11th Canadian-International Conference on Composites 2019

Canadian Institute for Composite Structures and Materials

UNDERSTANDING THE NATURAL AGING OF GFRP COMPOSITES: A LONGITUDINAL STUDY

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ABSTRACT

In this article, the degradation processes induced by natural ageing have been investigated for different sets of glass fibre/polyester composite material. Test samples, varying in their processing factors, were aged and monitored for one year, in a natural semi-arid climatic condition. Using the Principal Component Analysis (PCA), the climatic agents were then correlated to the resultant changes in the material properties. The most destructive climatic agents were also identified for samples, along with the most influential processing variables, modifying their weathering performance. The X-ray microtomography scanning was finally performed to internally inspect the aged samples and validate the mechanisms involved in their degradation.

Keywords: *Glass Fibre Reinforced Plastics, Natural Ageing, Multivariate Analysis*

1 INTRODUCTION

The application of the glass fibre-reinforced polymer (GFRP) composites continues to be widespread in the industry, mostly because of their considerable strength-to-weight and stiffness-to-weight ratios [1, 2]. Their long-term durability, however, is still a major concern, especially under harsh and inconsistent environmental conditions [3]. As a case in point, the weathering-induced failure of GFRP composite turbine blades has been repeatedly reported in recent years, under the extreme climatic condition at offshore wind farms, comprising wet/dry periods, high/ low-temperature cycles, UV radiation exposure, and even sand erosion [2, 4]. The combined effect of these climatic agents, besides the impact of reciprocating fatigue loading, is deemed to be crucially affecting the long-term performance of GFRP composites. Long-term exposure to UV radiation may cause photo-degradation/ photo-oxidation [4], resulting in chain scission [5] and affecting both the visual [6] and structural [7] performance of GFRP composites. Prolonged exposure to moist and humid environments may also degrade mechanical properties [8] through different phenomena, such as matrix plasticization [9], weakened bonding at fibre-matrix interfaces [10, 11, 12], and hydrolysis reactions [13, 14]. The weathering performance of GFRP composites is also dependent upon several design and processing factors, such as fibre volume fraction [15], orientation of fibres [16], architecture of reinforcements [15], permeability of fibres and matrix [21], polarity of the matrix molecular structure [22], the initial curing condition [8], and the application of surface coating [6].

Although the individual impact of each climatic agent has been carefully studied in the past, the combined interaction of these natural climatic agents has remained inveterately under-investigated [17]. Manufacturers are therefore compelled to overdesign their products in the absence of a reliable life cycle assessment, leading to the higher weight of parts and loss of the resources [15]. This study aims to fill the above gap in the natural weathering of GFRP composites and obtain a better understanding of the involved degradation processes. To this end, different sets of glass/polyester composite plates were fabricated and employed to evaluate the degradation processes induced by natural weathering of GFRPs. Samples varying in their reinforcement architecture, surface coating application, and initial curing state were naturally aged for one year in a semi-arid climate in Kelowna, BC. To carefully study the root causes of degradation, not only were the natural degrading agents monitored during the ageing period, but also properties, such as surface hardness, surface roughness, flexural strength, and density were seasonally measured for test samples. Using the Principal Component Analysis (PCA) as an exploratory multivariate data analysis tool, the correlations were determined between the climatic agents and their resultant changes in the material properties. Finally, the internal structure of aged samples was inspected to validate the ageing-induced degradation mechanisms.

2 EXPERIMENTAL WORK

2.1 Sample Preparation

Test specimens used in our experiments were fabricated with three major variations in their constituents and processing conditions, including fibre preform architecture (continuous E-glass plain weave $400\ g/m^2$ or randomly chopped E-glass fibre mat $800\ g/m^2$), coating application (either treated with Polycor® isophthalic white gel coat on the surface or without this treatment), and the initial curing state (either cured in ambient lab condition or at elevated temperature). All samples were made of EnvirezTM unsaturated polyester resin with methyl ethyl ketone peroxide as an initiator in 1.25% of resin weight. Along with unreinforced neat resin test specimens, reinforced samples were provided at approximately 35% and 40% of fibre volume fraction, respectively for the randomly oriented chopped fibre mats and continuous plain weave laminates.

