Proceedings of the Canadian Society for Mechanical Engineering International Congress
32nd Annual Conference of the Computational Fluid Dynamics Society of Canada
Canadian Society of Rheology Symposium
CSME-CFDSC-CSR 2025
May 25–28, 2025, Montréal, Québec, Canada

MECHANICAL PERFORMANCE UNIDIRECTIONAL GLASS FIBER-REINFORCED THERMOPLASTIC COMPOSITES UNDER VARYING AGING CONDITIONS

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Abstract—Continuous fiber-reinforced thermoplastic composites are gaining attention in the industry due to their high toughness and recyclability, making them a promising alternative to traditional thermosetting composites for primary structural applications. However, challenges such as hydrothermal aging at elevated temperatures must be addressed before these composites can widely be adopted. Elevated temperature conditions increasingly fall within the operating range in field conditions for thermoplastic composite pipes. Although designs aim to prevent direct water exposure of the load-bearing reinforcement phase in these pipes, unexpected or accidental water ingress must be considered for safety-critical pressure piping, as it can lead to the degradation of the reinforcement material over time and eventual pipe failure. This study focuses on evaluating the mechanical resistance of unidirectional glass fiber-reinforced thermoplastic composite tapes (UGFTC) under elevated temperature and different aging conditions. Two types of thermoplastic matrices, polypropylene and polyethylene, were examined. Samples were subjected to thermal aging at 95°C and water immersion in distilled and deionized water at the same temperature for up to 4 weeks. Mechanical resistance was assessed by measuring strength reduction, with a decline of only 10 to 30% being considered as significant, across the three aging conditions during this period. Water uptake behavior was also analyzed by weighing the samples over the 4-week aging period and after its completion. To further understand the damage, optical microscopy was used to compare the condition of the UGFTC samples under each aging condition. This study provides novel insights into the damaging effects on the fiber-matrix interface and the polymer matrix itself when exposed to aging in different conditions, which are key to the mechanical performance of UGFTC materials. Results indicate that samples aged in deionized water experience greater strength reduction compared to those in distilled water. Additionally, samples with polyethylene matrices show more surface damage and reinforcement degradation than those with polypropylene matrices.

Keywords- Unidirectional glass fiber-reinforced thermoplastics, hydrothermal aging, thermal aging, water uptake behavior

I. INTRODUCTION

Continuous fiber-reinforced thermoplastic composites (CFRTC) have superior toughness and thereby are attracting interest as a viable alternative to traditional thermosetting composites for key structural applications [1]. However, certain obstacles must be overcome before the adoption of these thermoplastic-based composites becomes prevalent [2]. Processing CFRTC currently presents greater challenges than their thermosetting counterparts due to elevated processing temperatures and increased melt viscosity. Certain thermoplastics, such as polyethylene (PE) and polypropylene (PP), feature comparatively low glass transition temperatures and can be vulnerable to harsh thermal conditions with long exposure times. Under severe conditions, the mechanical characteristics can significantly diminish due to matrix softening as well as a loss of integrity of the fiber/matrix interface perpetuated by thermal stresses at elevated temperatures. CFRTC are increasingly utilized as a reinforcing component in thermoplastic composite pipes for service at elevated temperatures for extended durations. However, concerns about their durability in different environments restricts their greater use in a larger variety of applications. Consequently, a deeper comprehension of the mechanical behavior of CFRTC under aging conditions is essential.

A variety of scientific studies have investigated the moisture absorption behavior and the influence of moisture on thermal and mechanical properties of polymer composites, such as by Yilmaz and Sinmazcelik [3] for glass–fiber/polyetherimide laminates. The laminates underwent hydrothermal aging at two distinct temperatures and elevated moisture levels. They analyzed the characteristics of as-received and hydrothermally aged materials. The results indicated that the hydrothermally aged laminates retained significant moisture, leading to a reduction in the glass transition temperature and a decline in mechanical properties. Nayak and Ray [4] delivered an

