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Water Exposure, Tensile and Fatigue Properties of Treated Hemp Reinforced Vinyl Ester Composites

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Abstract. Recently, due to the safety issue, the incorporation of fire retardant in natural fibre composites is becoming necessary. Nevertheless, since it is potentially utilised as low load bearing material, less work has been done on the fatigue properties and the effect of water to its mechanical properties. In this work, untreated and fire retardant treated (FR) composites made of woven hemp fabric (WHF) were investigated for their tensile and fatigue properties. Tensile properties of fabricated composites were studied with an addition of water absorption factors. Both composites were immersed in the water for up to 2688 hrs and tensile properties were analysed periodically. Based on the results, tensile properties of both fabricated composites are reduced after being immersed in the water for 2688 hrs. Comparing both samples, the FR treated composites exhibited lower tensile properties than the untreated. In terms of fatigue test, each sample was tested at 50, 60, 70 and 80% stress levels in tension-tension mode for both composites. It was observed that, as the number of fatigue cycles increased, the fatigue strengths of both composites gradually decreased. Their fatigue strength coefficient was also similar (0.12) indicates that the treatment did not affect the fatigue strength of composites. The safety limits for these fabricated composites to be utilised for any application is about 24 to 30 MPa.

INTRODUCTION

Natural fibre reinforced composites has high potential to be used in construction or infrastructure and also automotive industries at least as the non-structural parts [1-4]. Looking at the targeted applications, safety issues especially in their ability to inhibit fire have become a priority for these materials to remain relevant to be used. Therefore, there is a lot of work has been done to enhance the fire retardant properties of this kind of material [5-7].

However, the most critical issue for natural fibre composites that being emphasised by researchers is moisture or water absorption. Water affects the properties of composite materials by infiltrating natural fibre composite and reducing the adhesion among the composite elements. Hemp fibre is hydrophilic, that is, it absorbs moisture and water [8-10]. Similar to other natural fibre composites, hemp fibre composites are expected to absorb moisture and water, degrading its properties. Therefore, it is important to understand the effects of water absorption on the mechanical properties as well as on the fire retardant treatment applied to the WHFs in the composite.

Another major concern with bio-based composites is their long-term behaviour when exposed to continuous loading as well as the prediction of lifetimes. The long-term behaviour of material will define its durability and serviceability. Consensus from the literature is that very little fatigue work has been carried out on natural fibre composites [2, 11-13]. Thus, there still lack of knowledge about the fatigue behaviour of treated hemp fabric composites, especially on the effect of fire retardant on the fatigue behaviour of woven hemp fabric composites.

In this work, untreated and fire retardant (FR) treated WHF were used to reinforced vinyl ester resin (HVE-UT and HVE-FR). Both HVEs have gone through water absorption testing in accordance with the nominal standard

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method. Water absorption testing was selected rather than moisture exposure to study the degradation of composite properties at the worst scenario, thus the measured degradation of HVE properties can be considered to be an extreme level. After more than 2500 hrs water immersion, samples were taken out and subjected to tensile test. Another test that has been carried out on the fabricated samples was fatigue test. The test was carried out in different stress level to investigate fabricated samples' S-N diagrams, fatigue life, fatigue strength coefficient and fatigue degradation. The objectives of this work are to determine the tensile properties of HVEs affected by water penetration and durability degradation of HVE affected by the continuous loading.

Materials

Woven hemp fabric (WHF) was supplied by Hemp Wholesale Australia. According to the specifications given by the supplier, the fabrics were produced with 100% yarn hemp in both warp and weft. Vinyl ester resin, SPV 1356 PROM THIX and the catalyst methyl ethyl ketone peroxide (MEKP), NOROX 925H were supplied by Nuplex® Composite Industry (Australia). Flame retardant (FR) chemical was supplied by Cyndan Chemicals, Australia. According to the supplier, the main active ingredient in this flame retardant is ammonium polyphosphate.

Chemical Treatment

The WHF was treated with the FR chemical. In this work, the 'dips and nips' method was employed to treat the fabrics. The nipping process was set carefully so that the wet pick-up was consistently maintained in the range of 100-105%. The treated fabric was then left to dry at room temperature for eight hours.

