Review Article

Aging Effects on the Mechanical Performance of Carbon Fiber-Reinforced Composites

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Carbon fiber-reinforced composite (CFRC) is a well-known hi-tech material with diverse applications. The CFRC faces several environmental conditions during its application, e.g., elevated temperatures, humidity, exposure to UV radiation, and acidic and alkaline environments. These environmental factors strongly affect the performance of CFRC, and they tend to age with time. Aging reduces the mechanical properties of the composite and ultimately its service life. In this review, the focus is on the aging of composite, common types of aging (thermal, hydrothermal acid and alkaline, and UV radiation), and the role of the third phase (fillers) in the aging process. There are numerous factors involved in the aging of composite. Aging starts with microcracks and proceeds towards delamination which further exposes the internal surface to environments. When the depths are exposed, free radicals are released and further deteriorate the internal structures. They create more pathways for oxygen to reach every millimeter of composite, thus reducing the mechanical performance of the composite. Usually, a trend is seen that introducing filler might slow down or compensate for the mechanical performance after aging. This trend is explored in the review article. However, usually, the third phase remained neutral and sometimes reduced and/or enhanced the mechanical properties after aging. In thermal aging, different metallic oxides have a noteworthy effect on mechanical performance. The synergistic effect of the third phase and aging on CFRC mechanical performance is also tabulated.

1. Introduction

Polymer composites have proved to be an ideal engineering material owing to their ability to be customized and tailored to perform under different environmental conditions. An additional advantage offered by carbon fiber-reinforced composites (CFRC) is their high strength-to-weight ratio as compared to other structural materials. Therefore, the CFRC is a preferred choice for various hi-tech applications in the aerospace and defense sectors. The CFRC used in these applications endures a variety of environmental conditions during its service life and leads to defect generation in the part. Some other defects can be induced in the form of matrix cracking, delamination, fading, thermal instability, moisture absorption, etc. [1, 2]. These impairments hinder the mechanical performance of the composite and may cause catastrophic failure of the part.

Composite materials are fabricated mainly from two components, i.e., matrix and reinforcement. Moreover, a third phase can be added according to the application. These constituents exhibit diverse chemical/mechanical/physical properties [3, 4]. The most well-known matrix used as a resin for hi-tech applications is polyester, vinyl ester, epoxy, phenol-formaldehyde (PF), polyimide (PI), polyamide, and polyether ether ketone (PEEK) [5, 6]. The popular fibers found to be perfect for hi-tech applications and compatible with the abovementioned resins are glass, carbon, aramid, and hybrid boron-woven fibers to acquire superior properties [7]. Modern composite materials comprise carbon fiber-reinforced epoxy composite. These are stiff, strong, and lightweight composites with exceptional mechanical properties. It has been used for aircraft [8] and rail components, civil structures [9], marine applications, automotive parts, and high-quality consumer products. Some typical applications of carbon/epoxy composites are as follows:
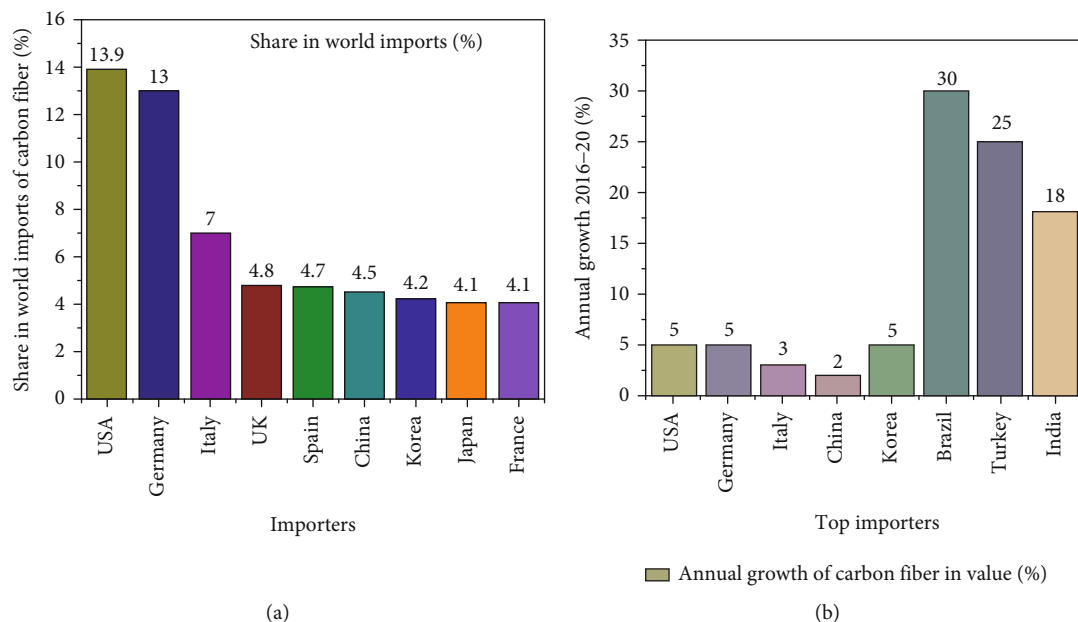
Figure 1: (a) Worldwide carbon fiber utilization and (b) annual growth.

(i) Flame retardant application [10]

(ii) Fuel cells [11]

(iii) Tissue engineering [12]

(iv) Biomedicine [13]

(v) Biomedical actuators [14]

(vi) Military applications [15]

(vii) Space applications [16]

(viii) Marine applications [17]

(ix) Shape memory composite [18]

(x) Racing car structures [19]

However, the diverse properties required for these applications are not obtained by using only carbon fiber and some resin. It is either hybridized with some other reinforcement material [20] or a third component is usually added to achieve the required properties. This third-phase material can be either chopped fiber, filler, or particulate [21–23]. The synergistic effects of all components in a composite help to achieve mechanical properties that cannot be possible by individual contributing material [24, 25]. The addition of rigid nanoparticles in a polymer matrix has proved to increase its in-plane shear properties significantly [26, 27].

Figure 1 shows the global data on imports and the annual growth of carbon fiber, respectively. It can be observed that the USA is the largest importer and thus produces a large number of products to meet the industrial demand of the world. Then next in line are Germany, Italy, Japan, and many other countries importing carbon fiber mainly for hi-tech applications. They majorly export high-performance products to other countries.

