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Moisture Ingress Effect on Properties of CFRP

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SUMMARY

Standard design procedures at present for aircraft components made from CFRP typically assume ‘HOT/WET’ material properties. This assumes that the part is fully saturated with moisture and operating at elevated temperature. This represents the worse case condition for a component, as the ingress of moisture combined with a high operating temperature, significantly weakens the materials mechanical properties. As future aircraft move towards primary loaded CFRP materials it is essential that the use of such HOT/WET design allowables is investigated in order that potential weight savings can be exploited. This is a novel requirement. Thin structures are likely to reach saturation so that the worse case HOT/WET condition can be considered acceptable. For thicker components this is likely to be too conservative. The objective of this work was to generate material property data for varying levels of saturation in order to quantify the effects of moisture ingress on mechanical properties.

Keywords: mechanical properties, failure behaviour, carbon fibre-reinforced polymer, moisture, hot-wet.

1. INTRODUCTION

Carbon fibre/epoxy composites are materials used in the aeronautical industry to manufacture many different components. Composite materials in practical use can be subject to a wide variety of different loading conditions. The most important conditions are mechanical stresses and environmental attacks. To evaluate these materials it is important to know their mechanical properties and how they are affected by differing environmental conditions [1].

The standard procedure at present is to design aircraft components made from CFRP using ‘HOT/WET’ material properties. This assumes that the part is fully saturated with moisture and operating at elevated temperature. This represents the worse case condition for a component, as the ingress of moisture combined with a high operating temperature, significantly weakens the materials mechanical properties. In the past this approach could be considered to be satisfactory as previous composite components have been relatively thin (<8mm). An aircraft manufacturer considers anything less than 8mm to become saturated throughout the aircraft lifecycle. However, thicker structures, typically found on primary wingbox componentry, (wing skins, spars, pylon fitting, and landing gear attachment structure) have thicknesses up to 70mm which will in reality never reach saturation and therefore the ‘HOT/WET’ knock down can be considered too conservative.

As future aircraft move towards primary loaded CFRP materials it is essential that the use of HOT/WET design allowables are investigated in order that weight savings can be optimized. This is a new requirement, because with thin structures that will reach saturation, this worse case HOT/WET condition can be considered acceptable.

During the initial phase of this test programme, unidirectional lay-up test coupons were uniformly saturated at different percent relative humidities, RH, (the higher the %RH the greater the final equilibrium moisture content). The coupons were conditioned to full (equilibrium) saturation to evaluate the effects of moisture on the mechanical properties of CFRP. Tension, in-plane and interlaminar shear testing were carried out; the standard test methodologies, including specimen dimensions are detailed in references [2-9].

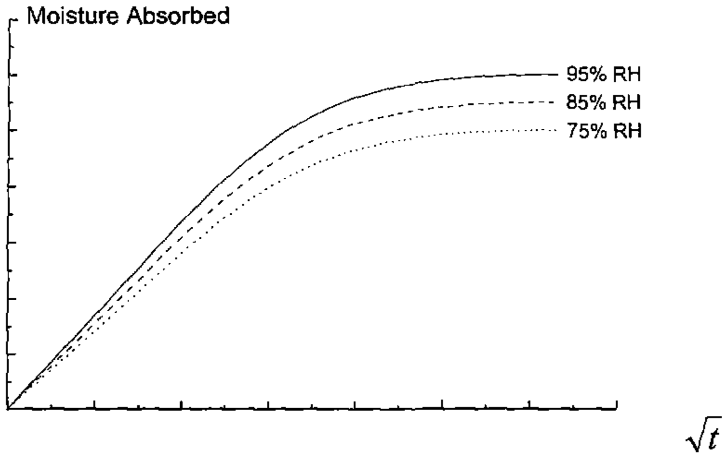

Figure 1: The effect of RH on the amount of moisture absorbed with time, t.

2. EXPERIMENTAL PROCEDURES

2.1 Materials

All materials were composites of carbon-fibres with Cytec 977-2 epoxy resin. The panels were manufactured using a hand lay-up process and autoclave cured at Airbus UK. After manufacture all panels were C-scanned to check for defects. The quality of the laminates was deemed suitable for use in aerospace applications.

The average cured per ply thicknesses was 0.184mm. The material system is presently being used by certified general aviation aircraft.