Composite Fabrication

The resin was prepared by adding MEKP into the vinyl ester at the ratio of 1:44 by weight. This prepared resin was then applied to 10 fabric layers (300 × 300 mm for each layer) by employing the hand lay-up technique. The fabrics were layered in warp and weft alternately ($[0,90]_5$). The mixture (wet fabrics) was then laid between thick glass plates (400 × 400 × 100 mm in dimension) which were coated with a polymer mould release agent. This assembly was compressed with a weight placed on top of the mixture to remove the excess resin and the calculated pressure given to this assembly was 4.360 kPa. It was then left to cure at room temperature for 24 hours. After the 24 hours, post cured in an oven for four hours at 80°C. Two types of composite were fabricated using untreated WHF (HVE-UT) and FR treated WHF(HVE-FR).

Water Absorption Test

The effect of water absorption on the HVEs was investigated in accordance with BS EN ISO 62:1999 [14]. The samples were cut into dimensions similar to tensile testing. The samples were immersed in a distilled water bath at 23°C. The water content for each specimen was determined by measuring the mass, m, of each specimen periodically until saturation was reached. When the specimens were removed from the water bath for measurement (sample weighing), all surface water was removed with a clean dry cloth. Upon completion of mass measurements, the specimens were returned to the water bath.

Measurements were completed more frequently (every 24 hours for the first five days) at the beginning of the test because of the initial high rate of change of mass. The percent of water uptake, *M*, was calculated for each mass measurement as follows:

$$M = \frac{m_i - m_d}{m_d} \times 100 \qquad (1)$$

where *m* is the mass of the specimen, the subscript *i* refers the *i*th measurement, and the subscript d refers to the dry state prior to water immersion.

The percentage weight gain of the samples was measured at different time intervals and the moisture content versus the square root of time was plotted [15]. Samples were taken for mechanical test after 2688 hrs of water immersion. Thickness of sample was also measured until the water immersion process was completed. This determines the dimension of stability of the HVE when exposed to the water.

In order to analyse the different water absorption behaviour of HVE-UT and HVE-FR, the diffusion of water into the composite samples was measured by using the diffusion coefficient (D) which is commonly described by Fick's law [14-17].

Tensile Test

Tensile test was performed on a universal testing machine MTS Alliance RT/10. The tensile properties were characterised in accordance to ASTM D638. Ten specimens with the dimension of 250 × 25 × 5 mm3 were cut from the fabricated samples. The tensile load was applied at a constant displacement rate of 2 mm/min. A laser extensometer was used to measure the axial strain.

Fatigue Test

Fatigue testing was performed in accordance with BS ISO 13003:2003 (Fiber-Reinforced Plastics-Determination of Fatigue Properties Under Cyclic Loading Conditions). They were performed using pneumatic fatigue machines at a frequency of 3 Hz and a stress ratio (R value) of 0.1 for tension–tension fatigue. The choice of frequency ensured that the heating effect due to hysteresis was minimal. The R value of 0.1 in tension–tension was chosen to maximize the cyclic effects without invoking the complications of compressive stresses and the likely variations in failure mechanisms. Three specimens with dimensions similar to tensile specimens were tested to failure at a minimum of four levels of maximum stress (80, 70, 60 and 50%). The maximum stresses during cyclic loading were recorded as stress level of fatigue. The number of cycles to failure were recorded for each specimen, and these data were plotted in the form of S-N (Wohler) curves [18]. After plotting S-N diagrams, the power-law regression equation was determined for each material to obtain the fatigue strength coefficient.

