

Investigating the Environmental Durability of Vitrimerized Commercial Thermosets for Aerospace Composites

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Abstract

An increasingly pressing issue in composite material sustainability research has been the difficulty associated with recycling thermoset composites, commonly used in aerospace structures. This is due to permanent crosslinking between polymer chains, which inhibits epoxy resins from chemical dissolution or flow at high temperatures. Vitrimers are a polymer class investigated for their reprocessability, due to dynamic covalent bonds that can be broken and reformed with the application of stimuli like heat or moisture. However, concerns have arisen regarding the impact of environmental exposure on vitrimers. This study focuses on the influence of hygrothermal effects on the mechanical performance of a novel carbon fibre vitrimer composite, compared to a commercial thermoset benchmark. A transesterification-based vitrimer system was derived from thermoset and used to manufacture neat resin and carbon fibre composite samples. Hygrothermal conditioning was then performed in an environmental chamber until moisture equilibrium was reached. The durability after exposure was assessed using matrix-dominated mechanical testing, including neat resin tensile, composite in-plane shear, and composite short-beam shear. The results of this work indicate a greater susceptibility of the vitrimer system to hygrothermal effects.

1. Introduction

Composites have become one of the most prevalent materials used in high-performance industries, such as in aerospace, where carbon fibre-reinforced polymers (CFRPs) make up over 50% by weight of the Airbus A350 and Boeing 787 aircrafts [1]. The most commonly used polymer matrix in the aerospace industry are thermosets like epoxy resins, accounting for up to 90% of the market [2]. Waste generated by composites has caused growing concern though, as governments and global markets look to transition to a circular materials economy, with less than 5% of thermoset composites being recycled at end-of-life [3] [4]. A major contributing factor to this issue is the difficulty associated with recycling thermosets due to their permanent chemical bonds. Thermosets are generally not considered a sufficiently recyclable candidate to meet sustainability goals [5].

The covalent crosslinks formed during the curing of thermoset polymers are irreversible, preventing effective reprocessing [6]. Fibre degradation can be caused when breaking these bonds, generating considerable decreases in mechanical properties. Consequently, the majority of recycling techniques result in a product of lower value and are highly energy intensive [1] [3].

Research centred on eco-design has recently identified more sustainable alternatives to traditional thermosets that can still provide exceptional mechanical properties. Vitrimers are a promising class of polymer that has been developed for their applications as matrices in composites. They are described as sharing traits of both thermosets and thermoplastics, coupling the mechanical performance of thermosets at service temperatures with the reprocessability of thermoplastics at elevated temperatures [5]. Vitrimers possess outstanding self-healing properties and capability for reprocessing. They are covalent adaptable networks (CANs) that can participate in associative bond-exchange reactions. Their dynamic covalent bonds (DCBs) are reversible with the application of stimuli like humidity, heat, light, and chemicals [7]. Several vitrimer systems have been developed, such as transesterification, transcarbamoylation, disulfide exchange, among others [7]. Transesterification vitrimers have been of particular interest due to the ability to induce DCBs in commercially available thermosets, through the incorporation of chemical catalysts during synthesis [8]. These DCBs are specifically ester linkages that undergo transesterification, causing rearrangement of the network topology [9]. This allows for rapid stress relaxation, and thus more facile recycling and valorization [5]. Fibre-reinforced vitrimer composites are investigated for these reasons, showing environmental and economic potential with near 100% closed-loop recovery processes [10]. They have demonstrated mechanical performance retention of over 90%, observed for multiple consecutive recycles [11] [12] [13] [14].