interesting study on the effect of nano-Al2O3 filler concentration on moisture absorption kinetics, residual mechanical and thermal properties of hydrothermally treated glass fiber-based nanocomposites. The incorporation of 0.1 wt% of nano-Al2O3 into the nanocomposites decreased the moisture diffusion coefficient by 10%, enhanced the flexural residual strength by 16%, and increased the interlaminar residual shear strength by 17% relative to the neat composites. Nonetheless, the incorporation of nano-Al2O3 filler did not enhance the glass transition temperature. Gibhart et al. [5] experimentally determined the impact of salt water on the fatigue behavior of glass fiber-reinforced polymers. The results showed a significant decrease in fatigue life for saturated specimens and the formation of debonding and cracks in the fiber–matrix interphase in samples with no applied mechanical loading. Later, Zhu et al. [6] investigated the effect of a hydrothermal environment on mechanical properties and the electrical response behavior of continuous carbon fiber/epoxy composites produced by a pultrusion method. Their results indicated that the bending strength decreased quickly within 3 days of hydrothermal treatment, followed by a stable trend. Moreover, a fracture surface analysis indicated that the interfacial properties of carbon fibers in the epoxy matrix also deteriorated, and more carbon fibers could be pulled out from the composite material. Recently, Cheng and Cao [7] investigated the changes in mechanical properties of glass fiber-reinforced plastic cross-ply laminates as a result of hydrothermal aging. The composite specimens were immersed in distilled water at 25°C, 40°C, and 70°C for a period of 60 days. They found that after aging, the tensile, compressive, and bending strengths of the composite decreased by 31.7%, 12.9%, and 27.0%, respectively.

The assessment of the mechanical behavior of CFRTC materials when exposed to elevated temperatures for a long period of time and severe environmental conditions is still an ongoing field of investigation that requires more testing and analyses. Therefore, the present work aims to study the mechanical resistance of unidirectional glass fiber-reinforced thermoplastic composite (UGFTC) tapes at an elevated temperature under different aging conditions. The aging conditions utilized for this study are thermally aged samples as well as hydrothermally aged samples in distilled and deionized water for up to 4 weeks. As UGFTC materials are used as a reinforcement element in thermoplastic composite pipes, the selected temperature for the aging time is 95°C because it is consistent with the range of operational temperatures for these types of structures in actual field conditions in the oil and gas industry. This study considers two types of thermoplastic matrices: polypropylene and polyethylene. Mechanical resistance was assessed for the given aging conditions, considering a strength reduction greater than 25% during the aging time as severe. In addition, water uptake behavior was also analyzed by measuring the weight of the samples during the aging period of up to 4 weeks, with the aim of assessing whether UGFTC samples are subject by mass changes. After the aging time was completed, the weight measurements were analyzed to investigate mass changes of the samples. An optical microscope was used to compare and observe the damage of the UGFTC samples for some of the aging conditions. The novelty and contribution of this study lie in enhancing the comprehension of the damage effects on the fiber-matrix interphase, the polymer matrix and the glass fibers, as these elements primarily determine the mechanical performance of UGFTC materials.

II. MATERIALS AND METHODS

A. Materials

In this study, two commercially available glass fiber tapes with polyethylene matrix (GF-PE) and polypropylene matrix (GF-PP) were used for the experimental tests because of their low thickness, which allows for accelerate testing and thus serves to further the understanding of water uptake behavior and material behavior employing comparatively short aging times. Some basic parameters of the GF-PE (UD-002, Qingdao Trusmax New Material Co. Ltd., Shandong, China) are the following: Thickness is 0.35 mm and width is 50 mm. The parameters of the GF-PP (GPP62-1050, Jiangsu QIYI Technology Co. Ltd., Jiangsu, China) are as follows: Thickness of 0.5 mm and width of 49 mm. The fiber weight fractions of both thermoplastic tapes were confirmed by ash content tests [8, 9] and were determined to be within the manufacturers' specifications of 60 ± 5%.

B. Aging conditions

The aging conditions involved different environments and an elevated temperature that is interesting for scientific purposes and industrial processes alike. Water has been identified as the most common and also rather severe aging media. The target temperature is 95°C, while the aging environments are deionized (DI) and distilled water (DW), as well as pure thermal aging. The aging conditions lasted 1 to 4 weeks. The selected temperature correspond to a broad set of applications in the industry, where the maximum temperature exposure is typically limited by the polymer constituents (such as by their melting). The present authors considered a loss in properties even by only 10 to 30% as significant. Samples will be placed inside glass jars with the different aging fluids in an oven as shown in Fig 1.

Figure 1: Samples inside glass jars for aging at 95°C in DW and DI water.