RESULTS AND DISCUSSION

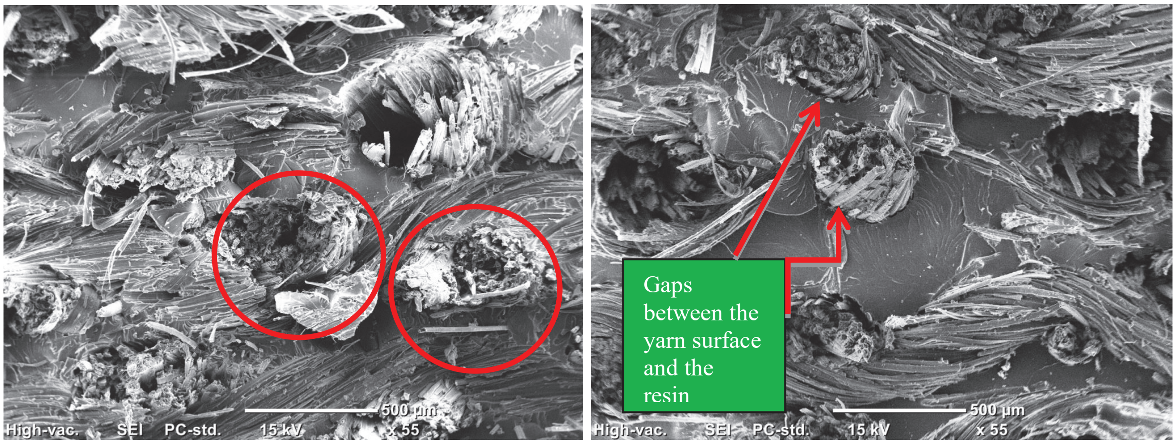
Water Absorption

TABLE 1 shows the water absorption properties for both samples. Both samples were immersed in the water exceeding their saturation points and the test stopped exactly after 2688 hrs. Sample HVE-UT reached it saturation point after 1848 hr immersion while sample HVE-FR reached it saturation point earlier than HVE-UT which was after 552 hr. Nevertheless, the maximum water uptake at saturation point for sample HVE-UT was recorded higher than sample HVE-FR which were 3.43% and 3.28% respectively. The higher value of diffusion coefficient (D) for sample HVE-FR than sample HVE-UT indicates that it has faster water uptake or diffusion into the sample. This reflects how quick the HVE-FR sample to reach the saturation time in comparison with sample HVE-UT.

Water flow rate at the edge (at the cut or thickness part) is higher than that through the faces because fibres are exposed more at the edge (Christian and Billington [16]). This explains why the water diffusion of HVE-FR is faster and higher than the HVE-UT since it possessed higher thickness. Another factor that could lead to higher water uptake properties is the compatibility between the treated WHF and vinyl ester that gives higher water diffusion of HVE-FR. **FIGURE 1** shows scanning electron microscope images of the fracture surface for each sample. As shown in **FIGURE 1**(a) yarns and vinyl ester have slight gap, indicating that the adhesion between the yarns and vinyl ester is good. However, for sample HVE-FR, several gaps appear, indicating the incompatibility in between the yarn surface and the vinyl ester (**FIGURE 1**(b)). This is due to the ammonium polyphosphate content in the FR chemical which affects the adhesion in the composites [19, 20]. Therefore, with respect to water absorption, the water molecules are not only diffused via the fibre surfaces, but also permeated via the gaps between the fibres and resin. This is the other reason of faster water diffusion and higher diffusion coefficient of sample HVE-FR in comparison with HVE-UT.

TABLE 1. Water absorption properties of HVE-UT and HVE-FR samples

Sample	Sample Thickness (mm)	Time to saturate (hr)	Maximum water uptake, Mm (%)	Diffusion Coefficient, D (mm^2/s)
HVE-UT	5.17	1848	3.43	1.45 E-06
HVE-FR	5.36	552	3.28	4.71 E-06



(a) (b)

FIGURE 1. SEM micrographs of tensile fracture surface; (a) HVE-UT and (b) HVE-FR

Tensile Properties

FIGURE 2 and **TABLE 2** shows the typical curve of stress-strain responses and mechanical properties results for both samples at 0 and 2688 hr water immersion, respectively. Generally, specimens immersed in the water at 2688 hr immersion showed lower peak but longer strain than samples without immersion. Comparing both types of samples, sample HVE-UT possessed higher tensile strength and strain than sample HVE-FR. The typical failure mode can be seen from the FIGURE 3 for both samples, with ruptured protruding yarn as well as pulled-out yarn from the surface indicating that, as the yarn pulled-out from the matrix at the failure surface, the fibres within the yarn ruptured.

Generally, the tensile properties of HVE-FR are lower in comparison to HVE-UT (**TABLE 2**). The loss of sample HVE-UT was only about 21% of the original strength by the end of the immersion time (2688 hrs). **FIGURE 3**(a) shows the fracture images for HVE-UT at 0 hr water immersion showing short ruptured protruding yarns as well as pull out fibres and HVE-FR fracture sample was not so different from this. **FIGURE 3**(b) shows the fractured sample for HVE-UT after 2688 hrs immersion which showing longer ruptured protruding yarns suggesting that the water weakened the adhesion between fibres and resin [14, 15].

Strength reduction for the sample HVE-FR was recorded (**TABLE 2**) even higher after water immersion which is 24%. Observing sample HVE-FR after 2688 hr (**FIGURE 3**(c)) water immersion specimen, we can see not only the ruptured and pulled-out yarns but also a lot of pulled-out yarns perpendicular to the loading directions (either warp or weft yarn) [16, 17]. This is due to the deposition of ammonium polyphosphate that leads to poor adhesion between the hemp fibres and vinyl ester resin [19, 20] on top of the diffusion of water [14-16].