However, advanced composite materials need to maintain performance in extreme environments for certification and use in the aerospace industry. Environmental durability is a critical factor, including moisture exposure, thermal, UV, and chemical resistances [2]. Material performance envelopes are widely used industry metrics, such as those developed by the National Institute for Aviation Research (NIAR), Wichita [15]. They display material properties as a function of temperature under different conditions (i.e. dry, wet). Room temperature dry (RTD) and wet (RTW) are property values obtained respectively after drying and conditioning to moisture equilibrium. Elevated temperature dry (ETD) and wet (ETW) properties are obtained by testing at elevated temperatures [15]. Fibre-matrix debonding followed by delamination is the main moisture damage mechanism in CFRPs, where the matrix swells causing voids and microcracks at the fibre-matrix interface [16]. Concerns have been raised regarding the combined impact of temperature and humidity on vitrimer performance [17]. Wu et al. reported a tensile strength decrease of 25% in a transesterification vitrimer, after 21 days of aging at 85°C/85% RH [18]. Luzuriaga et al. noted 30% lower tensile strength of a disulfide vitrimer compared to thermoset at 120°C, after 14 days of aging at 70°C/85% RH [6]. Cárdenas et al. observed a 22% reduction in in-plane shear strength for a disulfide vitrimer composite versus thermoset at 120°C, after the same preconditioning [19]. These existing studies have focused more on investigating vitrimer hot/wet testing behaviour. RTW behaviour simulates aircraft parts returning after temperature and humidity cycles, since it reflects the performance of materials that have undergone hygrothermal conditioning but are tested at room temperature. Thus, it can indicate if lasting damage from exposure has occurred.

Environmental effects on vitrimer composites are a novel subject, with many underexplored avenues. Benchmarking vitrimer durability is of considerable research importance since it is a necessary step in bridging the gap to feasible industrial applications. The approach outlined in this paper provides an exploration into vitrimer performance under extreme conditions. This work builds on that previously reported by Ilse and Hubert [20], utilizing the same vitrimerized commercial thermoset system. Samples for tensile, in-plane shear, and short-beam shear testing were manufactured from neat resin and composite panels. Moisture uptake curves for neat resin and composites were generated. Mechanical testing after hygrothermal exposure was conducted to obtain property degradation data comparing vitrimer to thermoset. The results of these tests reveal a higher degree of vitrimer property degradation versus the thermoset benchmark.

2. Materials and Methodology

2.1. Materials

Araldite® LY 556 / Aradur® 917 / Accelerator 960-1, an epoxy-anhydride system, was generously provided by Huntsman Advanced Materials and used as the thermoset epoxy resin system. Zinc acetylacetonate xhydrate was purchased from Ambeed Bio and utilized as the vitrimer catalyst. Non-crimp +45/-45 carbon fabric was obtained from Saertex. All materials were used as received.

2.2. Processing

All materials in this paper were produced according to procedures outlined in Ilse and Hubert [20]. Synthesis of the thermoset was performed according to the technical data sheet [21], with an epoxy:hardener:accelerator respective weight ratio of 100:90:3. Epoxy and hardener were mixed in a container for 1 min, after which the accelerator was mixed in until homogeneous. Synthesis of the vitrimer used an epoxy:hardener:accelerator:catalyst weight ratio of 100:45:3:14.2, providing 10 mol% of zinc relative to epoxy. Zinc acetylacetonate xhydrate catalyst was first mixed with the hardener until fully dissolved, then the epoxy and accelerator were mixed in. For all resins synthesized, a 15 min degas was performed at 50°C in a vacuum oven to remove any bubbles introduced. Degassed resin was poured into a 229 mm x 229 mm aluminum mould, preheated in an oven to 80°C, to cast plates to a nominal thickness of 3 mm. Cross-ply carbon fibre laminates with an 8-layer stacking sequence of $[0/90]_{2s}$ were manufactured through resin transfer moulding (RTM), to a nominal plate thickness of 2 mm and fibre volume fraction of 60%. A typical cure cycle as provided in the technical data sheet [21] was performed for all materials; a 4 hour cure at 80°C followed by a 4 hour post-cure at 140°C. To produce neat resin tensile, composite in-plane shear (IPS), and short-beam shear (SBS) mechanical testing samples, waterjet cutting was performed at the McGill University machine shop according to American Society for Testing and Materials (ASTM) D638 [22], ASTM D3518 [23], and ASTM D2344 [24] specifications.

