24]. The seawater submersion of the test coupons was conducted at room temperature, which was included the 6 wt% sea salt (about twice the average concentration in an ocean) [11, 17, 22]. This artificial seawater mixture was stirred with a mixer cyclically to achieve uniform seawater. The samples were withdrawn from the seawater after 1-year of immersion time to evaluate the long-term seawater aging effect on ILSS and DMA properties.

SBS test method was preferred to determine the ILSS of multi-scale composites due to its simplicity and feasibility. In this method, the test specimen subjected to three-point bending loading undertakes tension, compression, and shear stress. However, a dominant shear failure occurs in the SBS test with the lessened span-to-thickness ratios. In this study, SBS tests were applied to prismatic samples with 65 × 11 × 4 mm^3 following ASTM D2344 recommendations. The tests were conducted using a Shimadzu AGS-X10 at a 1 mm/min rate as five repetitive.

Dynamic mechanical analyses (DMA) were performed using a Perkin Elmer DMA 8000 analyzer to detect GNP's impact on dry and seawater-aged composites' dynamic mechanical performances. DMA tests have been performed following ASTMD 4065 standards. Specimens in 52 × 13 × 4 mm^3 dimensions were used for DMA tests, and a span to thickness ratio was arranged 12:1. The laminates have been subjected to three-point bending loading at 1 Hz constant frequency (under sinusoidal loading condition). The analysis carried out ranges from 0-200 °C (heating rate: 5 °C/min).

Raman spectroscopy was recorded with the WITEC Alpha 300 Confocal Raman system (4 cm^{-1} resolution at 3800-200 cm^{-1} scanning range) with 532 nm laser irradiation to reveal seawater's effect aging on the composite structure.

3 Results and discussions

SBS tests were performed to evaluate the interfacial properties of epoxy/basalt composites. Figure 1 depicts the ILSS properties for the dry and seawater aged composites depending on GNPs content. Generally, the obtained shear stress of the seawater aged specimens is lower than the dry composites. Additionally, the strong influence of the GNPs modification on the ILSS of the composites is explicit.

The ILSS of the neat sample is about 36 MPa before seawater immersion [25]. With the 0.5 wt% GNPs incorporation in the epoxy matrix, the ILSS value reached 44.8 MPa, which is about 24% higher than that of neat composite samples. GNPs and the epoxide groups create a cross-linking reaction, which interlocks structure in the GNP-epoxy through the covalent bond, resulting in reduced polymer chain mobility [26]. This interaction may have enabled an effective stress transfer, resulting in increased

ILSS for GNP modified composites (up to 0.5 wt%). However, a slight decrease in ILSS was observed for 0.7 wt% GNP modified specimens. This drop might be attributed to the agglomeration formation for high ratio GNPs included composites, because agglomerates can play a role in stress forming and increase the free volume by creating voids in the epoxy [24, 27]. The blunt force drop noticed in the neat epoxy specimen is thought to develop due to severe delaminations caused by weak interface strength. However, after seawater aging, the ILSS values are calculated as 10.6 and 13.6 MPa for the neat and multi-scale composite samples, respectively. Compared to those of unaged composites, there is a drastic decrease in the ILSS of all seawater aged specimens reaching about 70%. However, the GNPs modification aid in increasing the ILSS performance of the basalt/epoxy composites. The small concentrated nanoparticles in the matrix may not behave as an active barrier against water diffusion during the long-term immersion period.

Additionally, it is seen that in literature, the mechanical performance of epoxy is enhanced by GNP introducing in the range of 0-1 wt% [28-30], and also the rate of 2-5 wt% GNP preferred to provide effective barrier properties [31-33]. As a result, it is believed that the multi-scale composites modified with a low percentage of nanoparticles may absorb water molecules almost at the same rate after a long-term immersion period. Therefore, the same percentage of water absorption is seen as a possible result that deteriorates the structure to the same extent.

Figure 2 presents the different failure modes of the SBS fractured specimens. Before seawater aging, neat epoxy composites (Fig. 2(a)) compared with GNP modified epoxy composites (Fig. 2(b-e)), neat specimens failed with predominantly delamination failure modes. It is seen that the fiber breakage occurs more dominantly than delamination for the 0.5 wt% GNP modified samples. This situation can be attributed to the fact that nanoparticle modification enhances interlayer interactions and fiber-matrix interface interactions [10, 11].