C. *Mechanical properties testing*

Tensile testing UGFTC tapes was conducted in air at room temperature using the universal testing machine (type 810, MTS Systems, Eden Prairie, MN, USA) with a 100 kN load cell over a gauge length of 150 mm by prescribing a stroke rate of 5.0 mm/min, following ASTM D3039 [10]. At least five specimens were tested for each aging duration. To prevent damaging the specimens at the grips, sandpaper, tabs, and aluminum tape were applied to the specimen extremities, and uniform gripping pressure was maintained, ensuring that failure occurred within the gauge length of the specimens.

D. *Mass change measurement during and after aging*

Mass change measurements for UGFTC tapes were conducted according to ASTM D5229M-20 [11]. A balance with an accuracy of 0.001 g was employed for weight gain or reduction measurement, in which five identical specimens were tested for each aging condition in DI water and DW at different temperatures. Before aging, the specimens were placed into an oven to remove any moisture. During the aging phase, specimens were periodically taken from of the aging fluid, dried, and weighed. The weight change of each specimen was also determined after the aging time was completed to study the moisture desorption process. The mass gain or reduction due to exposure to DI and DW water was determined using Eq.(1) [12]. Since samples varied slightly in size, it was necessary to normalize the weight change of each sample based on its initial condition.

$$W = \frac{w_t - w_i}{w_i} \tag{1}$$

where W is relative mass gain (or loss) per sample, w_i is the initial weight of the sample and w_t is the weight of the aged sample at time t.

III. RESULTS AND DISCUSSION

A. *Water uptake behavior*

Figures 2 and 3 depict the water absorption behavior over the aging time at 95°C for the GF-PE and GF-PP tapes, respectively. Note the error bars in these and subsequent figures indicate one standard deviation from the mean. In the initial treatment stage, water absorption by both samples in both aging fluids increased rapidly with time, then gradually slowing to reach asymptotic behavior after a certain period indicating saturation. In Fig. 2, the GF-PE tape reached a saturated moisture absorption state in DI water in approximately 168 hours, with a weight gain of 0.63%. In contrast, saturation in DW occurred more rapidly, in about 48 hours, with a gain of 0.2%. It is interesting to notice that the weight gain of the GF-PE tape in DI water was about 3.15 times higher than that of GF-PE in DW, while the saturation time for GF-PE in DI water was about 3.5 times greater than that of GF-PE in DW. On the other hand, the GF-PP tape reached its saturated moisture absorption in DI water with a weight gain of 2.38% in about 336 hours, see Fig. 3, while it reached saturation in DW with a weight gain of 0.53% in 240 hrs. Therefore, the weight gain of the GF-PP tape in DI water was about 4.4 times higher than that of GF-PP tape in DW, while its saturation time in DI water was about 1.4 times higher than in DW. From the data obtained during the tests it is hypothesized that mass gains are related to diffusion of water molecules into micro-gaps between the polymer chains.

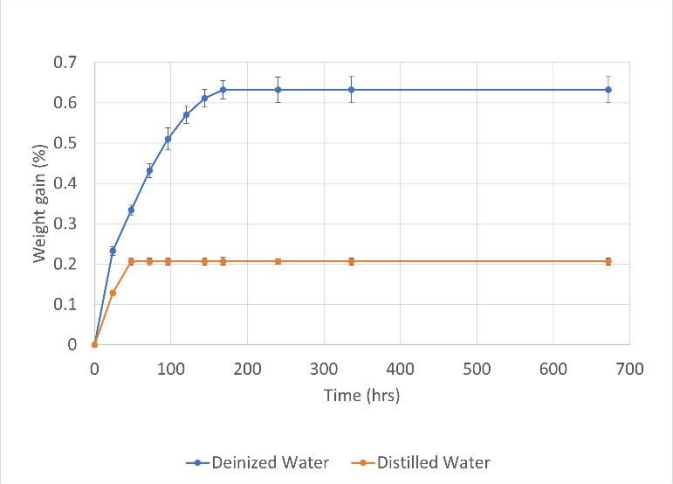

Figure 2: Percent weight gain versus aging time for GF-PE composite tapes in deionized and distilled water.