2.3. *Moisture Conditioning*

Moisture uptake measurements were conducted as per ASTM D5229 Procedure B [25]. Samples were initially dried in a 50°C oven until moisture was effectively removed. They were then placed in an ESPEC ETT environmental chamber set to 70°C and 85% RH. These values follow a worst-case aircraft service environment [25] [26]. During the conditioning, gravimetric measurements were made using a balance once per day, with multiple readings made on the first day to characterize the initial uptake. The moisture content, $M\%$, is described by Equation (1), where W_o is the oven-dry initial sample mass and W_i is the mass of each successive reading.

$$M\% = \frac{W_i - W_o}{W_o} \times 100 \quad (1)$$

Moisture conditioning was performed until effective moisture equilibrium was reached, which is defined by an average moisture content change of less than 0.02% across two consecutive readings. The final value was used to identify the moisture equilibrium content and the time to equilibrium.

2.4. *Mechanical Testing*

Neat resin tensile, composite IPS, and SBS tests were respectively conducted according to ASTM D638 [22], ASTM D3518 [23], and ASTM D2344 [24]. Neat resin tensile and composite SBS samples were tested with an MTS Insight 5kN, while IPS samples were tested using an MTS Landmark 100kN. An extensometer was used to record displacement for tensile samples, from which strain was determined. Tensile strength and modulus were calculated, respectively, from the maximum stress before failure, and the slope of the linear stress-strain region. The IPS strength was obtained from the ultimate stress before failure. The SBS strength was determined from the maximum stress before interlaminar shear failure. A total of 5 samples per experiment per material were processed, conditioned, and tested. Samples were mechanically tested at RTD and RTW, performed at standard ambient lab conditions. Two intermediate testing points before full moisture equilibrium of SBS samples were taken, in addition to RTD and RTW conditions.

3. **Results and Discussion**

3.1. *Moisture Conditioning*

Neat resin tensile and composite IPS moisture conditioning curves are presented, respectively, in Figure 1 and Figure 2. While the thermoset neat resin achieves equilibrium after 5 days to a moisture equilibrium content of 1.1%, the vitrimer keeps absorbing moisture until 20 days to a moisture equilibrium content of 2.8%. The data indicate a higher susceptibility of the vitrimer to moisture uptake. This has been observed and attributed for similar transesterification vitrimers in literature to the presence of hydrophilic free hydroxyl groups [27], as well as the ester DCBs being prone to hydrolysis [18].

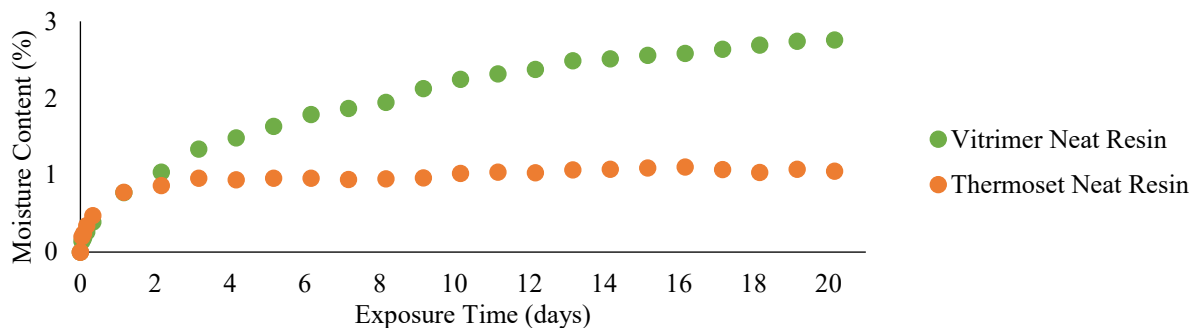


Figure 1. Moisture uptake of vitrimer and thermoset neat resin tensile samples at 70°C and 85% RH

Similarly, in Figure 2 while the thermoset composite achieves equilibrium after 10 days to a moisture equilibrium content of 0.37%, the vitrimer composite continues to absorb moisture until 30 days to a moisture equilibrium content of 1.0%.