The unidirectional (UD) $[0]_6$ $[90]_{11}$ $[0]_{11}$ $[\pm 45]_{2S}$ composite panels were prepared from 977-2 HTS 12K 34% carbon pre-preg. Panel specifications are shown in table 1. First pre-preg sheets were cut from the material roll then the specified numbers of pre-preg sheets were stacked together to fabricate composite laminates. The panels were subsequently cured in an autoclave by vacuum bag moulding to obtain the laminates nominal thickness.

Table1: Laminate panel and test standard specifications.

Panel Test Method	**Standard**	**Number of Plies**	**Lay-up**
Tensile Strength and Modulus	BS EN ISO 527-5	6	$[0]_6$
Tensile Strength and Modulus	BS EN 2597	11	$[90]_{11}$
In plane Shear Strength and Modulus	BS EN ISO 14123	8	$[0/90]_{2S}$
In plane Shear Strength and Modulus	BS EN ISO 14123	8	$[0/90]_{2S}$
Interlaminar Shear Strength	BS EN ISO 14130	11	$[0]_{11}$

2.2 Preparation of test coupons

Sets of test coupons each set consisting of 6 test specimens were prepared to study the effect of moisture absorption on the mechanical properties (Young’s modulus, tensile strength in longitudinal and transverse directions, transverse compression strength, in-plane shear modulus and strength, interlaminar shear strength) of the 977-2 34% 12KHTS CFRP composites. Specimens were also prepared to study the moisture absorption behaviour of the UD composite.

2.2.1 0^o and 90^o unidirectional tension specimens

To study the effect of different levels of moisture ageing on longitudinal and transverse Young’s modulus and tensile strength of composites, tensile specimens were prepared in accordance to British Standard, BS EN ISO 527-5 [7]. Longitudinal specimens were cut to size of 250mm x 15mm x 1mm with the major length in the fibre direction from the UD panel $[0]_6$. The transverse tensile specimens were prepared from UD panel $[90]_{11}$ using coupons of dimensions 250mm x 25mm x 2mm, with the major length perpendicular to the fibre direction. Both sets of specimens were tabbed with Tufnol end tabs (Fig. 2a and b).

2.2.2 $\pm 45^o$ specimens

To study the effect of moisture on the in-plane shear strength and modulus, tensile specimens were prepared in accordance to BS EN ISO 14129 [8]. The specimen dimensions were 250mm x 25mm x 1.5mm cut from the $[\pm 45]_{2S}$ panels. Tufnol end tabs were bonded on to the coupons (Fig.2c).

2.2.3 IILSS specimens

The interlaminar shear test (short beam shear test) was performed according to BS EN ISO 14130 [9], in order to assess the effect of environmental conditioning on the interlaminar shear strength (ILSS). The dimensions of the coupon were 20mm x10 mm x 2mm (length x width x thickness).

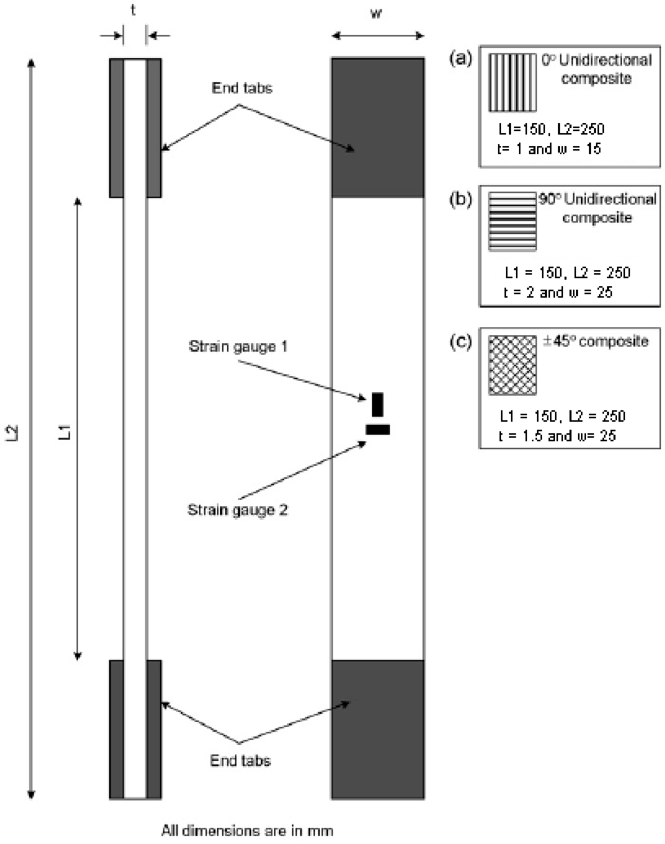

Figure 2: Schematic of CFRP laminate tensile specimen for studying mechanical properties.