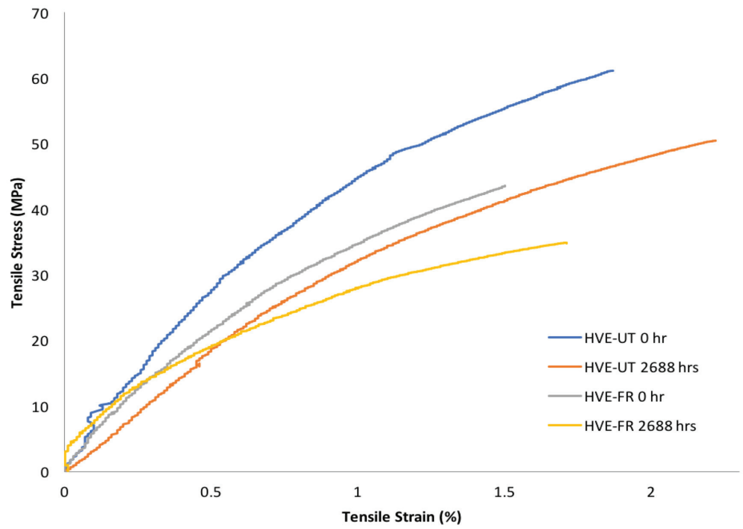


FIGURE 2. Typical tensile stress-strain curves for sample HVE-UT and HVE-FR.

TABLE 2. Results of tensile properties of sample HVE-UT and HVE-FR with respect to water immersion times

Tensile Properties	**Time (Hrs)**	
HVE-UT	0	2688
Tensile Strength	60.897 (0.933)	48.341 (1.756)
Tensile Strain	1.78 (0.283)	1.932 (0.203)
Tensile Modulus	6.073 (0.803)	3.8432 (0.745)
HVE-FR		
Tensile Strength	46.479 (2.930)	35.191 (1.63)
Tensile Strain	1.643 (0.130)	1.844 (0.270)
Tensile Modulus	4.690 (0.742)	3.378 (0.367)

Tensile strain for sample HVE-UT and HVE-FR increased with the increase of immersion time and the increment was up to 9 and 12% after 2688 hr immersion respectively. In terms of tensile modulus, both samples show a similar trend of the tensile strength with the reduction of up to 27%. The declining in mechanical properties of hemp composite after water immersion were also being experienced by other workers [14-16]. The increase in strain and the reduction in tensile modulus suggest that the samples became soft and ductile due to the plasticisation by water. Therefore, the composite material stiffness are reduced as plasticisers infiltrate between the polymer chains and push the chains apart, effectively lowering the glass transition temperature for the resin making it softer [16]. Water also plasticised the hemp fibres using a similar mechanism [14-16].