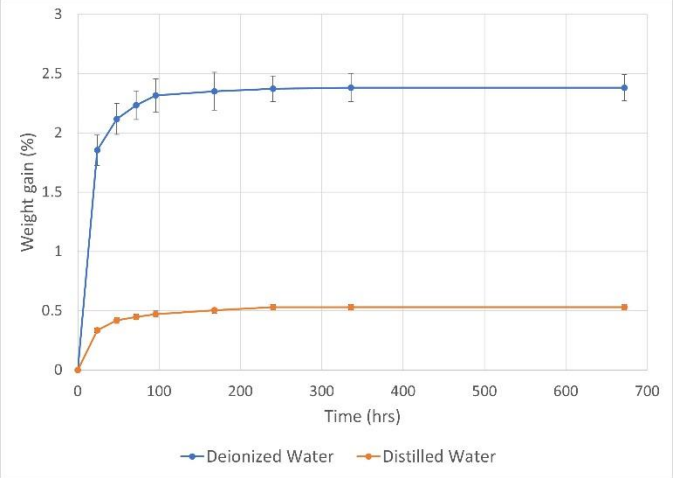

Figure 3: Percent weight gain versus aging time for GF-PP composite tapes in deionized and distilled water.

Figs. 4 and 5 illustrate the weight reduction over time post-aging for the GF-PE and GF-PP tapes, respectively. In Fig. 4, the GF-PE material immersed in DI water returned to its original weight after about 48 hours of post-aging time. For DW exposure, this process took only 24 hours. So, the process for samples to desorb water after expose and return to their initial weight was as twice long in DI water than in DW. Referring to

Fig. 5, the GF-PP material immersed in DI water returned to the original weight after 72 hours of post-aging time, while it returned to its original weight after 24 hours of post-aging time in DW. Notably, for both materials the post-aging time for samples to return to their original weight was higher in DI water than in DW. This observation suggests that aging in DI water may result in more severe damage in both materials compared to DW. This supposition is explored in the subsequent section which examines the strength retention of GF-PE and GF-PP composite tapes, considering both aging fluids at 95°C and purely thermally aged samples, which serves to assess the impact of moisture exposure at temperature. In this context, one should consider that moisture ingress into composite materials occurs primarily by permeation via the matrix and transport through microcracks which are often formed already during material processing [13].

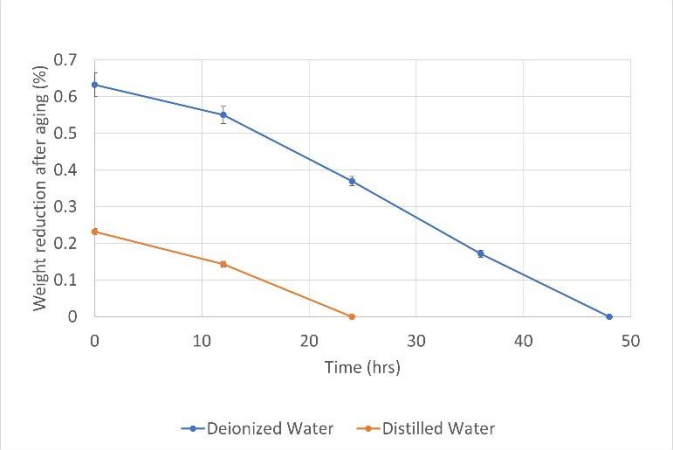

Figure 4: Percent weight reduction after aging versus post-aging time for GF-PE composite tapes. Aging fluids: deionized and distilled water.

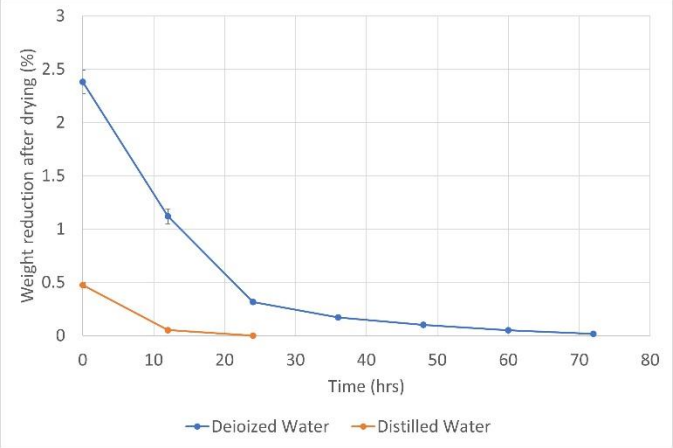

Figure 5: Percent weight reduction after aging versus post-aging time for GF-PP composite tapes. Aging fluids: deionized and distilled water.