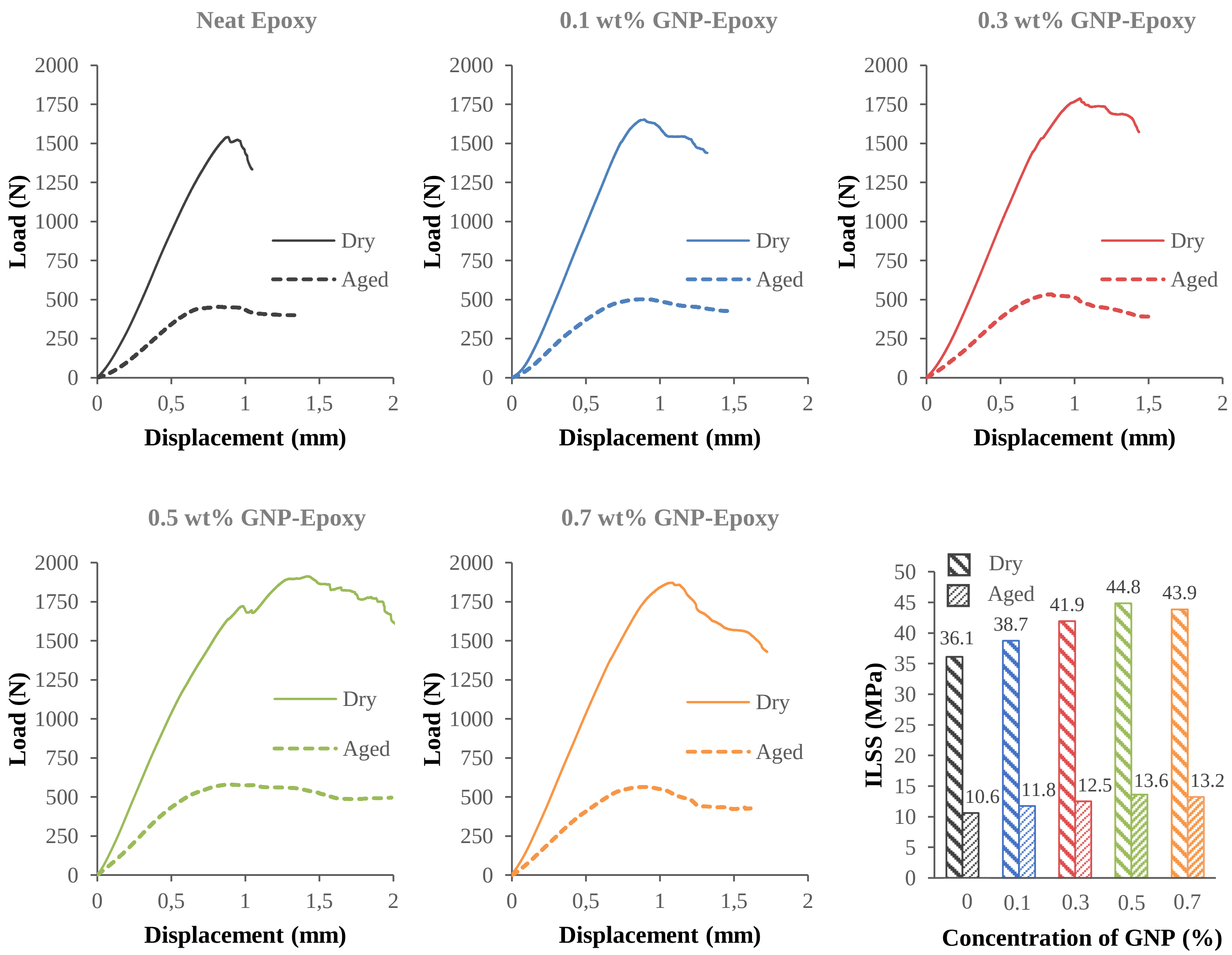

Figure 1. SBS test results of seawater aged and unaged samples containing different GNP ratio

The influence of aging on the damage behavior during the short beam shear tests are shown in Figure 2(g-j). After seawater aging, the composites' damage mode alters from a brittle to a more plastic failure mode. The reason for alter is thought as the water absorption increased the segmental chain mobility of epoxy and resulted in a plasticizing effect. This effect can be confirmed with all specimens' decreasing slope of the load-displacement curves after the seawater aging. Consequently, it is understood that the seawater acts as a plasticizer, the interfacial bonds were weakened, thus softening of the composite samples occurs

3.1 DMA tests

Figure 3 displays the experimental results of the dynamic mechanical analysis that depend on the GNP concentration and the effect of seawater aging. The DMA data are significantly influenced by GNPs concentration and seawater aging. The changing of storage modulus with temperature for various GNPs concentration is exhibited in Figure 3(a-b). It can be seen that specimens show higher storage modulus with increasing up to 0.5 wt% GNPs content for dry and aged samples. The increase is explicit in the glassy region below transition temperature (Tg), while there is not much influence in the rubbery plateau region. In the glassy state, composite structures' components are in a frozen situation (due to the close and tight packing), i.e., highly motionless. Therefore, since the samples are more rigid in the glassy area, it exhibits the highest modulus.