All samples were fabricated through a conventional open mould hand lay-up process, with laminate stacks consisting of 6 and 25 plies, for the chopped fibre mats and woven fabric laminates. Long pieces of samples were cured on a flat sheet of SAE 304 stainless steel as tooling, under two different processing scenarios: either in ambient lab condition or at elevated temperature in an industrial oven. Ambiently cured sheets were cured slowly in a controlled environment at $21℃$ and 33% of relative humidity for approximately one day, whereas others were cured swiftly in an oven at $50℃$ for a minimum of 6 hours. The initial degree of cure (DOC) was estimated using RAVEN™ (a cure modelling software) as roughly 84% and 96% for ambiently and in-oven cured samples, respectively. Using an abrasive waterjet cutting Omega 2652 JetMachining Centre, all sheets were cut into their final dimensions ($13\ mm \times 215\ mm$), which was in accordance with the ASTM D7264/D7264M standard for 3-point bending test (3PB test) on polymer-based matrix composites. All samples were about $5mm$ thick with approximately $0.84mm$ of thickness for the protective layer of gel coat in coated samples. Prior to the outdoor environmental exposure, trimmed specimens were finally sealed on their edges using acrylic latex-based resin to restrain the immediate moisture ingression, and hence more accurately simulate the large and flat composite sheets in practice [5].

2.2 Experimental Procedure

As shown in Figure 1, samples were placed on top of an unobstructed roof and were installed on an adjustable aluminium rack with their flat mould side facing upwards. The natural ageing experiment started in February 2014 (winter) in this study and took for one year. All plates were oriented towards the sun during the ageing period with a 45° of inclination to maximise the absorption of UV sunlight and prevent any potential pooling of water. To avoid any contamination from the rack structure, samples were manually fixed by clamps made of ultra-high molecular weight polyethylene, as shown in Figure 2.

To monitor climatic agents during the ageing period, Campbell Scientific® weather station was also incorporated in our experiment, which was equipped with a CR800 data logger, besides several sensors, including relative humidity (RH) and temperature probe, broadband UV radiometer, and TE525 tipping bucket rain gauge. Along with the climatic agents, select material properties, namely surface roughness, surface hardness, flexural strength, and density were measured during the ageing period. The first set of measurements was performed immediately after the fabrication of samples, followed by other sets of mechanical tests respectively at 30, 90, 180, 270, and 360 days after exposure.

Figure 1: The set-up used for the natural ageing of test specimens

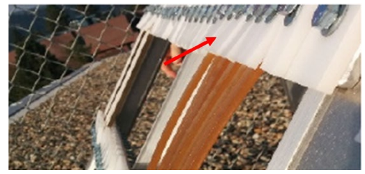

Figure 2: Clamps made of ultra-high molecular weight polyethylene

All mechanical tests were performed in a controlled lab condition, at 21℃ and 33% of relative humidity (RH), as follows. Surface roughness was measured based on its average value (R_a) for each sample. Ten points were selected randomly in the midsection of each sample and the average value of roughness was recorded. These measurements were carried out using a portable roughness tester QualitestTM TR110 and in accordance with its manual. The Shore-D hardness values were similarly measured, while with three measurements per each sample. Hardness tests were performed using the Stationary QualitestTM HPE-II Durometer, conforming to ASTM D2240-05 standard.

Unlike the two formerly discussed measurements, three-point bending (3PB) tests were only limited to reinforced samples. These tests were performed under ASTM D7264/D7264M-07 standard, using the Instron 5966 Dual Column Testing Machine. Two tests were taken at each monitoring stage, with the crosshead speed of $10\ mm/min$ and preloading of $35N$. Preloading was deemed necessary to stablise samples, owing to the uneven surface of the open mould side of laminates. After the 3PB tests, one section of the destroyed samples (roughly 50% of its initial mass), was used to measure its density, using a QualitestTM MDS-300 densimeter.

