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RESEARCH ARTICLE

Polymer COMPOSITES WILEY

Long-term residual properties and durability of glass fiber reinforced polymer composite exposed to alkaline solution and natural weather for a decade

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Graphical Abstract

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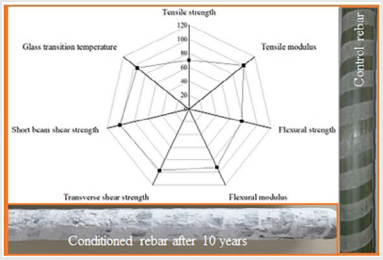

The residual thermal and mechanical properties of pultruded glass fiber reinforced polymer composite bar were examined following a decade-long exposure to alkaline solution and natural weather conditions, thus evaluating their long-term durability.

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Long-term residual properties and durability of glass fiber reinforced polymer composite exposed to alkaline solution and natural weather for a decade

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Abstract

This paper presents a comprehensive analysis of the residual thermal and mechanical properties of pultruded glass fiber reinforced polymer composite bars following a decade of conditioning in an alkaline solution and exposure to natural weather conditions. The study focuses on evaluating the changes in the glass transition temperature (T_g) of the polymer matrix and its impact on the bar's mechanical performance. The results indicate that the T_g retained approximately 94.7% of its initial value, with the decrease attributed to the plasticizing effect of absorbed water. The flexural modulus, flexural strength, and transverse shear strength were found to retain 91.8%, 77.2%, and 97.3% of their original values, respectively. The reductions in strength and stiffness were primarily attributed to a weakening in the bonding between the fibers and the polymer matrix. Fractographic analysis revealed that the failure of the plasticized and softened polymer matrix contributed to the observed reductions in strength. Interestingly, the short beam shear strength remained relatively unchanged, as the diffusion of water into the core of the bars at ambient temperatures had a minimal effect. This slower water diffusion in the core led to insignificant degradation of the short beam shear strength.

Highlights

- GFRP bars were exposed to alkaline solution and natural weather for a decade.
- The water diffusion was a key factor in determining the residual properties of the GFRP bars.
- The absorbed water plasticises the polymer matrix in GFRP bars.
- The mechanical property losses in GFRP bars were associated with changes in the matrix property.

KEYWORDS

alkaline solution, flexural strength, glass fiber polymer composite, glass transition temperature, natural weathering

1 | INTRODUCTION

The corrosion of steel bar in concrete structures due to carbonation and chloride attacks in marine and de-icing salt environments is a significant problem. Surveys conducted in the USA and Canada have revealed that 40% of bridges and parking buildings that are 40 years or older suffer from structural defects caused by steel bar corrosion.[1,2] The repair and maintenance costs associated with these deteriorated structures are substantial. Pultruded glass fiber reinforced polymer (GFRP) composite bar has the potential to enhance the corrosion resistance of concrete structures, thereby increasing their service life and reducing rehabilitation expenses compared to traditional steel bar.

Despite the growing use of GFRP bars in civil construction, there is still hesitation in adopting it due to a lack of long-term performance data in alkaline environments. In strong alkaline environments, glass fibers experience weight loss and strength degradation as a result of chemical reactions with hydroxide ions (OH—) present in alkali solutions, leading to the breakdown of the silica (—Si—O—Si—) network in the glass fibers. Additionally, water molecules can cause irreversible chemical changes in the GFRP bar matrix through hydrolytic reactions, resulting in reduced toughness and fracture strain. Consequently, there has been significant academic and industrial research interest in studying the durability of GFRP bar in alkaline solutions. According to the literature,[3] E-CR glass demonstrates greater resistance to strong alkalis compared to E-glass. In terms of the matrix, epoxy vinyl ester resin is less susceptible to hydrolytic reactions in moist environments due to a lower presence of ester groups (—O—CO—) in the backbone of polymer chain, unlike polyester resin. While epoxy resin is also resistant to chemical attacks, it is more expensive than epoxy vinyl ester resin.

To date, most research studies have focused on investigating the durability of GFRP bar under accelerated conditions involving variations in temperature, time, matrix, glass fiber, pre-load, and their combined influences, with an emphasis on determining physical, mechanical, and thermal changes.[3–16] Only a limited number of works have explored the durability of GFRP bar under ambient conditions.[10,17] Furthermore, some literature reports have presented a restricted number of tests such as water absorption, Fourier transform infrared spectroscopy, scanning electron microscopy, and energy dispersive x-ray analysis on GFRP bars that have been in service for extended periods, such as in bridges.

In existing literatures, it is common for GFRP bars to be conditioned at high temperatures for short periods, which represents a "worst-case" scenario that accelerates the diffusion of alkaline solution into the matrix and results in rapid deterioration of the fiber/matrix interfaces. Moreover, high temperatures can cause internal cracking in under-cured GFRP bar due to post-curing, allowing excessive diffusion of the alkaline solution within a short time frame. By conducting systematic investigations and observing the physical, mechanical, and thermal changes of the pultruded GFRP bar over a 10-year period, this study aimed to provide a more realistic assessment of the bar's long-term performance.

2 | EXPERIMENTAL

2.1 | Material and alkaline conditioning

The pultruded GFRP bars, with a core diameter of 13.2 mm, were manufactured using unidirectional E-CR glass and Epoxy vinyl ester resin. The alkaline solution required for the experiment was prepared according to the recommended method in the reference,[18] utilizing $Ca(OH)_2$, NaOH, KOH and deionized. The initial pH of the alkaline solution was approximately 13, and the final pH was measured to be around 9.8. To conduct the experiment, the GFRP bars, with a length of 950 mm, were fully immersed in a plastic container filled with the alkaline solution. The container was then exposed to natural outdoor weathering conditions for a period of 10 years. The local annual average outdoor temperature was about 13.9°C, with a temperature fluctuation of approximately 9.5°C.