B. Strength retention assessment

The longitudinal strength retention of the GF-PE and GF-PP composite tapes were studied after hydrothermal aging at 95 °C in DI and DW and after pure thermal aging at the same temperature. The machinal failure of these composite materials in the macroscopic level is particularly pronounced when the binding strength between the fibers and their polymer matrix is low. As the matrix fractures, the separation and detachment of the fibers from the matrix. The fibers experience increasing stress and eventually break individually or in groups as they resist the applied load and contribute to a gradual rupture. Figures 6 and 7 show the strength retention of the GF-PE and GF-PP composite tapes after 1 and 4 weeks of hydrothermal aging at 95°C in DI and DW, and after thermal aging at 95°C, respectively. In Fig. 6, the strength retention of GP-PE composite tapes decreased by 16.5% after 1 week of thermal aging and 20.8% and 42.6% after hydrothermal aging in DW and DI water, respectively. Recall that in the present work, a loss of 25% of the strength retention is considered severe for the composite materials. Therefore, after 1 week of aging in DI water, the GF-PE composite tape samples presented a severe loss in strength. These reductions in strength increased with aging duration for all conditions, i.e., after 4 weeks at 95°C, the samples showed a decrease in strength by 18.8% for thermal aging and a significant strength loss of 49% and 62.6% for aging in DW and DI water, respectively. It is interesting to note that the exposure to heat alone also affected the performance of the GF-PE composite tapes.

Assuming that aging in the different fluids and temperature are distinct phenomena, Table 1 discerns the strength reduction as separate effects for each aging condition and duration, based on the data presented in Figs. 6 and 7, for GF-PE and GF-PP composite tapes, respectively. If strength retention is analyzed under the notion of compounding degradation actions, the retention of GF-PE composite tapes after 1 week of aging decreased by 16.5% due to heat, 4.3% due to DW and 30.2% due to DI water exposure. In the case of an aging duration of 4 weeks, the strength is reduced by 20.8% due to heat, 26.1% due to DW and 43.8% due to DI water. In Fig. 7, the strength retention of GF-PP composite tapes decreased by a mere 0.1% after 1 week of thermal aging and 16.4% and 27% after hydrothermal aging in DW and DI water, respectively. Notably, the strength retention of the GF-PP composite tapes in thermal aging is considerably higher than that of the GF-PE composite tapes. In the case of a longer aging duration, GF-PP composite tapes showed a strength reduction of 8.5% in thermal aging and 31.3% and 42.9% in hydrothermal aging in DW and DI water, respectively. Considering the strength retention of GF-PP composite tape to be distinct phenomena of heat and fluid exposure, as shown in Table I, the effect of aging in DW represented 16.4% out of 16.5% reduction (including the effect of heat after 1 week) of aging and 22.8% out of 31.3% reduction (including the effect of heat after 4 weeks of aging). Clearly, the thermal effect during aging impacts GF-PE composite tapes to a greater extent than the GF-PP composite tapes. In addition, in hydrothermal aging, DI water is more aggressive than DW for both materials as an agent fluid. The strength retention related to hydrothermal aging in DI water for GF-PE composite tapes is around 1.5 times lower than that of GF-PE composite tapes in DW. In contrast, for GF-PP composite tapes, it is around 1.2

times lower. The technical literature is limited for research on the behavior of GF-PE and GF-PP composite tapes to DW and DI water exposed at elevated temperatures, and further investigation is warranted with other experimental approaches. Current testing demonstrates that at 95°C, GF-PP specimens exhibit superior heat and water resistance relative to GF-PE specimens.