2. Aging of Carbon Fiber-Reinforced Composite (CFRC)

The composite has a wide range of applications where it is exposed to various environments. It can be under direct sunlight and affected by UV radiation or working undersea and exposed to salts and minerals, etc. The different environment leaves a different impact on the mechanical properties of the composite. To understand the degradation behavior and the extent of deterioration of mechanical properties, composite samples are characterized by various techniques to predict their performance. The most common aging categories are as follows:

(i) Thermooxidative aging

(ii) Hydrothermal aging

(iii) Aging due to acidic and alkaline environments

(iv) Aging by UV radiation

2.1. Thermooxidative Aging. Thermooxidative degradation or aging is the combination of two conditions, one is by the action of temperature, and the other is by the presence of oxygen. Thermooxidation degradation refers to the disintegration of macromolecules by the variation in temperature and the presence of oxygen. Free radicals are formed, and they react with oxygen to produce peroxy/oxy radicals. Thermooxidation is destructive for composite structures because it disturbs not only thermal properties but also affects the mechanical properties of the composite. The weight loss of the materials is amplified with extensive aging time. The prolonged aging makes the sample more prone to deteriorate and delaminate. Furthermore, longitudinal and transverse microcracks also become more prominent which provides more space for additional oxidation in the internal
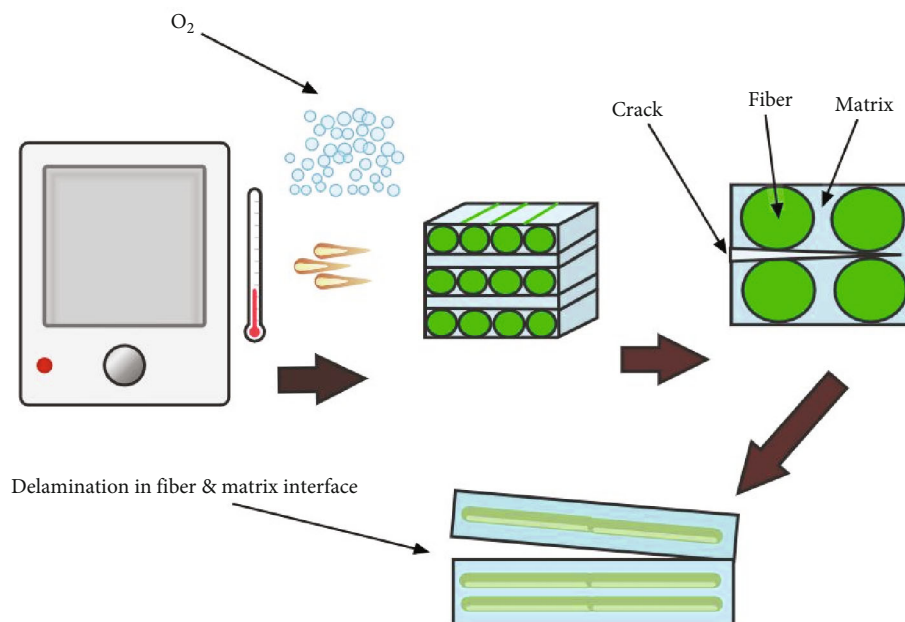
FIGURE 2: Delamination in composite under thermal aging.

areas. The appearance of microcracks between laminates is caused by prolonged exposure to thermooxidative aging. Figure 2 shows the schematic process of composite delamination.

The thermooxidative aging of composites is of two types: chemical and physical. Physical aging is referred to as reversible because the changes occur in the molecular conformation of the material without altering the structural configuration of molecules [28, 29]. The polymers, in response to thermal stresses, gradually evolute to thermodynamic equilibrium with changes in mechanical properties, volume, enthalpy, and entropy concerning time. It may be due to exposure of the composite to high temperatures below the Tg for a longer period [30]. The effect of a change in molecular conformation results in an increase in modulus, stress, density, and viscosity while reducing creep and stress relaxation [31, 32]. The carbon fiber and epoxy have different coefficients of thermal expansion (CTE); therefore, it intensifies thermal stress at the interface, making it more prone to microcracking [33, 34]. The intensification in microcrack density is caused by corrosion of the matrix after exposure to prolonged aging, which endorses delamination in the fiber/matrix interface [35, 36], as shown in Figure 2.

In the chemical aging process, the molecular structure collapses. It is instigated by mechanisms such as depolymerization, chain scission, oxidation, and changes in cross-link density. It is also known as irreversible change. Mostly, physical and chemical changes occur simultaneously. Moreover, elevated temperatures accelerate the degradation process [37, 38], but below 400°C, oxidation in carbon fiber is negligible [39, 40]. In the range of 150-300°C, the aging of pure epoxy begins, and it affects the mechanical properties of the composite [41]. The crack propagation or delamination of laminates is accelerated by thermal cycling in oxidative atmospheres [42, 43]. Elevated pressures of oxygen and air escalate the rate of thermooxidative degradation in polymer-based composite materials [44, 45]. It is observed that two parameters—elevated temperature w.r.t. time and exposure to oxygen w.r.t. time—play a pivotal role in the physical or chemical degradation of composite.

2.2. Hydrothermal Aging. The hydrothermal degradation of composite refers to the application where composite materials are in contact either with water, electrolyte, salt water, or water molecules present in the air. In the composite, epoxy resin is more prone to moisture attacks. However, it is made to have lower moisture absorption. The moisture absorption rate of epoxy depends on the free volume, type of epoxy, and concentration of polar groups present in it [46, 47].

In hydrothermal aging, the composite sample is emersed in water at a specific immersion temperature and time. Usually, it is in the range of 20°C to 80°C for 10-500 hours. However, the immersion temperature and duration can be changed. After that, the aged and unaged samples undergo characterization, and then the results are compared. Other methods are salt spray aging to evaluate the corrosion resistance of the composite or create an electrolytic environment [48]. It is evident from studies that sodium chloride itself does not harm the composite, and the osmotic pressure of the salt solution confines the absorption of moisture in the matrix. As compared to pure water, salt solution is less destructive. To ensure the safety of composite in marine applications, researchers need to fabricate water-resistant/hydrophobic coatings.