2.2 | Water absorption

The GFRP bar specimens, which had been conditioned in an alkaline environment for 10 years, were used for the experiment. These specimens had a length of 40 mm. To begin the experiment, the specimens were first weighted on a digital scale with an accuracy of ±0.0000 g. Subsequently, they were placed inside a glass bottle that was filled with deionized water. The purpose of this setup was to measure the water absorption of the specimens at a temperature of 60°C. The water absorption was recorded at various time intervals, up to a maximum duration of 13,008 h (~1.5 years). To calculate the water absorption, the following equation was utilized:

$$M_t = \{(w_t - w_o)/w_o\} \times 100 \quad (1)$$

In this equation, M_t represents the water absorption, w_t denotes the weight after exposure, and w_o indicates the weight before exposure. A total of six specimens were tested.

2.3 | Differential scanning calorimetry (DSC)

The glass transition temperature (T_g) of the polymer matrix in GFRP bar provides valuable information about the thermal properties and behavior of the bar under different conditions. In this study, T_g was assessed using a TA Instruments Modulated DSC 2920. For the analysis, a sample weighing approximately 20–35 mg was prepared and subjected to two consecutive scans. The heating rate was set at 5°C/min, starting from −40 and ending at 145°C. The DSC instrument utilized a modulation period of 60 s and a modulation peak of ±0.5°C. Five repetitions of each type of GFRP bar (control and conditioned) were scanned during the experiment.

2.4 | Short beam shear test

The short beam shear test is an effective and widely used method for measuring the shear strength of parallel fiber reinforced pultruded GFRP bar. The test procedure followed the Standard Test Method for Apparent Horizontal Shear Strength of Pultruded Reinforced Plastic Rods by the short beam method, ASTM D4475.

To determine the short beam shear strength (SBSS), five specimens from each type of GFRP bar (control and conditioned) were tested. The test involved applying a load until the specimen fractured in shear. The shear breaking load (P) and the diameter of the specimen (d) were recorded for each test. The SBSS was then calculated using the following equation:

$$\mathrm{SBSS} = 0.849 \times (\mathrm{P}/\mathrm{d})^2 \tag{2}$$

2.5 | Transverse shear test

The transverse shear strength (TSS) of GFRP bar was determined following the Standard Test Method for Transverse Shear Strength of Fiber-reinforced Polymer Matrix Composite Bars, ASTM D7617/D7617M. The test aimed to assess the material's resistance to shear force and provide valuable insights into its structural performance.

The maximum failure load (P_s) and cross-sectional area (A) of the specimens were used to calculate the TSS using the following equation:

$$\mathrm{TSS} = \mathrm{P_s}/2A \tag{3}$$

To conduct the test, a universal testing machine (WDW-200E) equipped with a 200 kN load cell was utilized. The specimens were subjected to a testing speed of 2 mm/min, and the load was applied until failure occurred. Five specimens from each type of GFRP bar (control and conditioned) were tested.

2.6 | Flexural test

The flexural test was conducted using the universal testing machine (WDW-200E) in the three-point bend mode. The test was performed at a crosshead speed of 1.5 mm/min. The span-to-depth ratio of the test fixture was set to 16:1.

To calculate the flexural strength (σ_{fs}), the following equation was utilized:

$$\sigma_{fs} = F_f L/\pi R^3 \tag{4}$$

In this equation, F_f represents the load at fracture, R denotes the radius of bar sample and L signifies the distance between support points. The distance between the support points was 212 mm. Five specimens from each type of GFRP bar (control and conditioned) were tested.

2.7 | Tensile test

The tensile test was carried out in accordance with the procedure specified in ASTM D7205/D7205M, which is the Standard Test Method for Tensile Properties of Fiber Reinforced Polymer Matrix Composite Bars. A summary of the tensile properties of the control and conditioned rebars are presented in Table 1 to compare with the other mechanical properties as presented in this report. Additional information regarding the tensile testing methodology, data and analysis can be found in the earlier report.[19]

2.8 | Scanning electron microscope (SEM)

The samples in this study were investigated using a Hitachi Regulus 8230 high-resolution scanning electron microscope (SEM). The SEM operated at an accelerated voltage of 3 kV and utilized a secondary electron detector. Prior to the analysis, the samples were metalized with platinum using a Quorum Q150V plus sputter coater to improve the conduction of electrons during the imaging process.

3 | RESULTS AND DISCUSSION

3.1 | Water absorption

The water absorption of GFRP bar can vary depending on several factors, including the specific type and composition

	Control GFRP bars	10-years GFRP bars	Retention, %
Tensile strength, MPa	886	619	69.9
Tensile modulus, GPa	55	55.1	100.2

TABLE 1 Tensile properties of the control and conditioned (10-years) bars.

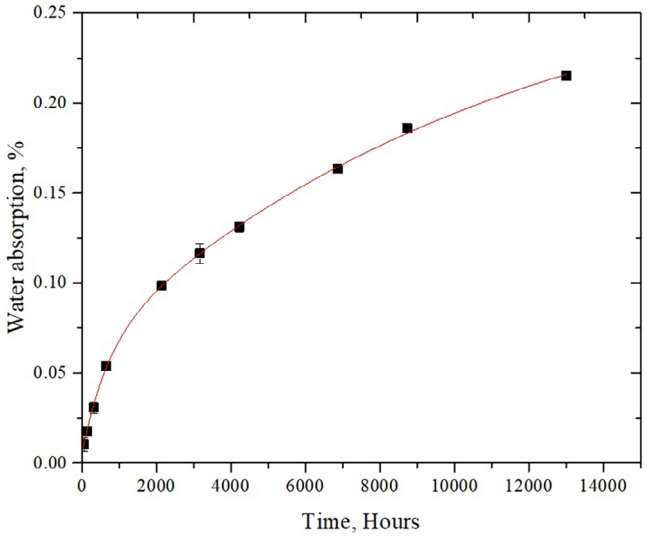


FIGURE 1 Water absorption of 10-years GFRP bar at 60°C.