3 RESULTS AND DISCUSSIONS

3.1 Changes in the Material Properties over the Ageing Period

In this section, the weathering-induced changes in the material properties are investigated. Figures 3, 5, and 6 graphically show these changes for all types of test specimens, either gel coated (GC) or uncoated (NG). Measurements corresponding to samples with chopped mat (C) and woven fabric (W) reinforcements are respectively marked by red and blue points in these figures, along with the measurements for the pure resin (R) samples which are depicted in grey points.

As shown in Figure 3-a, the application of surface coating dramatically limited the weathering-induced shifts in surface roughness during our experiment. Thus, aligned with the results in [6], surface quality can be effectively preserved by applying a thin protective layer of gel coat. After 180 days of natural ageing (roughly mid-summer), both types of uncoated fibre reinforced samples were of higher roughness value compared to their unreinforced pure resin counterparts (Figure 3-b). The inferior surface quality of uncoated reinforced samples may be justified by photo-degradation, material leaching, and fibre blooming. This ageing-induced rise in surface roughness is found to be even more dramatic for uncoated GFRPs with chopped mat reinforcements (Figure 3-b). Primarily, the lower fibre content in this type leaves the matrix highly prone to photo-degradation [15, 18, 19]; then, seasonal precipitation and its resultant flow of water can lead to leaching of the degraded resin chains and exposure of fibres. Surface degradation and the resultant fibre blooming is clearly shown in Figure 4 for an uncoated chopped reinforced sample, after 270 days (roughly in mid-autumn) of its natural weathering.

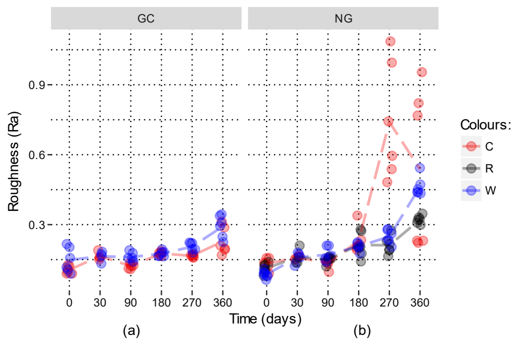

Figure 3: Changes in surface roughness for (a) gel coated and (b) un-coated samples during the ageing period; the dashed lines represent the trends for the average value, while the points depict each measurement

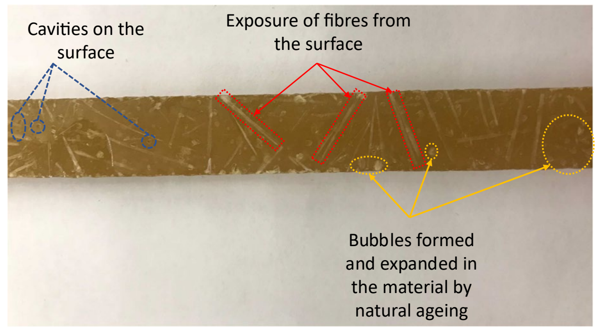

Figure 4: Weathering-induced defects in a chopped reinforced sample, after 270 days of natural ageing

Indeed, fibres may contribute to the preservation of the material performance in fibre reinforced composites [18], as they hold the resin bulk [15] and hamper leaching of the polymeric chains. Fibres also retard the penetration of UV rays to the internal layers [19] and accordingly hinder photo-degradation. The higher fibre content in composites, therefore, means better retention of properties during natural exposure.

The effect of gel coat in maintaining the surface quality is also detectable through changes in hardness, as the gel coat film similarly limited shifts in hardness values during the exposure period (Figure 5-a). For all uncoated samples, hardness was mostly decreasing during the humid winter, with a relatively steeper drop experienced by woven types (between the days 180 and 270 in Figure 5-b).

The phenomenon known as 'matrix plasticisation' chiefly leads to a decline in hardness values [9]. Penetration of water molecules between polymeric resin chains enhances chains mobility, resulting in a softer structure of the matrix [13, 20]. This plasticisation is typically known as the dominant degradation process among those that matrix may undergo [2, 21], whose effects are deemed to be recoverable after desorption [2, 5]. Signs of such recoverability can also be noticed in Figure 5-a and b under our natural weathering experiment.