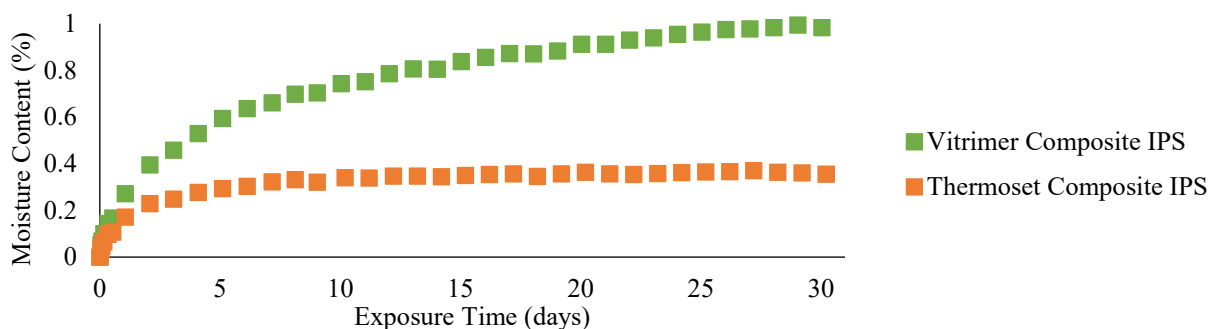


Figure 2. Moisture uptake of vitrimer and thermoset composite IPS samples at 70°C and 85% RH

Figure 3 displays the moisture conditioning curves for composite SBS samples. The thermoset composite reaches equilibrium after 4 days to a moisture equilibrium content of 0.58%, while the vitrimer composite reaches equilibrium after 6 days to a moisture equilibrium content of 0.88%. This difference in moisture uptake compared to IPS may be attributed to specimen geometry. While SBS and IPS samples both had a nominal thickness of 2 mm, SBS samples had a much lower surface area (width and length of 4 mm x 16 mm), versus that of IPS (25.4 mm x 200 mm).

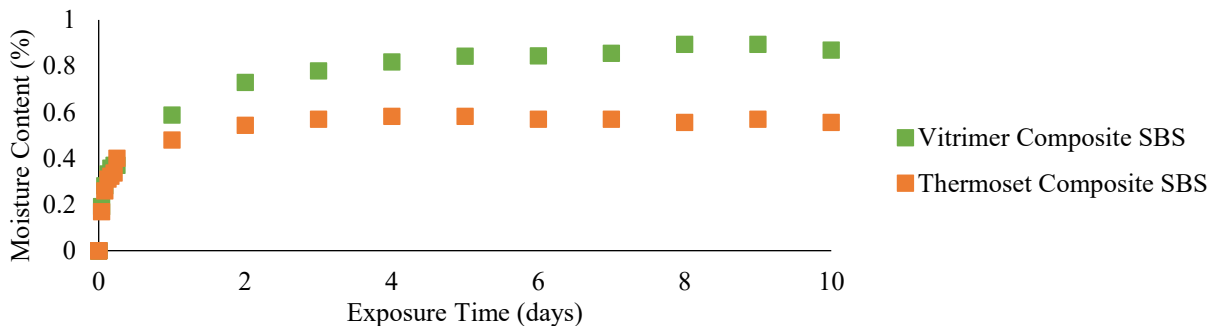


Figure 3. Moisture uptake of vitrimer and thermoset composite SBS samples at 70°C and 85% RH

3.2. Mechanical Testing

3.2.1. Neat Resin Tensile Testing

Figure 4 and Figure 5 demonstrate the reduction in tensile strength and modulus for thermoset and vitrimer neat resin after hygrothermal exposure. The vitrimer and thermoset have comparable tensile properties at RTD, with a higher vitrimer tensile strength observed. This may be due to a lower crosslink density from the sub-stoichiometric quantity of hardener in the vitrimer mix ratio, causing less brittle behaviour that better distributes internal stress [28]. As well, excess resin can cause a strengthening anti-plasticization effect, where unreacted epoxy chains favourably reduce molecular mobility through steric hindrance [29] [30]. At RTW however, the vitrimer neat resin displays a sharper decrease in tensile strength (-20.7%), versus the change in thermoset tensile strength (-0.9%). As well, the decrease in modulus for vitrimer neat resin (-31.6%) was greater than that for thermoset neat resin (-23.7%). Due to their ester DCBs, transesterification vitrimers have lower hydrolysis reaction barriers, which can lead to more severe structural degradation [18].