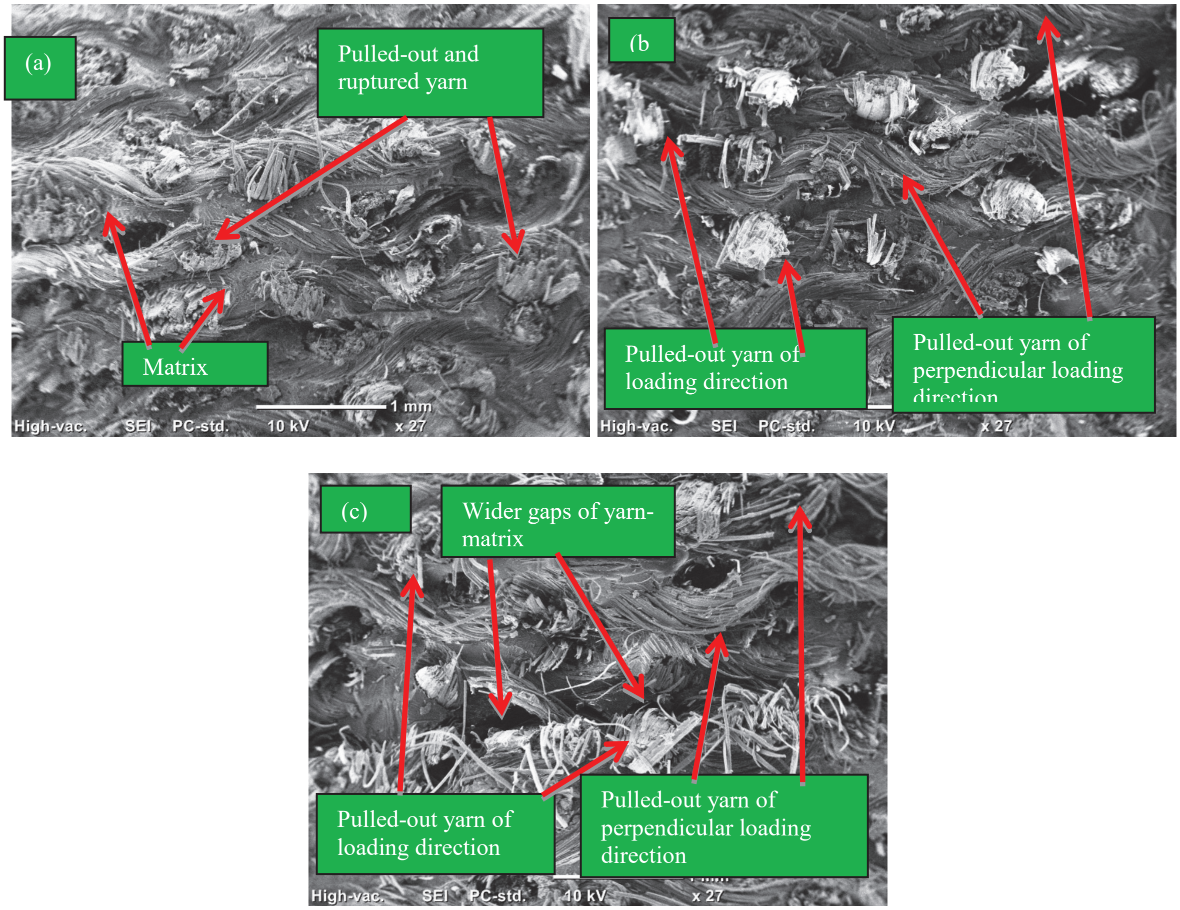


FIGURE 3. Scanning electron image of sample; (a) 0 hr water immersion for HVE-UT, (b) 840 hr water immersion for HVE-UT and (c) 2688 hr water immersion for HVE-FR

Fatigue Properties

TABLE 3 shows the combinations of fatigue testing parameters (% stress level, S_{max} and S_{min}) for both samples. The results of fatigue tests are presented by plotting Wohler stress-life (S-N) diagrams (such in **FIGURE 4** and **FIGURE 5**). The fatigue strength coefficient (b) is a very useful parameter to determine fatigue degradation [11]. The higher b values infer slower degradation of fatigue strength for every decade of cycles [11, 12, 18]. It was obtained from the Power–law regression equations and given by:

$$S_{max} = S_0 N^b \tag{2}$$

where S_{max} is the maximum stress applied, N is the number of cycles to failure, S_0 is the single cycle (static) ultimate strength of the material, and b is the material fatigue strength coefficient.

FIGURE 4 and **FIGURE 5** show S–N and normalised S-N fatigue data for the present samples. A gradual decrement in fatigue strength with an increasing number of fatigue cycles was observed. It is observed that the power–law model of Equation [11] fits to the experimental fatigue data. All regressions have an R^2 value > 0.95 which is generally characteristic of composites whose lifetime is matrix crack growth and inter-laminar cracking domination [11, 21]. From the S–N diagram in **FIGURE 4**. **FIGURE 5**, it is observed that even though the static strength of sample HVE-UT is greater than HVE-FR, their fatigue strength coefficient can be said to be similar; 0.12 and 0.128 respectively. Thus, it can be said that the fire retardant treatment imparted on the WHF composites did not affect the fatigue strength of the materials. Since the HVE-FR was tested under low cycle with the higher stress level (50-80%), it can be projected that this sample can reach up to of at least 10^5 cycles with lower stress levels based on extrapolating the curves in **FIGURE 4** and **FIGURE 5**.

TABLE 3. Values of S_{max}/S_{min} and stress levels (% of tensile strength) for sample HVE-UT and HVE-FR

Sample	Tensile Stress (TS) (Mpa)	S_{max}/S_{min} (Mpa/Mpa)			
		0.8 TS	0.7 TS	0.6 TS	0.5 TS
HVE-UT	60.9	48.72/4.87	42.63/4.26	36.54/3.65	30.45/3.04
HVE-FR	46.48	37.18/3.72	32.54/3.25	27.89/2.79	23.24/2.32