In composites with continuous fibres, moisture sorption is through the combined action of diffusion in matrix and capillarity along fibres (either diffusion through fibres or cracks at fibre-matrix interface region) [2, 4, 22]. These types of composites are proven to be more diffusive along their fibres rather than through their matrix structure [7, 23]. In these types, hydrolysis reaction contributes to the weakening of fibre-matrix bonds [20, 14]. Then, differential swelling stresses [24, 8, 11], together with the dissolution of fibres sizing in the absorbed water [9, 25], ultimately leads to interfacial debonding, the known prevalent damage among aged composites with woven fabric reinforcements [2, 22]. The additional free volume in woven types [2, 26, 27, 20, 28], imposed by this interfacial debonding [29], means their more potential for moisture ingression, and thus, the sharper reduction of hardness during the wet and humid seasons (between the days 180 and 270 in Figure 5-b).

All types of samples were generally gaining hardness during the sunny season of the ageing period (between the days 30 and 180 in Figure 5-b). This hardness gain would be representative of post-curing and polymerisation of un-reacted monomers under the action of sunlight UV radiation [30, 7, 31, 32]. Indeed, before the outset of photo-degradation, the two latterly discussed phenomena (polymerisation and post-cure crosslinking) lead to the chains with a higher molecular weight and stronger intermolecular bonds, ultimately resulting in a stiffer matrix structure.

Changes induced by UV radiation are also known to be dominantly limited to the irradiated surface [18] and more likely affect the measured hardness values. The irradiated surface protects its underlying internal layers from UV radiation and invasion of oxygen [4]. As a result, applying a layer of coating film, as a frontline barrier against UV, can effectively limit the ageing-induced shifts in hardness (Figure 5-a). Given the softer structure of the applied gel coat compared to the matrix, coated samples, however, were generally of lower value in their hardness than their uncoated counterparts during this experiment.

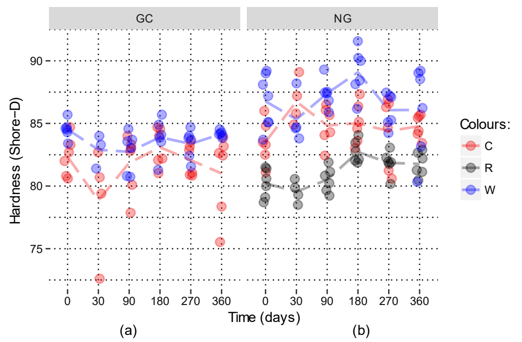

Figure 5: Changes in surface hardness for (a) coated and (b) un-coated samples during the ageing period; the dashed lines represent the trends for the average values, while the points depict each measurement

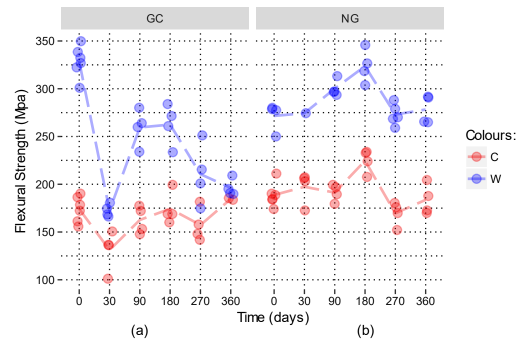

Figure 6: Changes in flexural strength for (a) coated and (b) un-coated samples during the ageing period; the dashed lines represent the trends for the average values, while the points depict each measurement

Similar to the weathering-induced changes in hardness values, the rise in the flexural strength (Figure 6) was under the action of sunlight UV radiation (post-curing and polymerisation effect) in our experiment, while its drop (Figure 6) was vastly under the influence of moisture ingression (plasticisation and affected interfacial bonds). For coated samples, the decline in the flexural strength, experienced within the first 30 days of ageing (Figure 6-a), was probably due to debonding of the coating layer from the resin bulk, shown in Figure 7. The additional interface imported by the coating application, and its subsequent debonding, may inflict the uptake of destructive agents, like moisture and oxygen, leading to the degradation of constituents [33]. This decline was found to be even sharper for coated samples with continuous fibres (Figure 6-a), where the debonding also followed the fibre path (depicted by red arrows in Figure 7).