2.3. Aging in Acidic and Alkaline Environments. In the degradation process, chemical corrosion plays a vital role in lowering the mechanical strength of polymer composites. The reaction between chemicals and composite surfaces
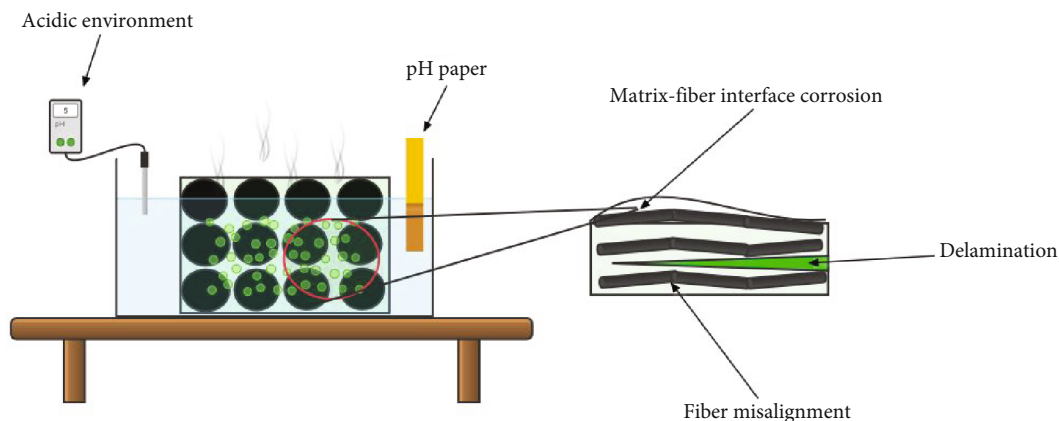
FIGURE 3: Aging in an acidic medium.

degrades the stiffness. Composites are being utilized in chemical industries for different purposes where they encounter other chemicals. It is a priority to fulfill the safety requirements and reliability of design and performance. Therefore, it is important to understand the behavior of the composite in an acidic environment to characterize the effect of eroding acid [49]. The surface of the polymer matrix is damaged by acid attacks, and it can generate a crack on the external surface. Then, the materials undergo mechanisms like flaking, and fiber is also damaged [50, 51].

The effect of acid on composite was observed by immersing it in an acidic environment in which a 10% NaOH solution and a 5% H_2SO_4 solution were prepared. In comparison, 10% NaOH solution gives better resilience than 5% H_2SO_4 solution, though H_2SO_4 solution initiates further degradation than NaOH solution. The researchers [52] premeditated the performance of the composite in a chemical environment. The composite immersion for 70-90 cycles in an acid environment showed declining properties, i.e., flexural strength. Moreover, a higher concentration of sulfuric acid causes further degradation in flexural strength. This is because acid deteriorates the matrix's chemical structure and reduces its strength by 5-10%.

The immersion of the sample concerning time and acid concentration creates the defects accordingly, as shown in Figure 3. For instance, if the sample is exposed to aggressive and corrosive acid fluids for the long term, then the extent of deterioration would be large in the form of blisters and swelling. The effect of degradation depends on the chemical structure and components of composites. The concentration of pH has a significant role in the degradation of composite laminate and durability. It has been observed that the strong acid like HCL present in a high amount reduces 16-18% of the interlaminar shear strength (ILSS) of composites. The researchers also observed that tensile strength increases at low pH levels and decreases at higher pH levels. This shows the stability of the composite in an acidic medium and its unstability in an alkaline medium. The solution with a pH > 7 in a higher concentration lowers the bond strength in composites.

It has been reported that stress corrosion in an alkaline environment decreases the strength of the fiber-reinforced polymer composites, which forms the calamitous failure of the composite. The most damaging chemicals are alkali and then acid if compared with salt, sulfate solution, and calcium solution [53]. The alkali can attack the composite surfaces by corroding the fiber diameter. It also caused delamination between the laminates. Hence, it causes fractures in composites. An acidic environment dehydrates the sample by diffusing the solution into a composite [9, 54]. The acid penetration is stimulated by the occurrence of microcracks on the surface of the composite present during manufacturing or due to working in harsh environments. The carbon fiber reinforcement slightly optimizes the mechanical properties and gets affected by moisture absorption or degradation of the composite. Afterward, debonding occurred, and cracks progressed at the fiber-matrix interphase [55, 56].

2.4. Aging by UV Radiation. Nowadays, composites are replacing conventional materials. Therefore, they are mostly used in different environmental conditions where they are exposed to direct sunlight radiation [57, 58]. There are numerous applications like marine fiber boats, aircraft structures, oil and chemical industries, wind turbine blades, and sports goods [59, 60]. In the mentioned applications, polymer composites are exposed to UV radiation for a longer period. Polymer composites are in progress towards special design fields, predominantly in aeronautic bundles [61, 62]. It includes funnel line-wearing sand slurries in petroleum refining, helicopter rotor slicing edges, severe rhythm autos and flying desktops working in fruitless field situations, and aircraft motors. Composite shows execrable disintegration resistance in comparison with metals. However, several researches show that polymers are vulnerable to UV radiation [63]. It was also seen that UV rays tend to lessen the working period of composites.

Moreover, it is very important to expect the lifetime durability of the material. On exposure to direct sunlight, the polymer composite reacts with photons of UV rays, and then the crosslinking of macromolecules gets affected, hence degrading mechanical and physical properties, as shown in Figure 4 [64]. These rays are destructive to the composite material and initiate the chain scission process
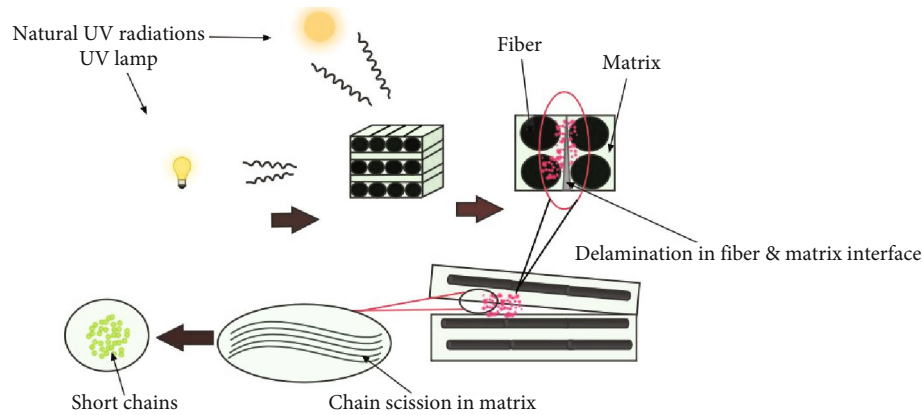
FIGURE 4: Chain scission in the matrix by UV radiation.