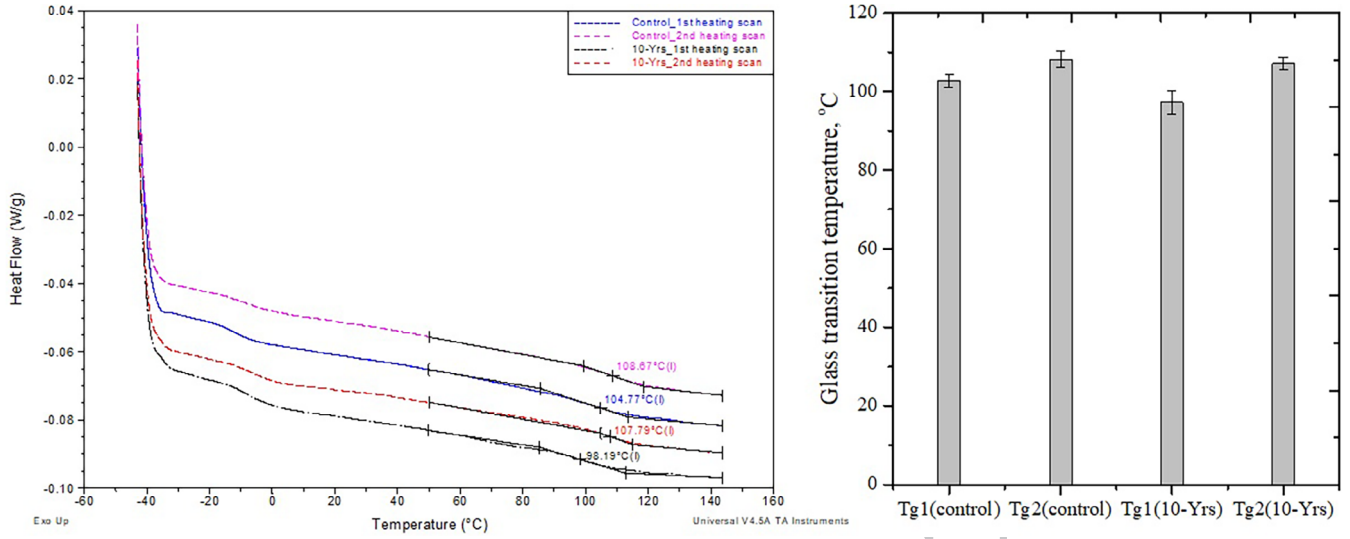
of the GFRP material, the manufacturing process, and the environmental conditions it is exposed to. In general, GFRP bar absorbs water through capillary action and diffusion. In this study, Figure 1 shows that the water absorption of 10-years specimens was continued to increase gradually over the time and demonstrated a non-Fickian diffusion process.[20] The non-Fickian behavior was due to gradual capillary transport of water into the polymer matrix and fiber/matrix interfaces during the immersion period at elevated temperature.[9] It is worth noting that the water absorption of GFRP composites does not always reach equilibrium even after a long immersion period. In this work, the average water absorption of 10-years specimens was determined to be 0.215% after being immersed for 13,008 h at 60°C. This water absorption value was lower compared to the other reported results in the literature regarding GFRP bar.[3,21,22] The lower water absorption observed in the 10-years GFRP bar can be attributed due to its lower porosity content, as mentioned in another report.[19] The reduced porosity likely limited the amount of water that could penetrated and be absorbed by the material, resulting in the lower absorption observed in this study.

3.2 | Glass transition temperature (T_g)

The T_g is an important property of the polymer matrix, as it affects the durability and performance of the GFRP bar in different hostile environment. The polymer matrix serves to protect the load-carrying glass fiber component from degradation, particularly in corrosive conditions. In the DSC traces shown in Figure 2A, the heat flow curves exhibit a phase change step, which is referred to as the glass transition. This glass transition represented the shift of the polymer matrix from an elastic behavior to a viscoelastic behavior within a temperature range of 90 to 115°C. Furthermore, the DSC heat flow curves also provided information about the curing process of the GFRP bars. The absence of any residual exothermic cure reaction during the first heat scans indicates that the GFRP bars were well cured. This means that the polymer matrix had its desired level of cross-linking and bonding, contributing to the overall structural integrity of the GFRP bar.

The purpose of evaluating the glass transition temperature of the first heating scan (T_{g1}) was to assess the thermal stability changes in the polymer matrix caused by conditioning. On the other hand, the glass transition temperature of the second heating scan (T_{g2}) was measured to determine the degradation of the polymer matrix resulting from conditioning. Figure 2B presents the average T_{g1} and T_{g2} values of both the control and conditioned GFRP bars. For the control bar, T_{g1} was found to be 102.6°C, which increased to 108.1°C after the second heating scan (T_{g2}). This increase in T_g of the control bar was attributed to anti-plasticization, caused by the evaporation of inherent moisture and volatile processing additives during the elevated temperature thermal scanning.

After 10 years of conditioning in an alkaline solution, T_{g1} of the GFRP bar decreased to 97.18°C. The decrease of approximately 5.4°C in T_g was attributed to absorbed water that plasticized the polymer matrix during the conditioning period. In the plasticization process, the absorbed water molecules caused swelling of the polymer matrix, resulting in increased distance between polymer segments and enhanced polymer chain mobility or free volume. Consequently, the T_g of the polymer matrix decreased. Following the second heating scan, T_g of the conditioned GFRP bar increased to 107.1°C, which approached the T_{g2} value of the control bar. This increase in T_g was again attributed to anti-plasticization due to the evaporation of absorbed water during the thermal scan. The exposure of GFRP bar to an alkaline solution led to a reversible chemical modification within the polymer matrix, thus conditioning it.



Poor quality

Q27 **FIGURE 2** (A) Typical DSC thermograms of the control and 10-years GFRP bars, and (B) the glass transition temperatures of the first and second heat scans of the control and 10-years GFRP bars.