2.2.4 Moisture absorption specimens

Test specimens were prepared according to procedure B of ASTM Standard D 5229-92 [10] to study the moisture ingress in the composite laminates. Five specimens of each material type UD, of dimensions 100mm x 100mm x 2mm were prepared. These specimens were used to calculate the diffusion coefficients of the material.

The detailed methods of composite laminate and test specimen preparation along with detailed account of mechanical test procedures can be found elsewhere [11].

3. TESTING

Moisture absorption specimens were dried in an oven at 80^{o}C to remove any moisture prior to conditioning in a humidity chamber at 70^{o}C and 85%RH. All other specimens were conditioned from the as-received condition. The temperature remained well below the resin glass transition temperature in order to avoid the onset of irreversible damage (swelling and cracks), which permanently changes the absorption characteristics of the material.

3.1 Determining the diffusion coefficients

Specimens were put into a humidity chamber at 70^{o}C and 85%RH and removed at regular intervals for weight measurement. The effect of this removal on weight gain determination has been shown to be negligible. Specimens were allowed to cool for a short time period before being weighed. The percentage weight gain was measured with an analytical balance having a resolution of 0.0001g. The conditioning process was continued until full saturation of the materials was achieved.

3.2 Environmental Conditioning

In order to assess the influence of the environmental conditioning on the mechanical properties, the carbon-epoxy test specimens were exposed to a combination of temperature and humidity within an environmental test conditioning chamber. The conditions selected to saturate the specimens before the mechanical tests were based on Procedure B of ASTM Standard D 5229 M-92 [10]. The moisture uptake rate in the laminate was periodically monitored as a function of time by measuring the mass of the samples until a moisture equilibrium state was reached. (Moisture equilibrium was defined when the average moisture content of the specimen changed by less than 0.05% for two consecutive readings within a span of 7 ±0.5 days.) All tests were conducted on material conditioned as follows:

- Dried (oven at 70°C)
- As-received
- Saturated at 75% RH and 70°C
- Saturated at 85%RH and 70^{o}C
- Saturated at 95% RH and 70°C

Differing levels of relative humidity give varying levels of maximum moisture content M_{max}. An example of this is shown schematically in Fig.1. Batches of six coupons were tested for each test type and condition. All mechanical tests were conducted at test temperatures of 23^{o}C.

3.3 Mechanical Testing

Coupons were tested once they had reached equilibrium moisture content. The conditioned test coupons were tested on a hydraulic Instron universal testing machine with a 100kN load cell. Flat faced hydraulic wedge grips were used for all tension test procedures and loading was applied under displacement control and rates specified in the standard test procedures. Where test fixtures were needed flat compression plates were used to sit the fixtures on and the tests were run in compression at a constant rate.

Where modulus measurements were being taken strain gauges were bonded onto the coupons prior to testing.

4. RESULTS AND DISSCUSSION

4.1 Moisture absorption

The moisture absorption of the UD CFRP laminate as a function of time is shown in Fig.3. Like any other polymers epoxies absorb moisture when exposed to humid environments. Moisture absorption takes place through a diffusion process, in which water molecules are transported from areas with higher concentration to areas with lower moisture concentration. Carbon fibres do not absorb moisture only the epoxy resin in the composite material. The laminates here the UD had a fibre volume fraction (V_f) of 65%. The diffusion coefficient, D, is a function of temperature and does not depend on the %RH so this is a constant for materials conditioned at a given temperature. The diffusion coefficient for the moisture in the UD CFRP composite, calculated from the initial slopes of the graph (Fig.3) and equation 1 proposed by Shen and Springer [12]. D was calculated to be 6.7 x 10^{-7} mm^2/s for the 977-2 UD coupons exposed to 70^oC and 85%RH.

$$D = \pi \left(\frac{h}{4M_{\max}} \right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2 - t_1}} \right)^2 \qquad (1)$$

Where M_1 and M_2, are the weight percentage moisture (wt %) taken at times t_1 and t_2 (in seconds) respectively, h is the laminate thickness, in mm and M_{max} is the equilibrium moisture level (wt %) for the given RH.