[65]. Then, chain scission at the polymer surface causes microcrack formation, diminishes color, and leads to covalent bond breakage, hence decreasing the weight of the composite structures [66]. The UV rays are responsible for creating free radicals on the polymer surface, and the rate of formation of free radicals depends on the exposure time of the composite to the UV rays, hence the loss in stiffness and strength [67].

The composite must maintain mechanical properties to provide the best service. If the composite mislays in its mechanical properties in the operational period, then disastrous failure arises, though the operating window is different and diverse for different polymers under UV radiation process conditions. It has been observed that mostly the rate of degradation depends on two things: the rate of change of chemical structures and the presence of peripheral chemicals in the composite [68]. It has been reported that the addition of metal to the matrix can protect the matrix from degrading under UV radiation. The aluminum powder addition to epoxy is to be coated on any surface as a protection layer against UV radiation [69].

There is another class of composite comprising natural reinforcements. Natural reinforcements are used in hybrid composite for a broad range of applications. However, the chances of failure are much greater than those of the composite with synthetic reinforcements. It is easy for UV rays to penetrate through natural reinforcement and induce high thermal oxidation [70]. The penetration of UV rays leads to chain scission and deteriorates the compatibility between the matrix and reinforcement, and eventually, the composite loses its strength. In natural reinforcements, the most common components that are responsible for degradation are hollow cellulose, hemicellulose, and lignin. Among these chemicals, a-cellulose content is responsible for maintaining the strength of natural fiber. When natural fiber loses a-cellulose, it also loses compatibility and strength with the polymer matrix. Subsequently, interfacial bonding is affected, and the matrix loses its mechanical properties and efficiency [71]. Nevertheless, the hybrid form of natural fiber with synthetic fibers in composites performs better because of the remarkable resistance of synthetic fibers against the UV radiation process [72].

The composite is tested against UV radiation. UV radiation is of three types: outdoor UV radiation, accelerated outdoor UV radiation, and accelerated laboratory UV radiation.

(i) In outdoor UV radiation, ASTM D 1435 [73] and ASTM G7 [74] standards are followed. The time duration mentioned in these standards is 10 days-180 days

(ii) In accelerated outdoor UV radiation testing, ASTM D 4364 [75] is followed, and a concentrated Fresnel focal point mirror is used to focus the sunlight UV rays on the surface of the sample

(iii) In the accelerated laboratory UV rays test, the ASTM G154 [76] standard is followed. In the dry ambiance, a fluorescent bulb emitting UV rays for 8 h at 60°C is used for the test. It is followed by a condensation cycle for 4 h at 50°C without UV rays

3. Effect of Aging on Mechanical Performance

Usually, the mechanical test is done after conditioning the sample as described above. The aged and unaged composites are compared to mention the changes that occurred. The most determined properties of the composite materials include tensile, flexural, impact, and shear. The interlaminar shear strength (ILSS) is a characteristic property that describes the delamination resistance of composite laminates. To characterize interlaminar shear strength, composite samples are placed in an oven for a few hours at low temperatures or elevated temperatures (depending on application). Then, samples are placed in a desiccator for at least 5 hours before the mechanical test.

The ILSS-BS EN 2563: 1997 is followed to perform ILSS tests and analysis. ILSS is determined by the following equation [77].

$$\tau = \frac{3.P_R}{4.bh},\tag{1}$$

where τ is the apparent interlaminar shear strength (MPa), P_R is the maximum load at the moment of first failure (N),

b is the width of the specimen (mm), and h is the thickness of the specimen (mm).

The other mechanical test methods are ASTM D3039, ASTM D790 [78], and ASTM D6110 [79] for the tensile test, flexural behavior, and Charpy's test, respectively, which are performed to characterize the composite further and determine its mechanical properties.

Normally, it has been seen that the unaged sample behaves according to the material's inherent property to bear the mechanical load. The unaged samples exhibit delamination but in a longitudinal axis along a mid-plane, while the aged samples undergo various defects and deformations in longitudinal and transverse directions. Accelerated aging causes crack propagation. However, several studies showed that strength varies in the scatter band and that compressive strength increased in aged samples as compared to unaged samples [80, 81]. The failure mode remains the same whether aged or unaged, and the fiber rupture remains in between the gauge lengths. Moreover, the interface can be affected by the aging process, but the fiber bulking in aged composites does not show declining behavior.

The reinforcement type plays a vital role in shear strength and flexural properties. For example, two composites were tested: one is tape reinforcement, and the second is fabric. The failure of tape laminate composite occurs more abruptly as compared to fabric laminate. The tape samples failed when the first tension was applied. However, in the fabric laminate case, the failure was gradual, and critical strength was proclaimed by laminate cracking. The structure of reinforcement has a direct impact on its mechanical properties.

Although aging causes delamination and other defects, sometimes the opposite action takes place. The increase in mechanical strength in ILSS testing of the aged samples is also possible. This happens due to the increase in residual stress during postcuring at elevated temperatures and moisture. The moisture acts as a plasticizer for the polymer matrix and encourages residual stress relaxation. The decrease in residual stress occurs during curing, and it reduces mechanical strength. The ILSS strength of the unaged carbon fiber-reinforced polymer composite was 90 MPa, and for the aged, it was 94 MPa. For aging, CFRC was exposed to UV radiation at 80°C and alternating cycles of water condensation at 50°C for 3 months. However, ILSS testing of aged and unaged CFRC has a different delamination pattern. Unaged composite cracks and delaminates longitudinally, while aged composite underwent multiple microcracks [47]. Moreover, a 3-point bending test also exposed that the crack density in the aged composite is higher than in unaged composites [53]. It is assisted by degradation in the interlaminar region and hence proceeds towards delamination [82].