Statistical analysis (paired sample t test) confirmed that the difference between T_{g2} of the control and T_{g2} of the conditioned bars was not statistically significant. Thus, based on the thermal analysis, it can be concluded that the polymer matrix of the GFRP bar did not chemically degrade after being conditioned in the alkaline solution for a period of 10 years. Other research studies[4,23,24] have also reported that the epoxy vinyl ester matrix of the bar is less susceptible to hydrolysis or degradation reactions at ambient temperatures.

3.3 | Transverse shear strength (TSS)

GFRP bars find a multitude of applications, including their role as dowels within joints of concrete pavements.[23,25] Transverse shear assumes particular significance in scenarios where the shear force on a dowel bar needs to be effectively transferred across a movement joint. This is notably relevant in contexts such as the construction of suspended floor slabs or ground floor slabs. The behavior of the control and 10-years conditioned GFRP bars under transverse shear load is discussed, the F3 results are presented in Figure 3A. It can be observed that the transverse shear load of the control bar increased linearly up to 43% of the maximum shear breaking load, afterwards the shear phase changed slightly due to buckling of the glass fibers under compression. Once the maximum breaking load was reached, the recorded transverse shear load was then diminished irregularly and caused to split the test specimen into three pieces as shown in the photos in Figure 3A. In contrast, the conditioned bar exhibited changes in the shear phase at relatively low shear loads, specifically at 11% and 39% of the maximum breaking load. These observed differences were attributed to softening of the polymer matrix (indicated by a decrease in T_g) due to water absorption during the conditioning process. Figure 3B presents the average TSS, which was found to be 178.5 MPa for the control bar. After 10 years of conditioning, the TSS decreased by 2.75%. This slight reduction in TSS was also attributed to the softening of the matrix in the 10-year GFRP bar. However, it is noteworthy that the TSS retention of the 10-years GFRP bar outperformed the reference works,[23,26,27] indicating its favorable performance.

SEM fractography was utilized to investigate the causes and mechanisms of bar failure following conditioning. In Figure 4A, the fractured surface displayed F4 fragmented fibers and matrix debris, which resulted from compressive shear load. The fibers were observed to be split into small pieces instead of being pulled out from the matrix while bearing the load; this was ascribed to strong bonding between the fiber and matrix. Figure 4B provides evidence of buckling of fiber and shear fracture of the bar due to transverse shear load. Moving on to Figure 5A, the presence of single fractured fiber ends pro- F5 vided proof of "chop marks" caused by compression failure. Additionally, smooth, featureless brittle fracture surfaces on individual fibers indicated tension failure. In Figure 5B, a mixed mode of tension and compression failures was observed on the fractured fiber end, resulting from micro-buckling. The well-defined anchoring region of the matrix onto the fiber once again suggested a strong adhesion between the fiber and the matrix. This strong

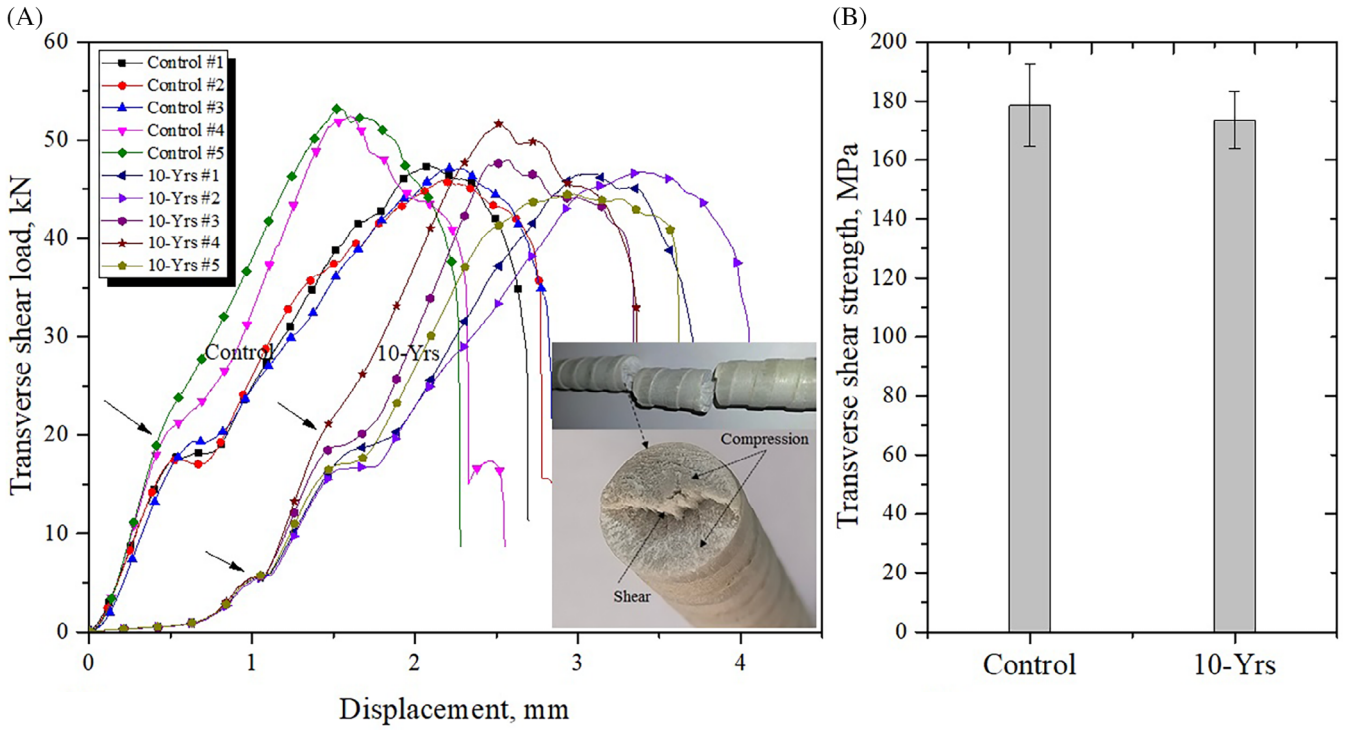


FIGURE 3 (A) Typical transverse shear load versus displacement curves of the control and 10-years GFRP bars, and (B) transverse shear strength of the control and 10-years GFRP bars.