Uncoated fibre reinforced samples, however, were all declining in their flexural strength quite similarly during the wet season and regardless of their fibre preform architecture (see Figure 6-b in the interval between the days 180 and 270). Using a micro-CT tomography inspection, two distinct mechanisms are found here to degrade the flexural properties in these two types, but with quite similar impacts. The weakened fibre-matrix bonds (Figure 8), mainly caused by moisture absorption, lead to the decline in flexural strength for composites with continuous fibres [2, 22], while for chopped fibre reinforced GFRPs, matrix degradation (ageing-induced defects in the matrix, like bubbles and microcracks, as shown in Figure 9) is known as the chief cause of this reduction [34].

Indeed, photo-degradation leaves the material with lower density on its outer layers, as UV mostly affects the irradiated surface. The resultant density gradient in the composite induces residual stress in the material structure [35, 36], leading to the formation of microcracks. Diurnal temperature variation is another climatic factor known for the initiation of microcracks [37]. As fibres and matrix differ in their thermal expansion rates, daily variation in temperature results in internal residual stress and ultimately in the generation of cracks. UV radiation may also increase the void content in the composite structure and degrade its flexural properties. Bubbles, shown in Figure 9, may form and expand in the material structure as a by-product from chemical reactions and photo-degradation [18].

The lower fibre content in chopped samples suggests a higher rate of matrix degradation, a higher density of microcracks, and more frequency of bubbles in the material, compared to their woven counterparts. Fibres, firstly, contribute to holding the resin chunk [15] and hindering the initiation of cracks. Fibres also retard UV radiation and make the composite more resistant against photo-degradation. Composites reinforced with woven fabrics are therefore deemed to more effectively preserve their matrix properties.

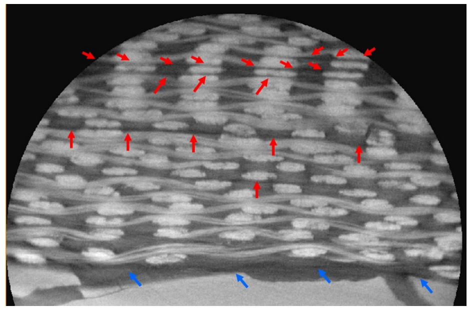

Figure 7: Micro-CT image from a coated sample with woven fabric, after 30 days of natural ageing; red arrows represent fibre-matrix interfacial debonding (cracks along fibre bundles); blue arrows denote debonding of the coating film from the material bulk.

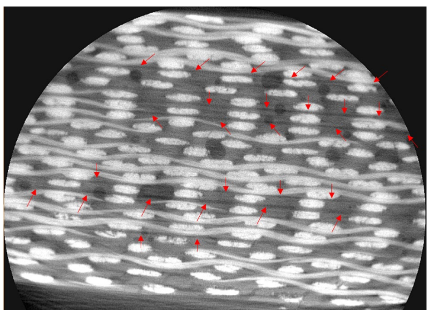

Figure 8: Micro-CT inspection of an uncoated woven sample, after one year of natural ageing

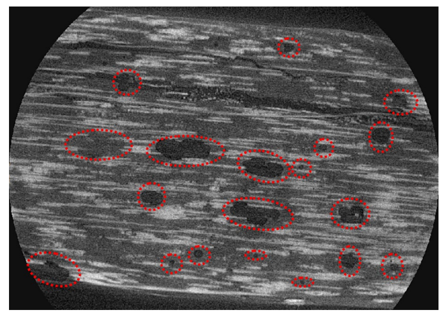

Figure 9: The Internal structure of an un-coated chopped sample, after one year of its natural ageing

3.2 Principal Component Analysis (PCA)

To extract correlations existing between the climatic agents and changes in the material properties, PCA is applied to the collected data as an exploratory multivariate data analysis tool. This method is also helpful to visualise how processing factors may affect the weathering performance of GFRP products. It is based on the projection of data to a reduced space, whose Principal Components (PCs) are defined as a linear combination of original variables, at directions along which the variation of data is maximised [38]. To facilitate the visual interpretation of correlations, the analysis is usually limited to the first two principal components (PC1 and PC2). Each observation is projected onto the reduced plane and displayed as a point (score) in its respective colour (based on its category), along with the variables represented by vectors in the score-loading plot figures (Figures 10 and 11). Observations located close to a vector in a score-loading plot are known to be more significantly affected by that variable. Also, the cosine of the angle between two variables (their angular closeness) is denoting the correlation existing between them.