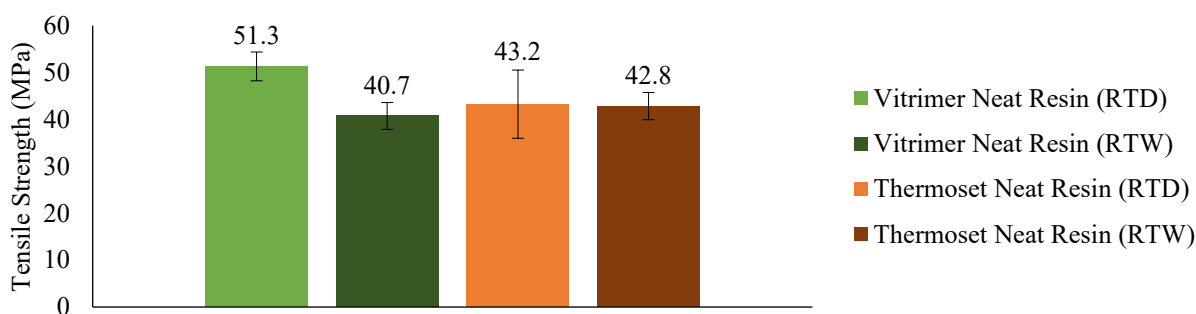


Figure 4. Average tensile strength of vitrimer and thermoset neat resin samples at RTD and RTW

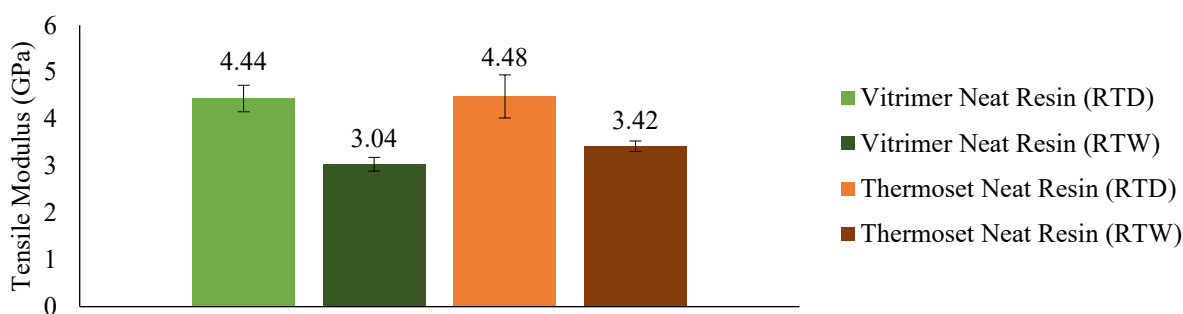


Figure 5. Average tensile modulus of vitrimer and thermoset neat resin samples at RTD and RTW

3.2.2. Composite In-Plane Shear Testing

The decrease in thermoset and vitrimer composite IPS strength from RTD to RTW is shown in Figure 6. At RTD, the vitrimer composite IPS is noticeably higher than that of the thermoset, which can occur for similar reasons to those discussed for the neat resin. A reduction in strength at RTW was only noted to occur in vitrimer samples (-23.5%), versus thermoset (+1.2%).

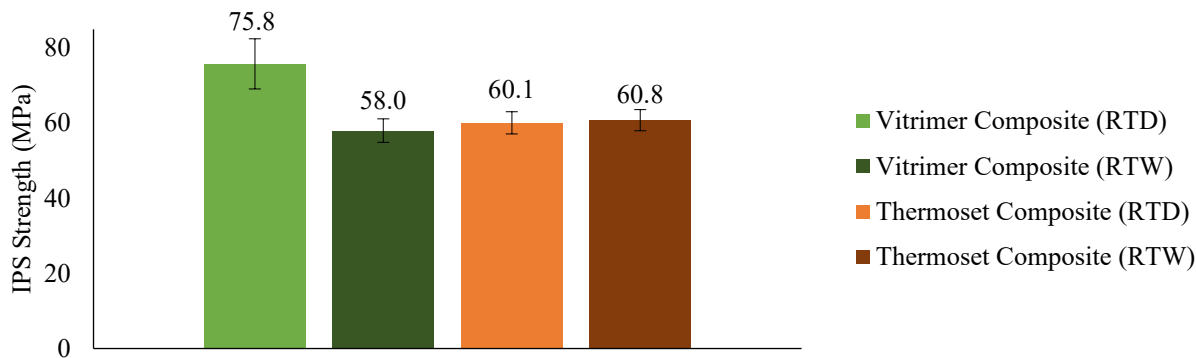


Figure 6. Average IPS strength of vitrimer and thermoset composite samples at RTD and RTW