On the other hand, as the temperature rises, the components gain more mobility and lose their close-packing ordonnance. As a result, this rising temperature causes a decrease in the module. The modules drop to almost the same level at elevated temperatures, as the materials significantly lose their rigidity. Hence, there is no noteworthy difference in the modulus due to high temperatures in the rubbery region (seen in Figure 3.a-b) [34].

The tan delta-temperature curves in Figure 3(c-d) shows the influence of GNP concentration on damping features of the aged and unaged composites. The peak of the curve is referred to as the Tg. The figure shows that the viscoelastic property is enhanced significantly by matrix modification, and it decreases by seawater immersion. The Tg increase noticed in the GNPs modified samples can be referred to as a chain-pinning mechanism encouraged by the GNP-epoxy interactive relation. Additionally, GNPs adding to the epoxy matrix could decrease and limit polymer chain mobility and thus improve the limitation on the rate of relaxation, leading to more superior Tg. Lower tan δ (delta) observed in case of GNPs addition up to only 0.5 wt% can be attributed not only to the toughening impact and ameliorated interfacial bonding but also to well epoxy-GNP interactions, resulting in dynamic mechanical properties [35]. The observed higher dynamic mechanical performances due to matrix modification for seawater aged composites better describes polymer chain mobility restriction.

Overall, Tg changes as a function of GNPs concentration for dry and aged specimens are shown as a bar graph in Figure 3(e-f). For GNPs modified and neat epoxy composites, after 1-year of seawater aging, Tg in all conditions decreased. However, at a 0.5 wt% GNP configuration, Tg reduction was slightly lower than that of other GNP configurations. The Tg value of the neat epoxy composites exhibited a tan delta maxima peak at 106.3 and 103.5 °C for dry and seawater-aged composites. Similarly, the Tg values of the 0.5 wt% GNP modified composite were measured as 110.4 and 108.3 °C for dry and seawater aging, respectively.