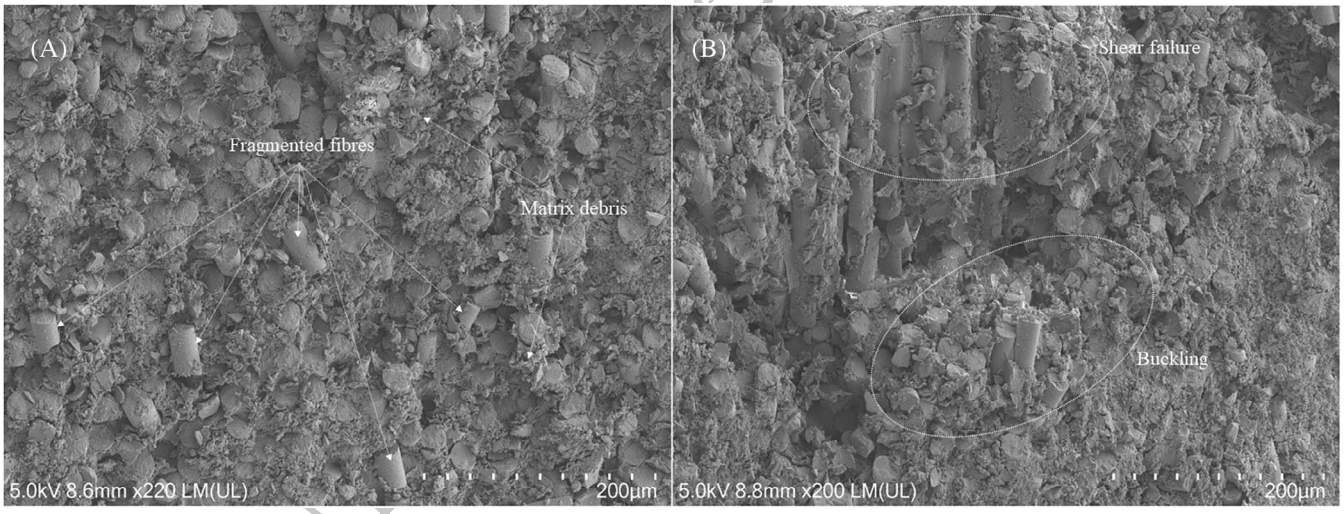


FIGURE 4 SEM micrographs of the transverse shear fractured GFRP bar of 10-years sample showing (A) fragmented fibers, (B) buckling and shear failure.

interfacial bonding played a significant role in the superior TSS retention of the 10-years GFRP bar.

3.4 | Short beam shear strength (SBSS)

The SBSS results are presented in Figure 6A. The average SBSS of the control bar was determined to be 66.5 MPa, which showed a retention of 101.3% after 10 years of conditioning. However, statistical analysis using the paired sample *t*-test indicated that the difference in SBSS between the control and 10-years GFRP bars was insignificant at a 95% confidence level. The GFRP bar specimens were failed in shear horizontally, initiating at the mid-span and propagating along the neutral axis, as depicted in Figure 6B.

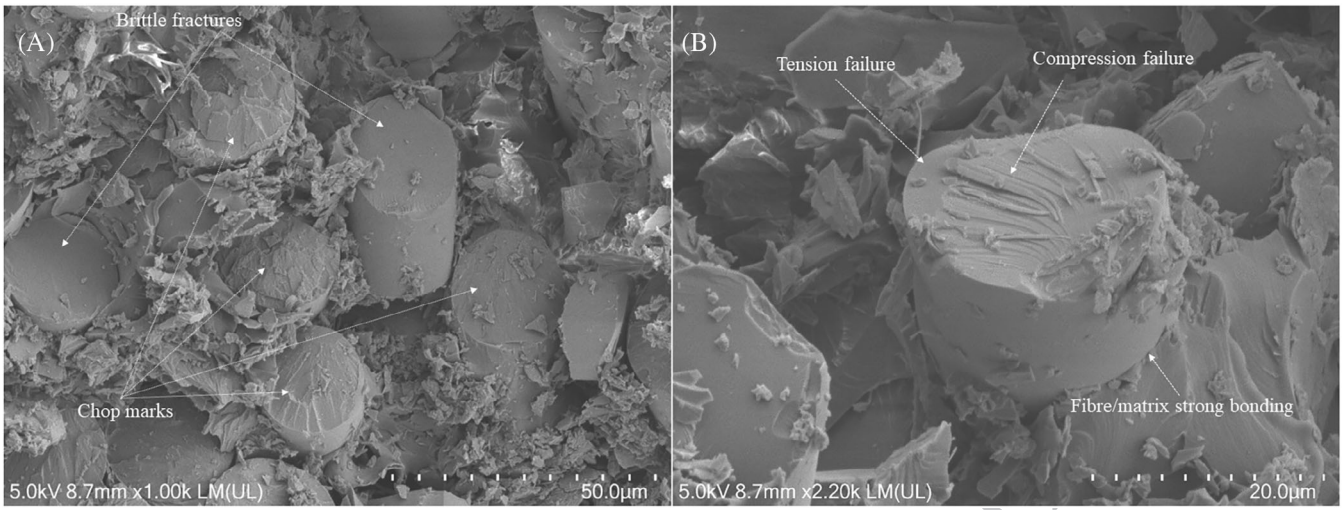
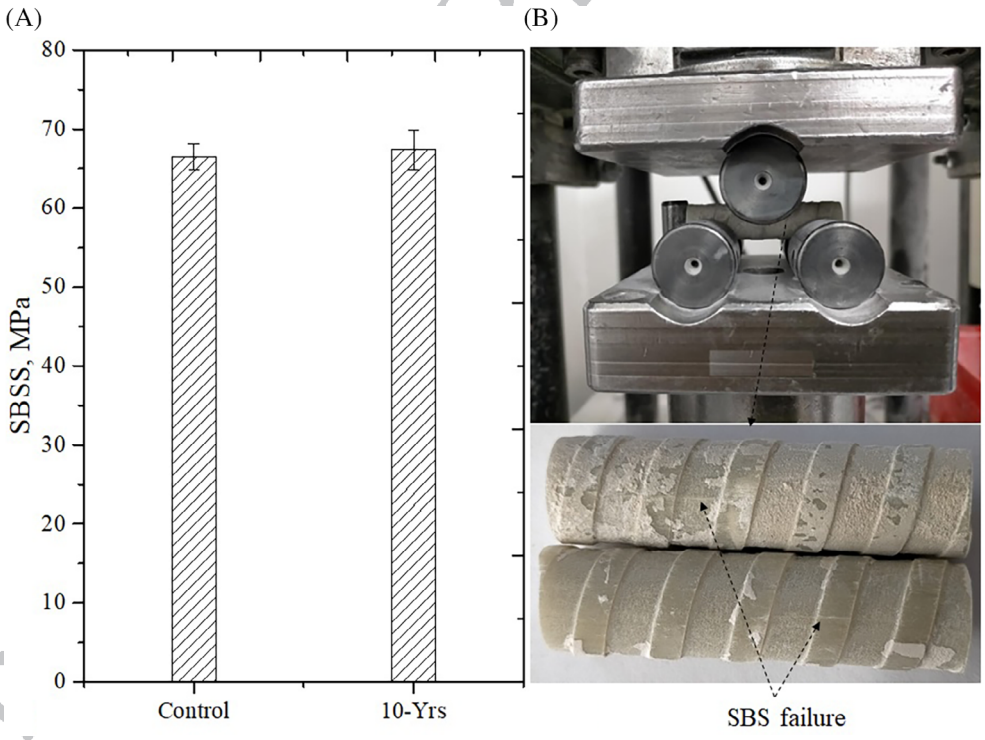