The moisture uptake of the materials displayed Fickian [12] type diffusion behaviour. In other words, each curve increases linearly to a level before the slope decreases. The transition from the linear region to full saturation occupies only a relatively small portion of the entire curve. Beyond the linear region, the slope soon drops to zero where the material is saturated. Along with experimentally measured data Fig.3 also plots a Fickian numerical fit as described by equation 2 showing excellent agreement [12].

$$M = M_{max} \left[1 - \exp\left(-7.3 \left(\frac{D.t}{h^2} \right)^{0.75} \right) \right] \qquad (2)$$

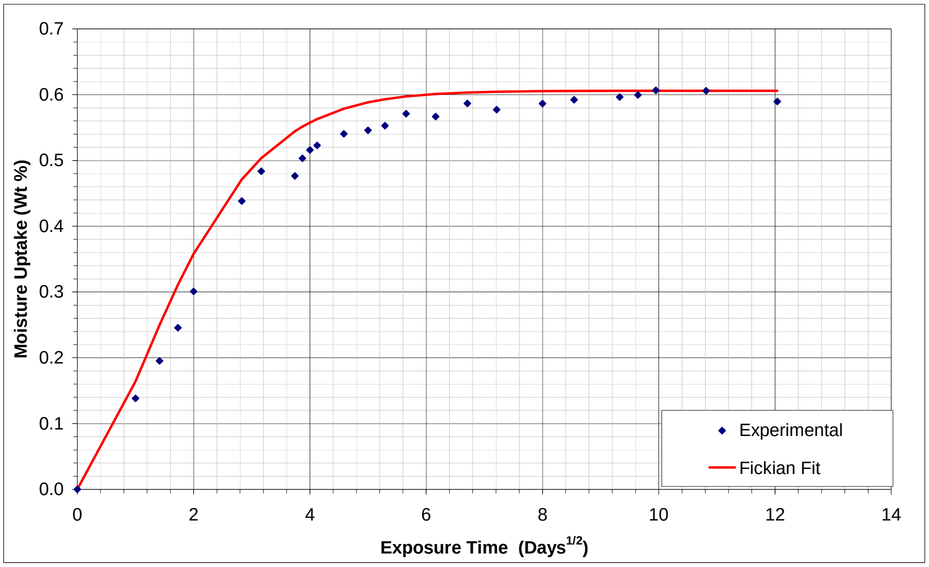

Figure 3: Moisture uptake behaviour, showing Fickian fit.

4.2 Tensile tests

To characterise the material behaviour under tension the strength and modulus were recorded during testing. In the graphs that follow the results are shown versus the moisture content.

It appeared from Fig.4 that the tensile strength was not affected by the moisture for specimens with a fibre orientation in the direction of the load. The variation of the mean values was smaller than the scatter. Thus the influence of moisture on this property was negligible. This property is fibre dominated and as fibres do not absorb moisture this result is expected.

Specimens with a fibre orientation perpendicular to the load direction showed different behaviour (Fig.5). The more the coupons absorbed moisture the lower the mean values of ultimate strength became. When subjected to the highest humidity condition the coupons absorbed 0.57wt% moisture and the transverse tensile strength decreased by almost 50% compared to the oven dry condition. This decrease in strength with increased equilibrium moisture content followed a linear relationship. This property is matrix dependent and from these initial results it can be seen that the more the tensile strength values depend on matrix and interface, the greater the influence of moisture absorption.

From figures 4 and 5 it seems that neither 0^o nor 90^o Young's modulus is affected by moisture ingress.