In hydrothermal aging, the polarity of the matrix matters a lot. For example, in a carbon fiber-reinforced polycarbonate composite, polycarbonate interacts with water molecules and absorbs the water [83]. As evident from Figure 5, the moisture absorption is increasing linearly with a square root of 5 days of aging. After the 5th day, hygroscopic equilibrium is established which shows a flat line from day 7. It can be observed that the composite shows a saturation point after 5 days.
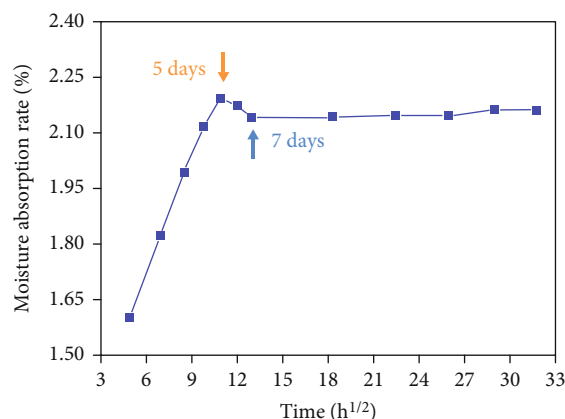

FIGURE 5: Water absorption of carbon fiber-reinforced polycarbonate composites [84].

From Figure 6(a), the unaged composite sample shows a compact and smooth surface. There are no voids or cracks that can be seen. However, in Figure 6(b), after 42 days, the aged specimen has developed deep cracks and voids. These cracks can be seen in red-circled areas. These cracks are a clear sign of water absorption in composite [84, 85]. These cracks are not only part of the surface but they are also developed in every space where water has penetrated. After evaporation, they can be seen as voids inside and on the surface. Moreover, the tensile properties of the aged sample decreased as compared to the unaged composite. The tensile strength of a composite sample aged in hot water at 80°C is measured with a frequency of every 7th day.

On initial days, the tensile strength increased; it is due to the tighter structure having entanglements of molecular chains. These entanglements start to loosen upon absorbing water, thereby decreasing the tensile strength. Further increases in aging caused more decline behavior in the mechanical strength of the composite, as shown in Figure 7. The polycarbonate reacts with water and absorbs it due to its strong polarity. The hydroxyl groups in water molecules tend to bind the chains of the polar polymer and eventually lead to chain breakage/hydrolysis. On the macroscopic level, overall mechanical performance decreases.

Figure 8 shows the flexural test results. The flexural performance of specimens increased and then declined with time, as shown in Figure 8(a)–8(c). Prolonged aging affects the mechanical performance of the composite. The bending test is done every 7th day to analyze changes, as shown in Figure 8(b). The starting flexural performance increased due to the hot temperature but then gradually decreased because of the water taken by the matrix [84, 86]. Figure 8(d) shows the flexural strain decreases as aging time increases.

4. Three Phase Carbon Fiber-Reinforced Composite

Composite fabrication is not limited to two distinguished entities, matrix and reinforcement, but it is extended to
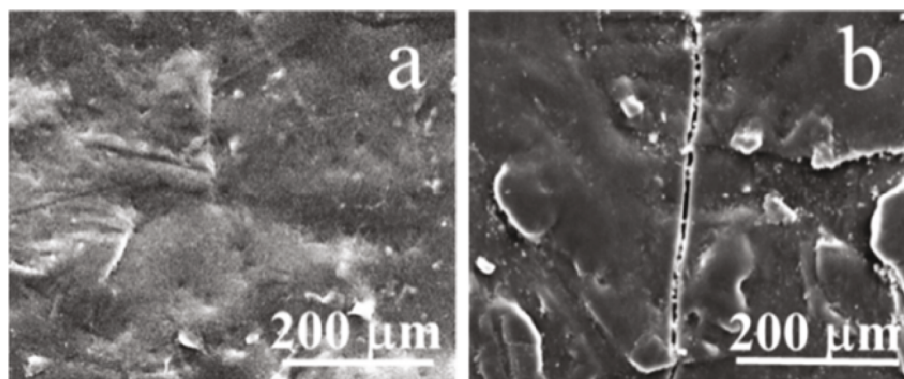
FIGURE 6: Surface topography of carbon fiber-reinforced polycarbonate composites. (a) The surface of unaged composites. (b) 42 days of hydrothermal aging at 80°C [84].
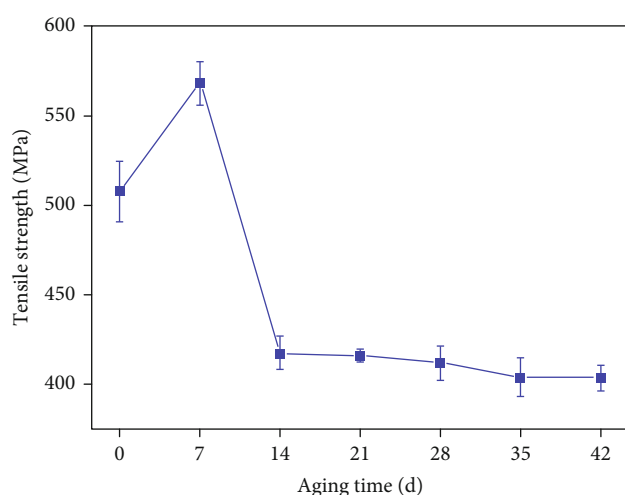

FIGURE 7: Decreasing tensile strength of carbon fiber-reinforced polycarbonate composite with increasing hydrothermal aging at 1 mm/min [84].

third-phase materials. Usually, the third phase is referred to as filler. The fillers have a purpose to serve; for example, they are introduced to enhance volume and mechanical performance, reduce flammability, impart electrical conductivity, reduce thermal coefficient, protect against UV rays, as pigments or dyes, etc. So, when fillers are used to provide protection against UV rays, fire, and thermal degradation, then here arises a question of "do they work while composite is aging."

4.1. Aging in Three Phase Carbon Fiber-Reinforced Composite. The filler or third phase imparts a wide variety of properties according to their chemistry. In some cases, it has been observed that several fillers affect aging in both ways: as a barrier or as an enhancer/accelerator. For example, the CNT is the third phase in the carbon/bismaleimide resin (BMI) composite. This three-phase composite with 2.5 wt% of CNTs after 72 hours of hydrothermal aging showed a decrease in impact strength and bending strength compared to the two-phase composite [2]. It was due to the increase in the number of interfaces. An increase in the quantity of the third phase increases the defect probability.

In the same test, composites were aged for 168 h with 1.5 wt% of CNTs. The samples had a maximum value for flexural and impact strength. With CNTs, there was a 45% increase in bending performance and 41% in impact performance [87].