FIGURE 5 SEM micrographs of the transverse shear fractured GFRP bar of 10-years sample showing (A) failure characteristics of the glass fiber, and (B) fiber/matrix interfacial bonding.

FIGURE 6 (A) Short beam shear strength of the control and 10-years GFRP bars, (B) photographs of the shear test fixture and shear failure of 10-years GFRP bar.



In a study by Ramanathan et al.,[28] it was reported that the SBSS of GFRP bars decreased by up to 30% after 18 years of service life due to the presence of voids in the fiber/matrix interface, fiber/matrix delamination and damage of matrix. In contrast, another research[29] found that SBSS remained unaffected when GFRP bars were exposed to concrete under high level of sustained load for 10 years in natural weathering conditions. Additionally, in a separate report,[30] SBSS of GFRP bars extracted from seven bridges after 15 to 20 years of service was found to be 5% to 16% higher than the original bar. This increase in SBSS was attributed to post-curing of the matrix over the time.

However, it is commonly reported that the SBSS of GFRP bars decreases when conditioned in an alkaline environment at higher temperatures. For example, in a previous study,[14] SBSS decreased by 8% to 20% after being conditioned in an alkaline solution for 44 days at 60°C. Another work[9] found a decrease of approximately 8.5% in SBSS after being conditioned in an alkaline solution for 24 months at 60°C. In the present study, the SBSS remained unaffected because the core of bar was