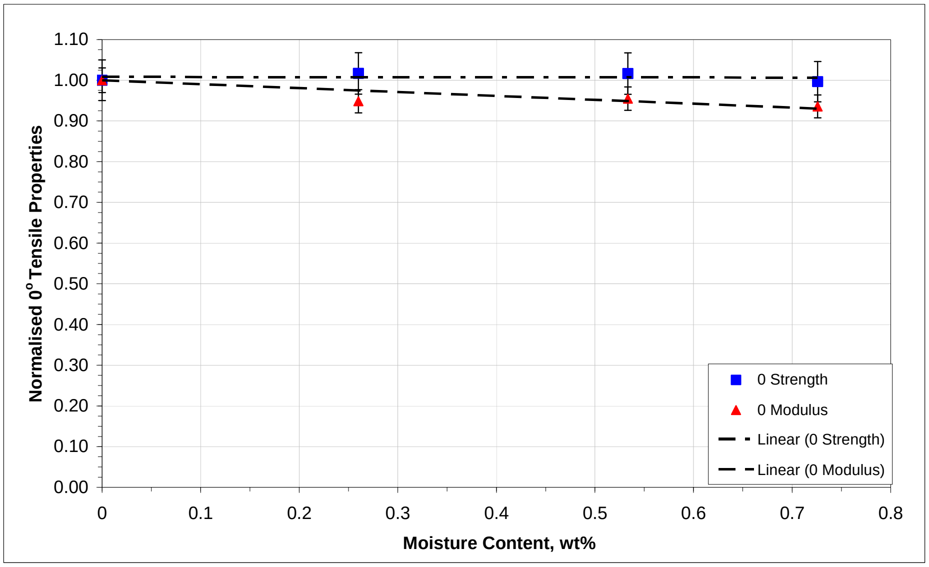

Figure 4: Effect of moisture on 0^o normalised transverse tensile strength and modulus.

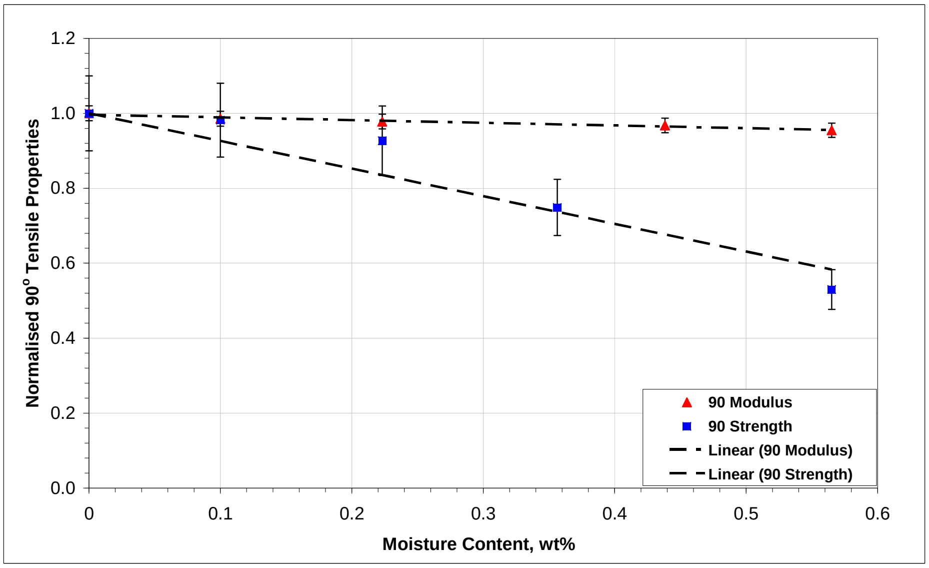

Figure 5: Effect of moisture on 90^o normalised transverse tensile strength and modulus.

4.3 Shear Properties

The ILSS was measured using the three-point bend test. The ILSS of the material increased by 10% when a small amount (0.25wt %) of moisture was present (Fig.6). On increasing the %RH to produce higher equilibrium moisture contents, the ILSS continued to decrease in a linear manner. The failed specimens showed a general shear deformation throughout the specimen thickness. The load/extension curves showed a gradual decrease in slope with increased equilibrium moisture content.