The polymer composite undergoes irreversible changes when exposed to UV rays [88, 89]. In another case of filler addition, organophosphorous is blended with thermoplastic polymer to protect the composite from UV rays [82]. For UV protection, 5 wt% nanosilica and 15 wt% microsilica in a carbon-reinforced epoxy composite showed barrier properties. The samples were aged by UV radiation lamps for 1000 h. The aged specimens became opaque and more yellowish. The UV radiation absorbed by epoxy oxidized the aromatic groups present in it. The sample with 15 wt% microsilica showed a less yellowish color, and the sample with 5 wt% nanosilica showed the least degradation as compared to the neat sample. The nanosilica showed enhanced properties because of its intactness and high surface energy [90, 91]. Composite coatings with numerous materials are also in trend to avoid permanent deformation and enhance durability. The coatings are against the action of the weather, humidity, temperature, and UV rays. Polyurethane is a well-known coating material [92]. The other UV-resistant materials that can be added to coatings are carbon black, zinc oxide, titanium oxide, silicon rubber, graphene, and other suitable nanoparticles [93, 94]. From the literature, the use of carbon black in coatings worked not only as UV-resistant but also controlled the microcrack propagation in the coating [83, 84].

In contrast to barrier properties, sometimes the presence of filler does not affect aging. It can be true for specific aging that a filler might not have a particular feature to accelerate or slow down the process, for example, the addition of 12 phr of kaolinite clay in the composite. The composite was pultruded with the inner core as carbon fiber and the outer shell as glass fiber. The composite samples were aged at 200°C for 672 h. The results showed that the incorporation of inorganic filler into the matrix helps the composite resist oxygen transport, but it does not play a significant role in the oxidation mechanism. Moreover, fillers can also worsen the situation. For instance, in thermal aging, the addition of silica being inert has a negligible effect, and aluminum
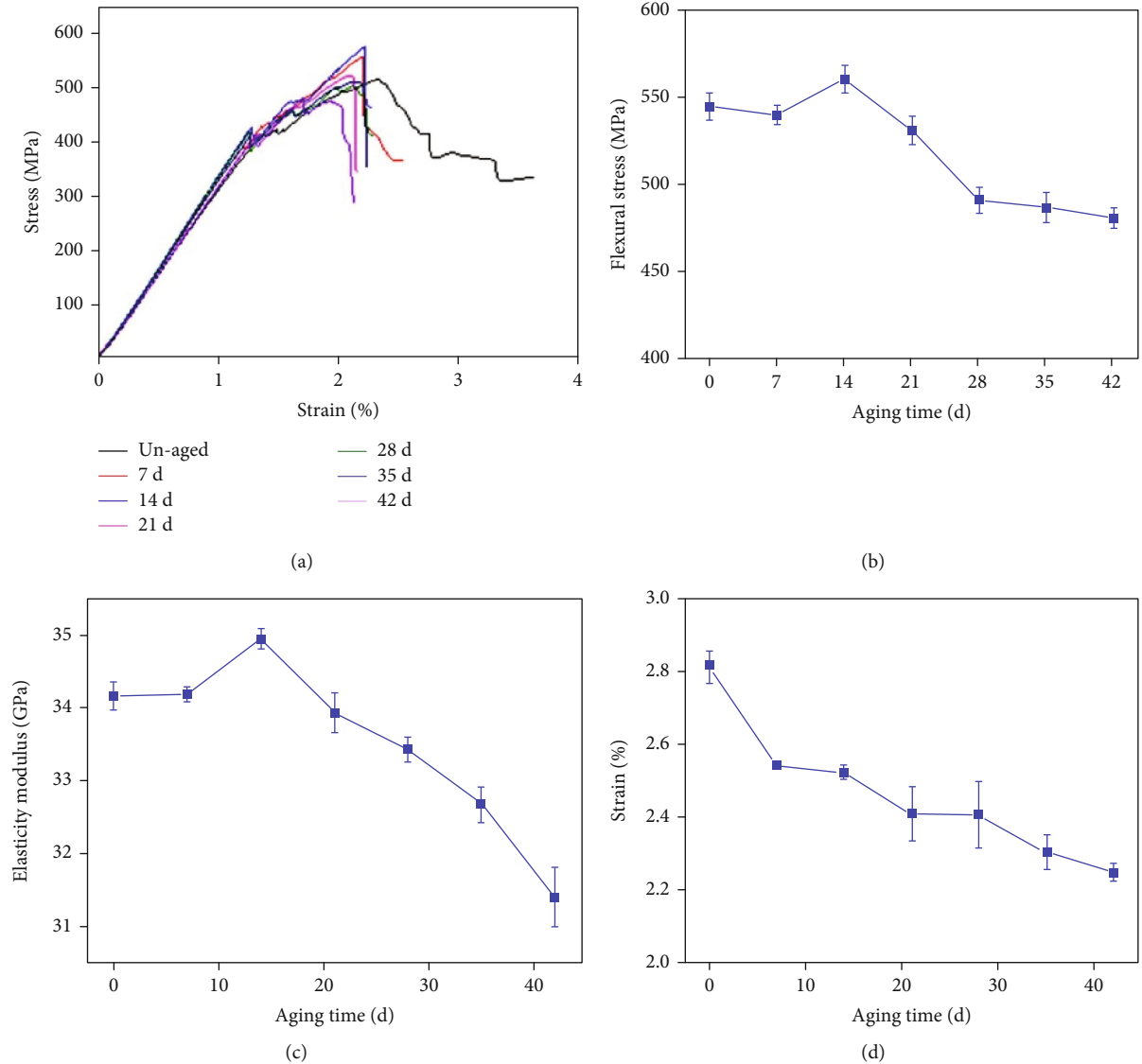
(a)

(b)

(c)

(d)

FIGURE 8: Flexural properties for composites as a function of hydrothermal aging time. (a) The S-S curves of composites, (b) flexural stress, (c) elastic modulus, and (d) % strain [84].

hydroxide accelerates the oxidation process. Therefore, we can say that all fillers have distinguished performance, and thermal oxidation is affected by filler chemistry [95]. Table 1 exhibits the details of the three-phase composite on the composite performance.