The select design and processing (controlled) variables are firstly analysed here, and their effect on the weathering performance of the tested GFRP composites is found. The analysis reveals that by shifting from the chopped mat to woven fabric as reinforcement, flexural strength is increased in the product (see the positive correlation between "Reinforcement" and "Flexural" axes in Figure 10). The higher fibre content in woven types implies their higher density and superiority in their flexural properties. The effect of the protective coating film in limiting the surface degradation is also discernible in Figure 10, where the "Coating" axis is negatively correlated with both "Roughness" and "Hardness" variables. The gel coat film, which is inherently resistant against UV radiation, can block the UV rays and prevent their penetration to the main bulk of the material; thus, hindering post-curing and retarding the matrix degradation. The initial curing state, however, is found here to present minor effects on the weathering performance of GFRP composites, compared to the two formerly discussed design factors (see the "Cure" axis in Figure 10 and its negligible length). As discussed in Section 2.1, the Degree of Cure (DOC) is estimated to be in close ranges for both curing scenarios in this study (96% and 84% respectively for samples cured in an oven and those cured ambiently), resulting in similar weathering patterns for both cases.

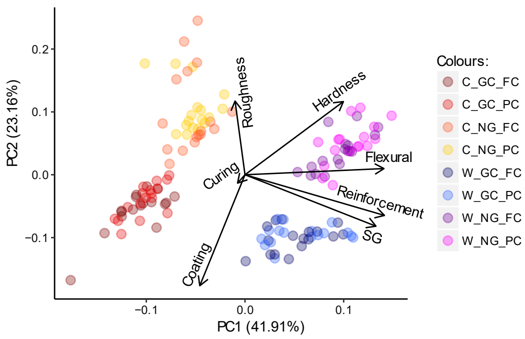

Figure 10: Effects of different design and processing variables on the natural weathering of GFRPs

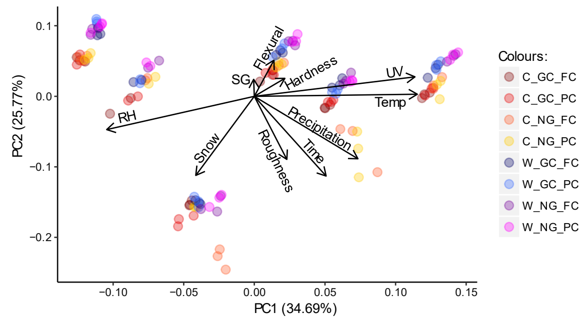

Figure 11: Effect of climatic agents on the performance of aged samples

Climatic agents are also analysed in this study to explore their effects on the weathering performance of the tested GFRP samples. The effect of exposure time is included in the analysis as well, to see how the interaction of these agents may affect GFRP composites during their long-term outdoor exposure. Climatic agents included in the analysis are as follows: (1) UV Radiation (The scale of UV Index), (2) Environmental Temperature (℃), (3) Precipitation Level (mm), (4) The Depth of Snow Cover (cm), (5) Relative Humidity ($RH\%$), respectively named as “UV”, “Temp”, “Precipitation”, “Snow”, and “RH” in Figure 11. Vectors labelled as “Time” and “SG” refer to Ageing Time (days) and Sample Density (gr/cm^3) (Specific Gravity), respectively.