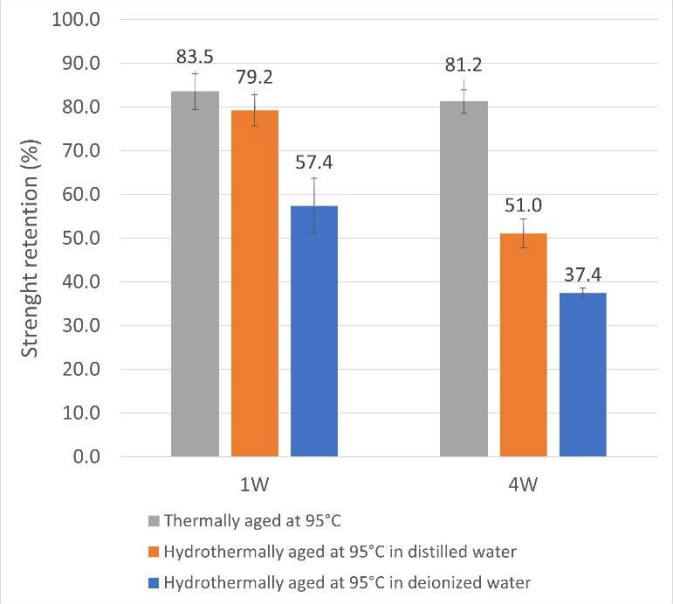

Figure 6: Variation of tensile strength at 95°C under different aging conditions of GF-PE composite tapes: thermally aged samples, and hydrothermally aged samples in distilled and deionized water.

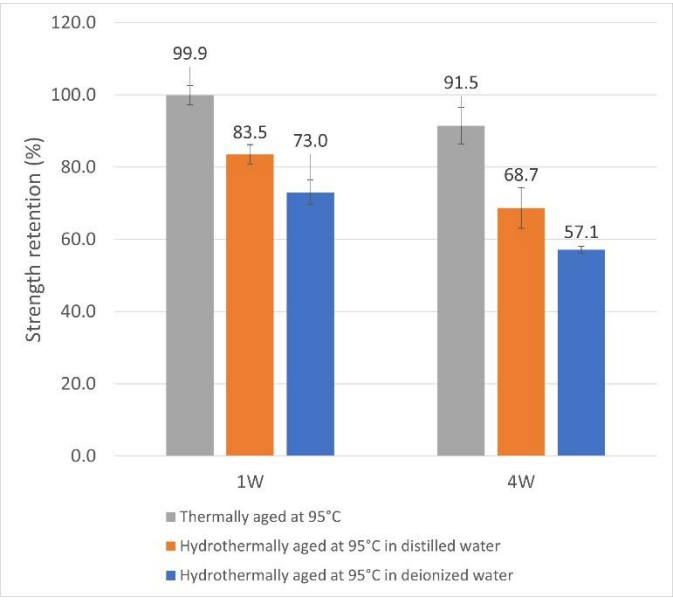

Figure 7: Variation of tensile strength at 95°C under different aging conditions of GF-PP composite tapes: thermally aged samples, and hydrothermally aged samples in distilled and deionized water.

TABLE I. PERCENT STRENGTH REDUCTION ASSUMING DISTINCT AGING EFFECTS DUE TO HEAT, DW AND DI WATER EXPOSURE AT 95°C OF GF-PE AND GF-PP COMPOSITE TAPES.

	1 week	**4 weeks**
Effect of heat in aging of GF-PE composite tape	16.5%	18.8%
Effect of DW in aging of GF-PE composite tape	4.3% out of 20.8% reduction	30.2% out of 49% reduction
Effect of DI water in aging GF-PE composite tape	26.1% out of 42.6% reduction	43.8% out of 62.6% reduction
Effect of heat in aging of GF-PP composite tape	0.1%	8.5%
Effect of DW in aging of GF-PP composite tape	16.4% out of 16.5% reduction	22.8% out of 31.3% reduction
Effect of DI water in aging of GF-PP composite tape	26.9% out of 27% drop	34.4% out of 42.9% reduction

C. Optical microscopy analysis

Degradation effects were further studied using pristine and aged samples by observing the damage on the surfaces of the aged samples using optical microscopy, with the intent to better understand how the exposure to the aging fluids affected the UGFTC tapes more than pure thermal aging. Figure 8 shows images of the surfaces of pristine and hydrothermally aged samples of GF-PE composite tape. The hydrothermally aged sample shown in Fig. 8(B) in the result of 4 weeks of aging in DI water at 95°C. Damage due to the ingress of DI water in the polymer matrix can be appreciated on the sample surface. With a continuous increase in moisture content, associated the weight gains for the given fluid and aging temperature condition, the composite appeared to have entered a stage of rapid degradation, which can be observed in Fig. 6 for the GF-PE composite tape having a strength retention of merely 37.4% after 4 weeks of aging. One of the most common damage mechanisms that can occur because of the water ingress in the composite is the deterioration of the fiber-matrix interface, which is pivotal in the aging process of composite materials. The water interaction of the fiber-matrix interface directly affects the load transmission and load sharing between the fibers and polymer matrix material, resulting in a reduction in the mechanical performance of the UGFTC material, such as the strength properties. However, a detailed analysis of the surface morphology of the samples might be done using scanning electron microscopy in future work.