4.2. Synergistic Effect of Third Phase and Aging on Composite Performance. The MWCNTs are proven to enhance the mechanical properties of the composite. The composites with MWCNTs were aged by soaking them in water at 20°C and 60°C, HCL solution (5 wt%) at 20°C, and NaOH solution (10 wt%) at 20°C for an immersion time of 720 h. After that, the composite samples were dried for 48 hours at 40°C and then placed at room temperature for 120 hours. According to ASTM 3039, the results showed that hydrothermal degradation was mainly caused by the matrix absorbing water at high temperatures. The microcracks appeared in the matrix and hence decreased the mechanical

strength. The three-phase composites when aged sustained their mechanical properties due to the hydrophobic nature of CNT. A three-phase unaged composite that had 0.5 wt% MWCNTs showed a tensile strength of 1354 MPa, and after aging (water soaking at 20°C), it showed a tensile strength of 1336 MPa. It has slightly less but sustained mechanical performance.

The CNTs can resist γ radiation. As a third phase, CNTs were introduced in both the interface and matrix of the carbon fiber-reinforced epoxy composite. The modified composite had remarkable achievements in bending strength and bending modulus after the fatigue testing. In comparison to the unmodified composite before fatigue, the modified composite bending strength had a 28.5% increase, and the bending modulus had a 41.4% increase due to CNTs. However, after fatigue, the modified composite had a 32.1% and 73.5% increase as compared to the unmodified composite for bending strength and bending modulus,

TABLE 1: Effect of the third phase on the performance of composites (before aging).

Third phase	Specs of the third phase	Weight %	Reinforcement	Matrix	Test method/standard	Properties/remarks
AFMWCNT [96]	Diameter: 20-30 nm Length: 20-30 μm	1.5 wt%	Carbon fiber	Araldite LY556 epoxy	Tensile test Shear test ASTM D 3039 [99]	In-plane shear modulus 3.89 GPa
Electrospun functionalized polyacrylonitrile grafted glycidyl methacrylate (PAN-g-GMA) nanofibers [97]	PAN Mw: 150 kDa GMA 97% purity	5 layers of nanofibers (1 g nanofiber per square meter)	Carbon fiber	Araldite LY 564	Tensile test Short beam shear strength test 10 mm × 30 mm × 75 μm ASTM D 3039 ASTM D 2344 [98]	Short beam shear strength 46.8 MPa
GNPs [99]	GNP (xGNP M-25)	1.0 wt%	Carbon fiber	Epon 828 epoxy	Tensile test ASTM D 638-15 [21]	Tensile modulus 70.65 GPa
MWCNT [100]	Code: US4306	1.0 wt%	Carbon fiber	Epoxy	Tensile test ASTM D 3039	Tensile modulus (transverse) 6.8 GPa
SiC particulate filler [101]	35 μm and bulk density of 1.49 g/cm^3	10.0 wt%	Carbon fiber	Epoxy (MOMENTIVE-MGS) L285	ILSS ASTM D 2344	ILSS 139 MPa
Carbonized ethyl acetate/sucrose at 290°C in air to modify the surface of carbon fiber [102]	Tianjin Fuyu Chemical Reagents Co.	—	Carbon fiber	TDE-85 epoxy resin (4,5-epoxycyclohexyl-1,2-diglycidyl diformate; epoxy value, 0.85)	ILSS ASTM D 2344	ILSS 34 MPa
GNP [23]	Diameter: 0.5-5 μm Thickness: 100-200 nm	5 wt%	Carbon fiber	DGEBA (GY6010)	In-plane shear test ASTM D 5379 [106]	In-plane shear modulus 6.8 GPa
PEEK [21]	—	6 wt%	Twill weaved carbon fiber	bisphenol-A-based epoxy resin (Araldite LY 556)	SBS ASTM D 2344	Short beam shear (SBS) 45 MPa

TABLE 2: Synergistic effect of fillers and aging on the performance of composites.

Fillers	Filler specifications	Reinforcement	Matrix	Type of aging	Test method/standard	Properties/remarks
Carbon nanofillers & MWCNTs [106]	CNF Avg dia 130 nm, Avg length 1.5 μm; CNT Avg dia 9.5 nm, Avg length 20 μm	—	Epoxy (RTM6, Hexcel)	Hydrothermal aging at 70°C for 4 weeks	3-point bending ASTM D790	Elastic modulus before aging 3.03 GPa and after aging 3.10 GPa
CNT grafted ceramic microfiber by CVD 2 wt% [107]		Aluminoborosilicate fiber mats (ultra temp 391 fibers, Cotronics Corporation, areal density of 371 g/m^2)	Epoxy (RTM6, Hexcel)	Hydrothermal aging at 60°C for 90 days	3-point bending ASTM D790	Bending modulus before aging 12.05 ± 0.87; After aging 12.42 ± 2.25
ZnO nanoparticle at 10 wt% [108]	Particle size: 30 ± 10 nm	—	Epoxy resin by Wuxi Resin Co., China	Water-heat water aging at 80°C for 105 days	Tensile test ASTM D3039	Tensile strength of ZnO added before aging 65 ± 1 MPa; After aging 58 ± 2.0 MPa
MWCNT at 0.5 wt% [109]	Surface-treated with sulfuric acid and nitric acid (3 : 1) MWCNTs were purchased (Zhongke Nano Materials Co., Ltd, Chengdu, China)	Carbon fiber Wt. 300 g/m^2 Thickness is 0.167 mm	Epoxy bisphenol-A E51 resin	Hydrothermal aging at 60°C Acid & alkaline immersion	Tensile test ASTM D3039	Tensile modulus before aging = 124 ± 2 GPa; At 60°C = 120 ± 2 GPa; In acid = 123 ± 2 GPa; In alkaline = 123 ± 2 GPa; Tensile strength before aging 1355 ± 1.5 GPa; At 60°C = 1220 ± 1.5 GPa; In acid = 1323 ± 1.5 GPa; In alkaline = 1323 ± 1.5 GPa
Nano clay at 2 wt% [109]	Surface-treated halloysite nanoclay (Mingchuang Materials Co., Ltd, Xi'an, China)	Carbon fiber Wt. 300 g/m^2 Thickness is 0.167 mm	Epoxy bisphenol-A E51 resin	Hydrothermal aging at 60°C Acid & alkaline immersion	Tensile test ASTM D3039; 3-point bending test ASTM D7264 [110]	Tensile modulus before aging = 124 ± 2 GPa; at 60°C = 120 ± 2 GPa; In acid = 123 ± 2 GPa; In alkaline = 123 ± 2 GPa; Tensile strength before aging 1315 ± 1.5 GPa; at 60°C = 1190 ± 1.5 GPa; In acid = 1303 ± 1.5 GPa; In alkaline = 1303 ± 1.5 GPa

TABLE 2: Continued.