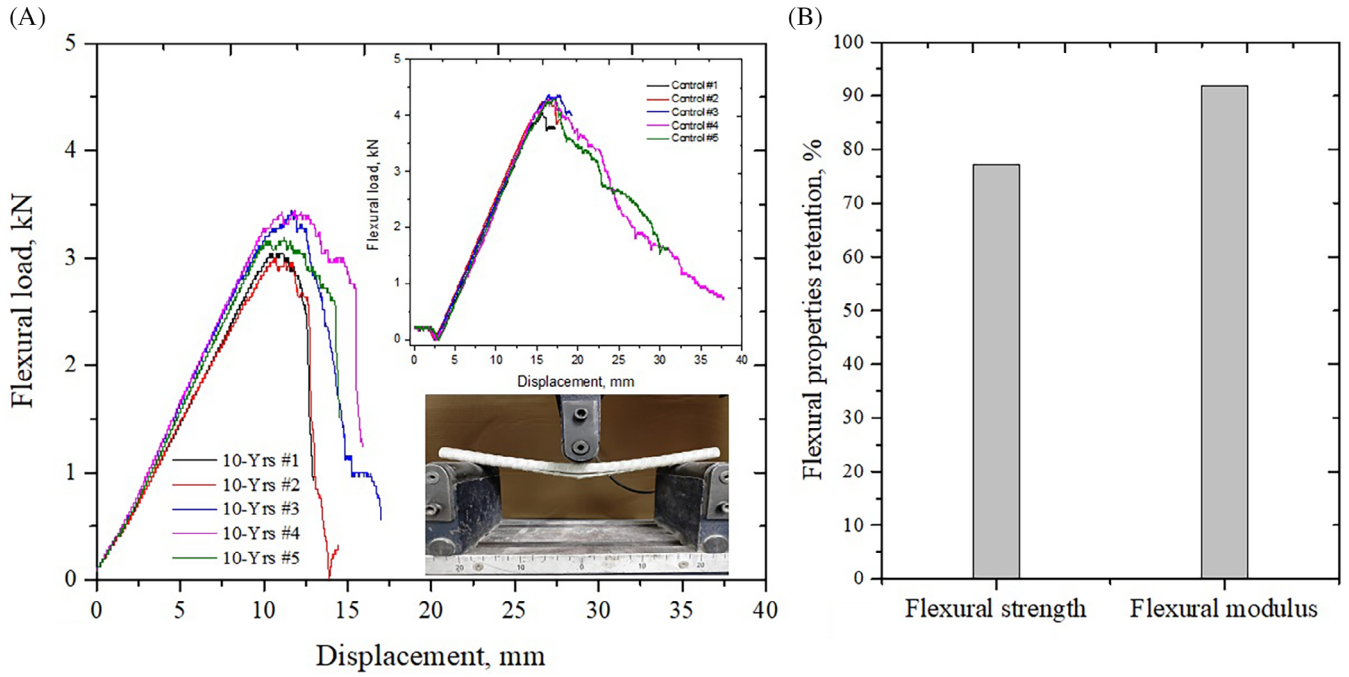


FIGURE 7 (A) Typical 3-point flexural load versus displacement curves of the control and 10-years GFRP bars, and (B) flexural strength and flexural modulus of the control and 10-years GFRP bars.

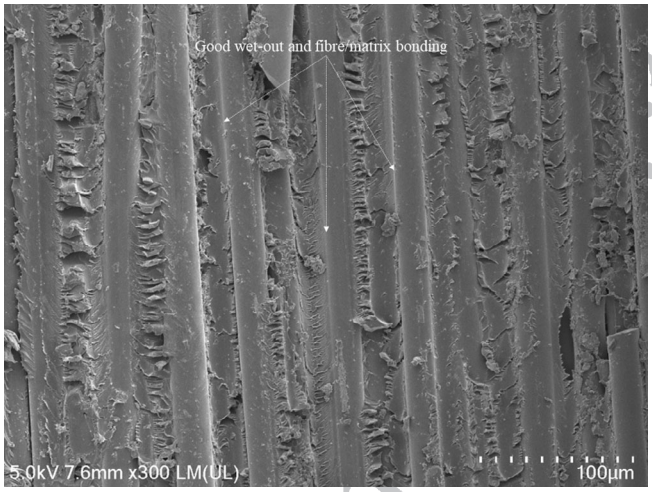


FIGURE 8 SEM micrograph of the flexural fractured GFRP bar of 10-years sample showing fiber wet-out and fiber/matrix bonding.

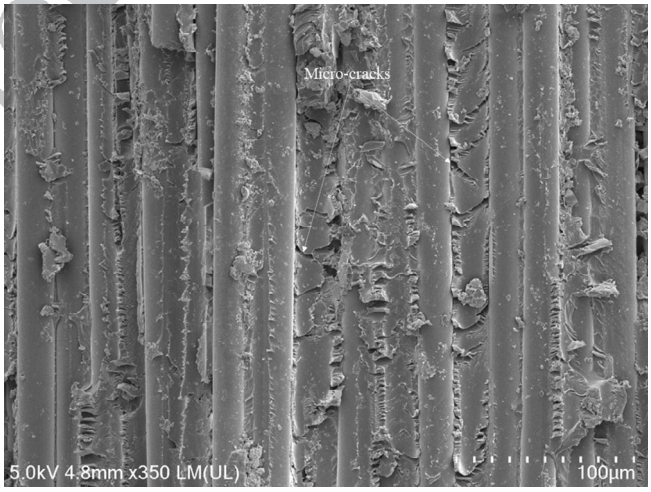


FIGURE 9 SEM micrograph of the flexural fractured GFRP bar of 10-years sample showing micro-cracks and matrix failure.

minimally affected by water diffusion, which was a result of low porosity content of the GFRP bar as reported in previous work.[19]

3.5 | Flexural strength and flexural modulus

While reports on the flexural analysis and design of GFRP bar reinforced concrete beams exist,[29,31–34] there is limited documentation on the effect of conditioning on the flexural properties of GFRP bar sample alone. This work focuses on the residual flexural properties and failure analysis of the 10 years conditioned bars.

Figure 7A illustrates the flexural load versus displacement curves of the control and conditioned bars. The curves exhibited a sharp increase with increasing load until reaching the maximum load, at which point the fracture process was initiated. Subsequently, irregularities and staggered decreases in load occurred due to tensile

shear failures. The maximum axial fiber stresses were concentrated in the outer fibers, and the shear failure was primarily influenced by the interfacial strength between the outer fibers and the matrix, as evident in the photo in Figure 7A. It is also noticeable that the areas under the curves decreased after conditioning. This decrease was attributed to the failure of the softened matrix and the weakening of the interfacial bonding between the fibers and matrix on the outermost surface, which has been in direct contact with the alkaline solution for an extended period. However, it is important to note that the core of the GFRP bar remained unaffected, as confirmed by the earlier SBSS findings.

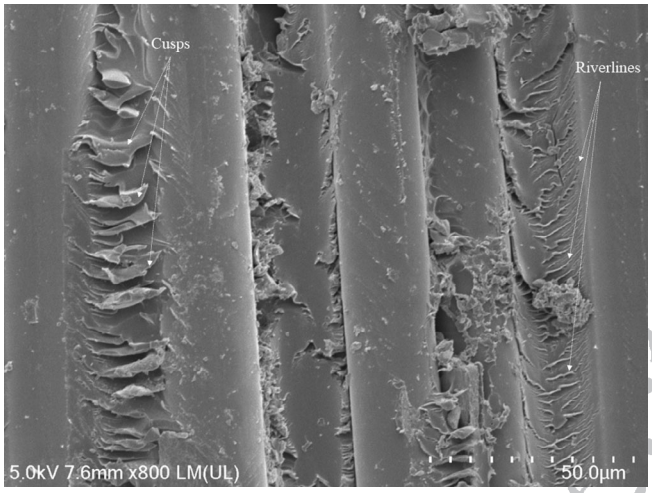


FIGURE 10 SEM micrograph of the flexural fractured GFRP bar of 10-years sample featuring the matrix failure and crack propagation at the fiber/matrix interface.