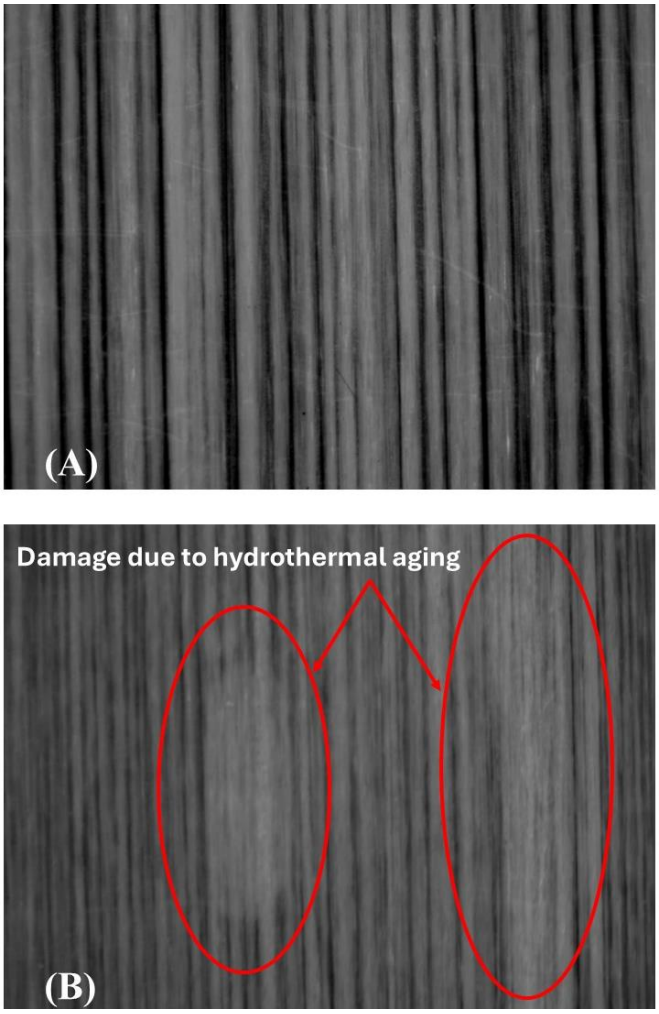

Figure 8: (A) Pristine and (B) hydrothermally aged samples of GF-PE composite tapes. The aging time of the sample was 4 weeks in DI water.

IV. CONCLUSION

This study examined the thermal and hydrothermal aging of GF-PE and GF-PP composite tapes subjected to 95°C in deionized and distilled water. GF-PP composite tape demonstrated greater water absorption in distilled and deionized water relative to GF-PE composites. In both materials, the weight gain was higher in DI water than in DW. The weight gain in DI water after 4 weeks for the GF-PP composite tapes was 2.38%, while for GF-PE composite tapes was 0.63%. In the case of aging after 4 weeks in DW, the weight gain was 0.53% for the GF-PP composite tapes, while for the GF-PE composite tapes was 0.2%. After hydrothermal aging, both the GF-PE and GF-PP composite tapes exhibited a considerable loss in tensile strength, with samples aged in DI water showing the highest reductions. The GF-PE samples lost 62.6% of their tensile strength after 4 weeks of aging in DI water, while the GF-PP samples lost 42.9% in the same aging duration in DI water. Considering the impact of heat and fluid exposure as distinct effects, degradation due to fluid aging decreased strength to a greater extent for prolonged aging durations for both composites. The outcomes of the present work provide a context for a better understanding of the mechanical performance of unidirectional glass fiber-reinforced thermoplastic composites during and after aging in different aging fluids at elevated temperatures. Moreover, exposing these materials to hydrothermal aging at elevated temperatures is shown to be a critical aspect that demands further investigation to reveal the mechanisms of composite degradation over time and thus provide the theoretical foundation for predictive methods for these composite materials. Hence, further research in this field is required.

ACKNOWLEDGMENT

The authors would like to gratefully acknowledge the support of the machine shop at the University of Alberta, which provided the facility for conducting the experimental tests.

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