Fillers	Filler specifications	Reinforcement	Matrix	Type of aging	Test method/standard	Properties/remarks
Inorganic nano clay at 2 wt% [111]	Alkylammonium surfactant treated inorganic nanoclay I-28E from Sigma-Aldrich Co. It has a large surface area of 750 m²/g, density of 0.42 g/cc, thickness of 1 nm, and lateral dimension of several microns	Carbon fiber (4 harness satin weave, 3 K tow size, and 0.2286 mm thickness) was obtained from US Composites Inc.	SC-15 epoxy resin from Applied Poleramic Inc.	Seawater with 35 phr salinity for 12 months	Low-velocity impact characterization	Nanoclay-added CFRC had low water absorption (19%) as compared to conventional composite (21%) due to the barrier properties of nanoclay. It also showed overall better performance than conventional composite at different aging periods. However, increased period of aging showed catastrophic failure
Graphene nanoplatelets (5 wt%) & CNTs (0.1 wt%) [112]	Thickness lower than 4 nm and 5 μm of lateral size were supplied by Cheap Tubes & CNTs NC7000 from nanocyl with L = 1.5 μm, Diameter = 9.5 nm	—	Epoxy Araldite LY556 from Huntsman	Hydrothermal aging at 40°C	Thermomechanical analysis	Tg is the same for aged and unaged composite. Aging decreases the modulus of hybrid composite (T < Tg).
CNTs GNPs Carbon nanohorns (CNH) at 0.5 wt% [113]	CNT of diameter 10e20 nm and length of 10e30 mm were obtained from Cheap Tubes Inc. GNP having a thickness of 3-10 nm were purchased from Redex Nano (Ghaziabad, India) CNH was synthesized in-house by using a DC arc discharge technique	—	Epoxy EP-415	Hydrothermal aging cycles Each cycle with duration of 24 h for 12 weeks	Lap shear test ASTM D1002 [114]	Lap shear strength Epoxy = 6.5 ± 0.5 MPa Epoxy/CNT = 14.1 ± 0.5 MPa Epoxy/GNPs = 15.2 ± 0.5 MPa Epoxy/CNHs = 11.8 ± 0.5 MPa

respectively. Moreover, by the ultrasonic microscopic examination of fatigued composites, internal damages in the modified composite were the smallest, and the unmodified composite had larger damage. Also, the storage modulus of the composite was improved from 2.53 GPa to 5.66 GPa [87].

The novel structures also help in resisting the conditions encountered by a composite. To serve this purpose, a bifunctional interleaf consisting of phosphorous-containing polymer blended with thermoplastic polymer was used as the third phase in the carbon fiber-reinforced composite. After interleaving Mode-I and Mode-II fractures, toughness improved by 8.2% and 23.7% compared to unmodified composite, respectively. Similarly, atomic oxygen erosion rates of Mode-I and II delamination surfaces decreased by 45.3% and 31.3% in comparison with unmodified composite, respectively [82].

Other than carbon fiber, glass fiber-reinforced composite is also used for hi-tech applications. And it also finds applications in a corrosive environment with different fillers [103]. It was reported that carbon nanofibers (CNFs) in the glass-reinforced composite were added to check for aging effects. The composite was characterized against three solutions: acidic (HNO_3 pH = 1), seawater (pH = 8.2), and alkaline (KOH pH = 13) for 150 days at 18-25°C. The 0.1 wt% CNFs in the composite showed 10.6% lower water absorption than the 1 wt% CNFs (water absorption was 3.03% higher than that of the unmodified glass-reinforced composite). The CNFs are hydrophobic, but higher concentration increases the specific surface area and agglomeration; thus, an opposite effect occurs. After aging, the highest flexural strength among all three composites was obtained from the 1% CNF composite, which was 29% higher than the unmodified composite. In seawater aging, the 0.1% CNF and 1% CNF composites exhibit 8.80% and 28.5%, respectively, with higher flexural strength than that of the unmodified composite. In acidic aging, the unmodified composite was better than the other two. However, in alkaline aging at 1 wt%, CNFs were better than the other two [104, 105].

Furthermore, Table 2 exhibits the different nanofiller impacts on aging and on the performance of the composite.

5. Conclusions

The aging of composite material causes a loss of mechanical performance. It has been reported that the interface and matrix in a composite undergo aging first, while carbon fiber reinforcement remains intact. However, depending on the aging conditions and time of exposure, the composite serves its purpose, and regular nondestructive testing is required to investigate the defects produced in it. Different aging conditions hit composites differently. In thermal aging, time plays a major role. The more the time of exposure to elevated temperature or exposure in oxygen the more will be the damage in composite. In hydrothermal aging, there are different liquids, for example, salt water, salt spray aging, or pure water. These liquids mostly affect composites during their application span. It was seen that pure water causes more damage than sea or salt water. During acidic and alkaline exposure, alkaline causes more damage than acid. The UV radiation causes cracks, fading, delamination, and the release of free radicals from composites, thus reducing their performance and shortening their life span. To slow down aging or to protect the composite, some researchers introduced fillers. However, mostly, it was observed that the aging behavior of a three-phase composite was the same as that of CFRC without a third phase. For example, the delamination and crack propagation phenomena were almost the same for aged and unaged three-phase composite materials. However, the use of metallic oxides in the third phase can accelerate or inhibit the thermal aging of the composite material. It is also concluded that carbon nanofiller enables the composite to perform adequately in different environments.

5.1. Future Perspectives. Composites are made for diverse applications, e.g., electrical, flame retardancy, optical, and thermal performance. However, these attributes are not intrinsic. The composites are fabricated to achieve these characteristics. Mostly, fillers or reactive substitutes are introduced into the matrix to serve the purpose. But there needs to be studied the response of fillers/reactive substitutes during aging, mechanisms of aging, and solutions to keep the performance sustainable.

Data Availability

No new data was created in this study. Data sharing is not applicable to this article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Authors' Contributions

Ayesha Afzal contributed to the initial manuscript writing and literature review. Muhammad Kashif Bangash contributed to the literature review related to aging effects on carbon fiber-reinforced composites. Asif Hafeez provided expertise in mechanical testing and data interpretation.

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