Figure 7B presents the flexural strength and flexural modulus retentions of the GFRP bars. The average flexural strength and flexural modulus of the control bar were measured to be 1035 MPa and 52.5 GPa, respectively. Following conditioning, the flexural strength and flexural modulus were found to retain up to 77.2% and 91.8%, respectively. The reductions in flexural strength and flexural modulus were again attributed to the softening of the matrix and the deterioration of the fiber/matrix interfacial strength on the outer surface, as mentioned earlier. SEM fractographic analysis was conducted to gain insights into the flexural failure process.

Figure 8 reveals that the fibers were embedded within the matrix, demonstrating good wetting of the fibers and strong fiber/matrix interfacial bonding. The matrix exhibited excellent adherence to the fibers, indicating strong fiber/matrix interfacial bonding once again. During the failure process, the polymer matrix fractured first due to its lower modulus, upon the application of stress to the bar. As a result of the differing elasticities between the matrix and the fiber, interfacial stresses developed at the fiber/matrix interface during the flexural stress transfer process, led to the generation of micro-cracks (Figure 9). The coalescence of these micro-cracks under increasing stress ultimately caused the bar to fail. The "riverlines" observed in the matrix originated from the fiber/matrix interface (Figure 10). These lines, perpendicular to the direction of crack propagation, indicated the gradual development of the crack. Furthermore, the characteristic "cusps" in the matrix indicated shear fracture and exhibited greater deformation due to the increased plasticity of the softened matrix in the 10-years GFRP bar.

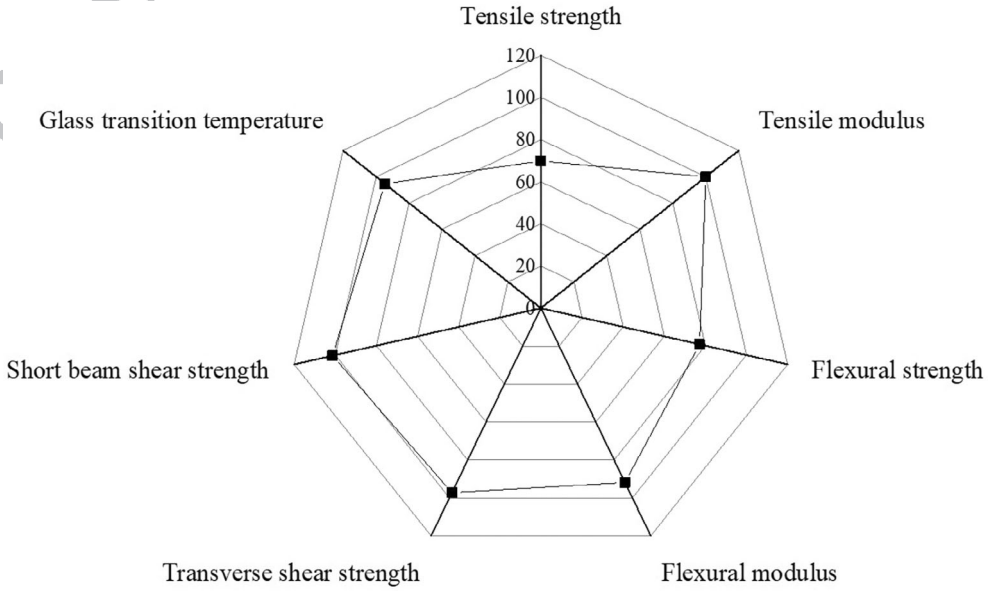


FIGURE 11 Residual properties of 10-years GFRP bar.

3.6 | Comparisons

The comparative residual properties of the 10-years GFRP bar, including the previously reported tensile properties,[19] are depicted in Figure 11. It is evident that the glass transition temperature of the matrix decreased as a result of plasticization and softening caused by water absorption during conditioning. Among the various mechanical properties, the flexural strength and tensile strength were significantly affected due to the softening of the matrix and the deterioration of the fiber/matrix interfaces in the circumferential areas of the bar. The flexural modulus was also affected by conditioning, whereas the tensile modulus remained unaffected. While the modulus of the GFRP bar was primarily influenced by factors such as fiber content, fiber type, and manufacturing process, the observations suggested that the properties of the matrix also impacted the flexural modulus, as the softened matrix deformed at relatively low stresses under flexural loading. Although the TSS experienced a slight decrease, the SBSS remains almost unchanged. These findings indicate that the softened matrix had a limited effect on the short beam shear and transverse shear properties, primarily because the fiber/matrix interfaces in the core of the GFRP bar are minimally affected due to the slow and minimal diffusion of water at ambient temperatures during the conditioning period.

4 | CONCLUSION

This paper focuses on investigating the durability and residual properties of pultruded GFRP bar after a 10-year exposure to natural weather conditions and an alkaline solution. The absorption of water was found to plasticize the GFRP bar matrix, leading to a decrease in the glass transition temperature. The mechanical property losses in the GFRP bar were strongly associated with changes in the matrix properties resulting from the decreased T_g. The analysis of micrographs showcasing matrix failures effectively identified the causes of mechanical failure in the bar. The losses in flexural modulus, flexural strength, and transverse shear strength were attributed to the weakening of the fiber/matrix interface at the outer surface of the bar and the failure of the plasticized, softened matrix. The presence of thicker cusps in the matrix failure indicated greater deformation of the plasticized matrix, resulting in failure at lower flexural stresses. On the other hand, the core of the bar remained unaffected due to the slow diffusion of water at ambient temperatures, which contributed to the relatively stable short beam shear strength. The findings highlight the importance of water diffusion as a key factor in determining the residual properties of the GFRP bar. Additionally, the matrix property serves as a valuable parameter for assessing the failure mechanism and residual properties of GFRP bar following conditioning.

CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT

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

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