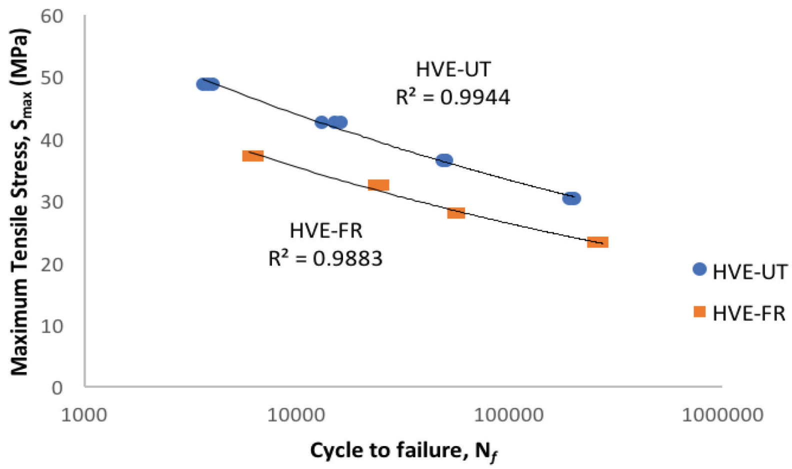


FIGURE 4. Lifetime S–N diagram for samples HVE-UT and HVE-FR.

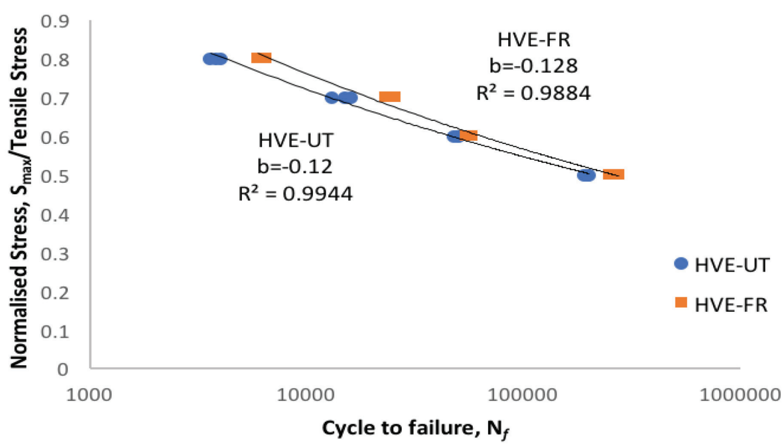


FIGURE 5. Normalised S–N diagram of HVE-UT and HVE-FR.

The reliability for safety, insurance risk, life cycle costs and high longitudinal stiffness of the material can be estimated from the low cycle fatigue [22, 23]. For this purpose, Harik, et al. [22] defined taking the low cycle fatigue at 10^4 cycles and 50% ultimate strength of the materials. Therefore, the fatigue strength of the present samples' (HVE-UT and HVE-FR) values which were tested under low cycle fatigue (at 50% tensile strength) can be used as a safety limit for the end products' application. In this work the safety limit strength utilisation for sample HVE-UT and HVE-FR are about 30 and 24MPa respectively.

CONCLUSIONS

The determination on the effect of water on the hemp fabric composite's mechanical degradation and also its fatigue properties were done in this work. In terms of water absorption properties, the maximum water uptake for both HVE-UT and HVE-FR upon the saturation point are 3.43% and 3.28% respectively. The water uptake for both samples can be said to be similar yet the time to reach saturation point for HVE-UT was far longer in comparison with HVE-FR. The diffusion coefficient for HVE-FR is far higher than HVE-UT. The faster time to reach saturation point and higher diffusion coefficient of HVE-FR are due to the incompatibility between the hemp fibre treated with FR chemical and vinyl ester resin which creates gaps between fibre and resin, thus increasing the rate of water penetration into the composites.

The tensile strength and modulus of both samples are reduced after 2688 hrs water immersion. Again, this is due to the penetration of water which weakened the adhesion between the fibres and resin. Another point worth addressing is a greater decline in tensile modulus than tensile strength is due to the plasticisation of water on the vinyl ester and hemp fibres.

In terms of fatigue strength, both samples show a similar fatigue strength coefficient which implies that the fire retardant treatment imparted on the WHF composites did not affect the fatigue strength of the materials. These samples were tested under low load cycle with higher stress level ranging from 50% up to 80%. As suggested by other work that employs low cycle fatigue (at 10^4 cycles) and 50% ultimate strength of the materials, the safety limits for sample HVE-UT and HVE-FR are defined to be 30 and 24MPa respectively. There is still room for improvement especially on enhancing the incompatibility in between treated hemp fabric and vinyl ester resin to widen its utilisation.

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