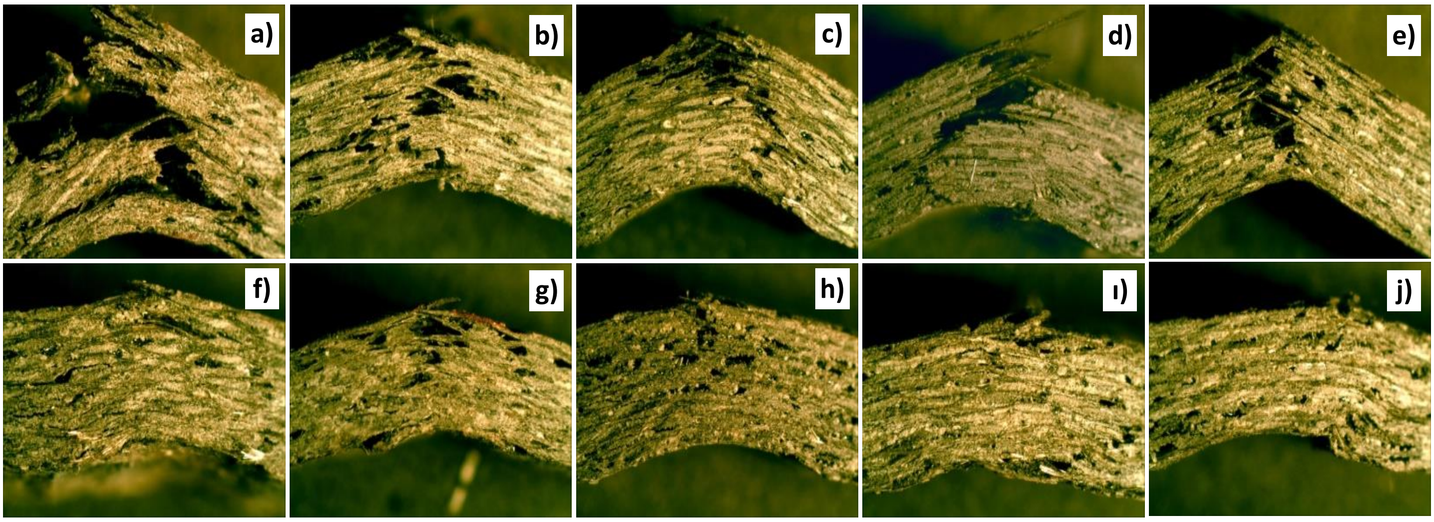

Figure 2. Optical microscope images of fracture surfaces after SBS tests for: (a-e) neat, 0.1, 0.3, 0.5, 0.7 wt% GNP modified unaged specimens, and (f-j) neat, 0.1, 0.3, 0.5, 0.7 wt% GNP modified seawater aged specimens, respectively.

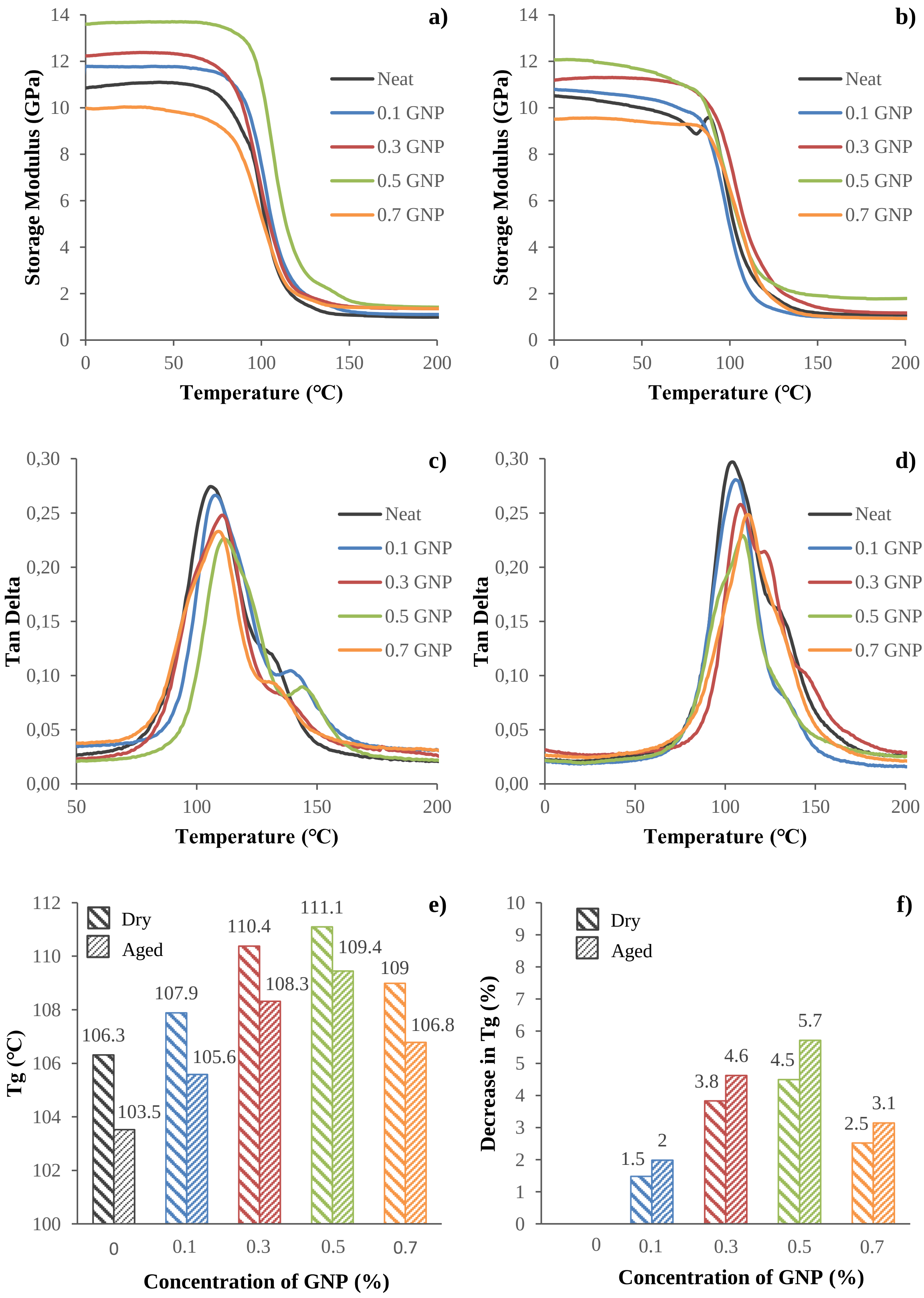

Figure 3. DMA properties of basalt/epoxy composites: (a) storage modulus as a function of temperature for dry samples, (b) storage modulus as a function of temperature for seawater-aged samples, (c) tan delta as a function of temperature for dry samples, (d) tan delta as a function of temperature for seawater-aged samples, (e) bar graph of Tg values as a function of GNP concentration, f) Decreasement of Tg values