Congestion of observations around the “UV”, “Temp”, “RH”, and “Snow” axes in Figure 11 indicates the more significant effect of these variables on the weathering of GFRPs, rather than the precipitation level. Observations accumulated around the “Precipitation” axis are mainly related to uncoated chopped reinforced categories, signifying the higher impact of seasonal precipitation on this type. Uncoated chopped reinforced samples are also found to deviate from their other counterparts at some ageing stages. This deviation, chiefly on the roughness axis, is denoting the crucial effect of gel coat in this type to achieve a better surface quality.

The direct correlation of roughness with both ageing time and precipitation level in Figure 11 means the inferior surface quality after long-term outdoor exposure and under seasonal precipitation. Exposure to UV radiation leads to photo-degradation and accordingly high density of broken chains on samples surfaces. Then, seasonal precipitations and their resultant flow take part in degraded chains leaching, and accordingly fibres exposure. Effects of both post-curing and polymerisation are also implicitly discernible in Figure 4-7 by the close and positive correlation of mechanical properties (hardness and flexural strength) with both UV radiation and environmental temperature. Among these two agents and aligned with [7], UV is found to have a higher correlation with rises in mechanical properties, revealing the higher effect of UV radiation on post-curing rather than temperature increase. As a clue to moisture ingression side-effects, such as matrix plasticisation and weakened interfacial bonds (discussed in Section 3.1), “RH” and “Snow”, resulting in the absorption of water respectively in its gas and liquid phase, are reversely correlated with mechanical properties (Figure 11). Snow immersion mainly degrades the flexural strength, while “RH” is notably involved in the loss of surface hardness. The more significant impact of snow immersion on flexural strength is presumably indicating the increased risk of decayed interfacial bonds when the absorbed water is in its liquid phase. On the other hand, penetration of water molecules in gaseous status mostly facilitates the mobility of polymeric chains and therefore broadly leads to matrix plasticisation.

Overall, both UV radiation and moisture ingression have mitigating effects on each other [15]. UV radiation energy is dissipated through the removal of absorbed moisture or its evaporation, making saturated composites less prone to photo-degradation. Further, in the exposure of UV radiation and before the outset of photo-degradation, the irradiated surface starts to shrink (crosslinking or post-curing effect). As the inner layers of a composite laminate are protected from UV rays, this shrinkage is hindered by unexposed layers. The resultant internal (residual) compressive stress promotes fibre-matrix bonding and also alleviates swelling of the matrix. Therefore, UV radiation makes up for repercussions of moisture ingression.

4 CONCLUSIONS

Polymer-based composites are known to degrade over time, and their long-term outdoor durability has remained a challenging subject in both academia and industry. In this research, a comprehensive experimental-statistical analysis was performed on different sets of polyester-based GFRP samples. The weathering-induced changes in the material properties were monitored for one year, and the degradation processes involved in natural ageing were obtained for each type. To systematically analyse the effect of long-term outdoor exposure, PCA was adopted as an exploratory data analysis technique, and the relative contribution of each climatic agent was effectively determined. The select samples were inspected at the end, both visually and structurally, to validate several degradation processes involved in natural ageing.

Long-term UV radiation broadly resulted in matrix photo-degradation, expansion of bubbles, and formation of microcracks among samples, although the short-term radiation can lead to post-curing and hardening of the matrix structure. Soaking agents, namely Relative humidity and Snow Cover, were found to degrade the internal structure of GFRPs, leading to a decline in mechanical properties. Among the studied design and processing variables, the reinforcement architecture was determined as the most influential parameter for the weathering performance of GFRP products, followed by the application of a coating film and the initial curing condition. A better weathering performance was generally observed among samples with plain weave continuous fibres, compared to their chopped fibre reinforced counterparts. The higher fibre content in the woven type samples may reduce the risk of photo-degradation, although the decayed bonding at fibre-matrix interfaces may still be a serious concern for this reinforcement architecture.

Overall, the PCA methodology employed in this longitudinal study was found to be constructive in systematically quantifying and visualising the correlations between the climatic agents and changes in the material properties. The influence that a particular variable may exert on each category of observations was also effectively identified by means of sub-PCA analyses on subset of data. In other terms, PCA presented not only a detailed overview of correlations, but also its visualisation capabilities helped us to gain a better insight into the causes and effects of degradation.

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