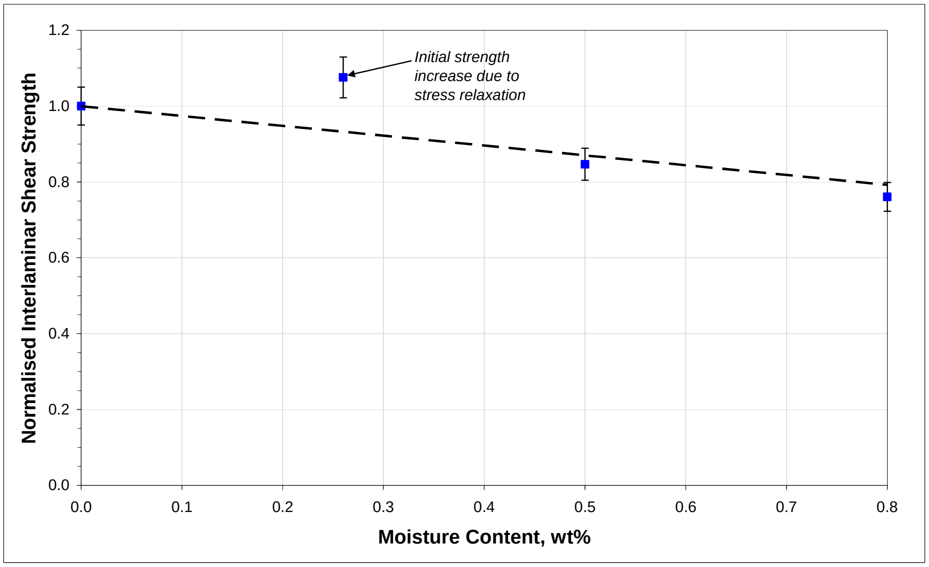

Figure 6: Effect of moisture on interlaminar shear strength.

An alternative shear test is the ±45^o shear test, where the in-plane shear properties are evaluated. A laminate constructed of ±45^o plies is subjected to tensile loading. The initial oven dry strength was measured to be 22% stronger than a coupon conditioned to equilibrium moisture content that absorbed 0.9wt% moisture as can be seen in Fig.7. In the same diagram, for the same condition the in-plane shear modulus saw a 10% reduction in value. Both properties decreased linearly with increasing equilibrium moisture content.

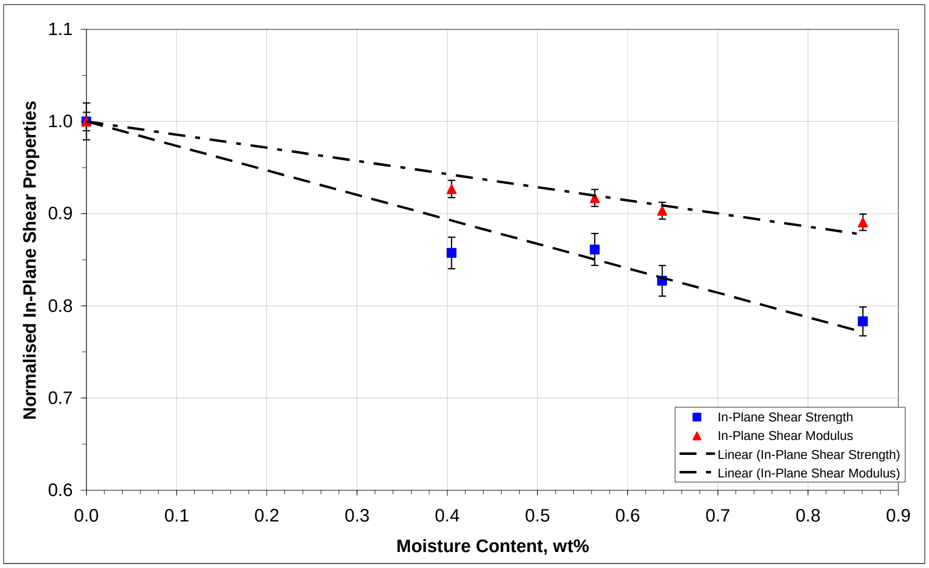

Figure 7: Effect of moisture on normalised in-plane shear strength and modulus.

5. CONCLUSIONS

This paper reports the moisture absorption behaviour and performance of unidirectional composite laminate made from 977-2-HTA 12H 34% carbon pre-preg. Moisture uptake in the material agreed well with Fick's second law. Longitudinal tensile, transverse tensile and in-plane shear properties were studied. Test coupons were prepared and tested to failure as per ISO BS test standards. The results revealed that no effect was measured in test coupons with fibres orientated in the direction of the load, regardless of equilibrium moisture content. For tensile strengths of coupons, with fibres orientated perpendicular to the axis of loading, the strength was seen to decrease linearly with increasing equilibrium moisture contents to a maximum of 50% strength reduction as compared to oven dry. In-plane shear strength and modulus both saw a decrease in strength with increasing equilibrium moisture content to a maximum 20% and 10% reduction respectively. ILSS saw an increasing-decreasing trend as maximum moisture content increased. Transverse modulus remained unaffected by moisture conditioning.

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