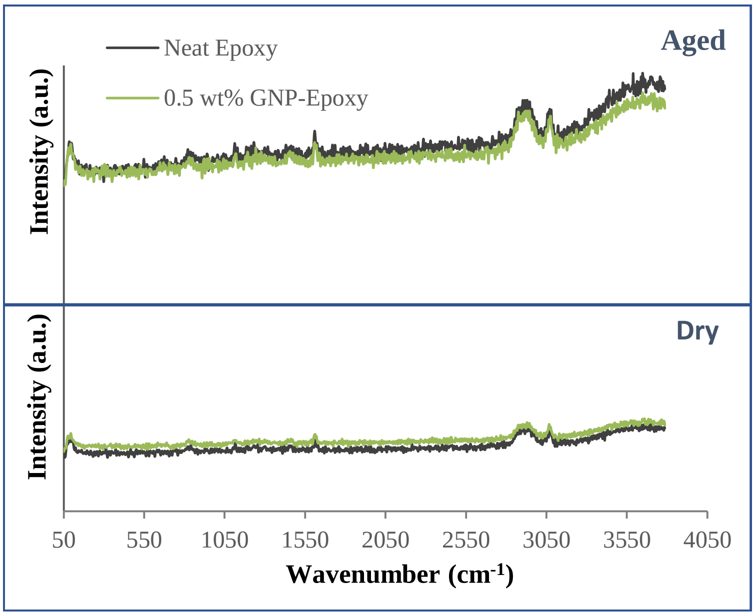

Figure 4. Raman spectra of dry and seawater aged basalt/epoxy composites

3.2 RAMAN analysis

Raman spectroscopy studies have been performed to confirm the impact of seawater aging on chemical interactions. It is seen in Figure 4 that the peak intensity of the hydroxyl groups (around 3000 cm^{-1}) of composites changed after seawater aging. This chancing peak intensity is associated with water content in the composites [11, 36]. Here, the increase of hydroxyl peaks due to water absorption for both composites are seen. However, it can be said that the addition of 0.5 wt% GNP has led to a small amount of peak intensity change depends on the barrier properties of GNPs after 1-year aging. Although it is known that the mechanical performance of epoxy in room conditions is improved by adding GNP in the range of 0-1 wt% [28-30], the rate of 2-5 wt% GNP adding is noteworthy in the literature to provide effective barrier properties [31-33].

4 Conclusions

Present research systematically deals with evaluating long-term seawater durability of basalt/epoxy composite modified with different levels of GNPs adding. The DMA results are in agreement with the SBS test results. According to the results of this work, the following observations can be drawn.

1. The presence of GNPs in the basalt/epoxy composites notably strengthens the interlaminar shear properties and Tg, which is reflected in the decreased tan delta maxima peak of DMA curves.

2. SBS test results show that incorporating GNP ranges from 0.1-0.5 wt% enhanced the ILSS of composites. However, at 0.7 wt% of GNP, there was not more enhancement of ILSS. The optical microscope investigations demonstrated that performed matrix modification by adding GNPs improve fiber-matrix interfacial bonding properties.

3. For GNPs reinforced multi-scale composites; the 0.1-0.5 wt% GNP included samples had the highest storage modulus because of the interlocking effect. Compared with the neat epoxy composite, the 0.5 wt% GNP included composites showed a much higher storage modulus because of enhanced fiber-matrix interface bonding.

4. It was observed that the intensity of the hydroxyl group peak of unaged composites relatively lower than the seawater-aged composites. On the other hand, the fact that the peak intensity was slightly lower in nanoparticle reinforced samples referred that GNPs addition relatively limited the amount of absorbed water.

In conclusion, this work has shown that 0.5 wt% GNP modified basalt/epoxy multi-scale composites exhibit significantly higher mechanical performance after long-term seawater immersion.

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Conflict of interest

The author declares that there is no conflict of interest.

Similarity rate (iThenticate): %7

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