3.2.3. Composite Short-Beam Shear Testing

A comparison of SBS strength versus moisture equilibrium content is shown in Figure 7. Figure 8 demonstrates the final decrease in SBS strengths from RTD to RTW. The vitrimer composite displayed a higher SBS strength until moisture equilibrium was reached. At RTW, a marked decrease was exhibited in the vitrimer composite (-40.4%), versus thermoset composite (-21.1%).

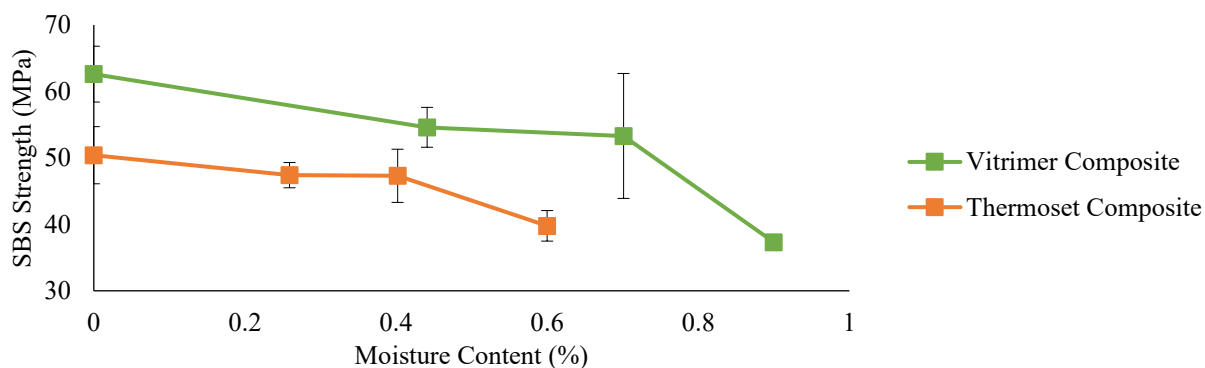


Figure 7. Average SBS strength of vitrimer and thermoset composite samples versus moisture content

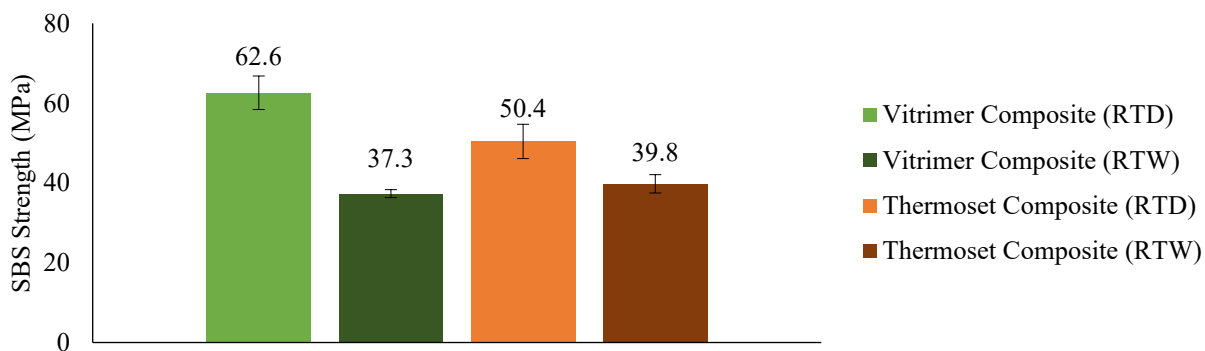


Figure 8. Average SBS strength of vitrimer and thermoset composite samples at RTD and RTW

The thermoset composite RTD and RTW data closely align with past research. For an epoxy resin CFRP, Joshi et al. noted a decrease in SBS strength at RTW, while no change in IPS strength [16]. SBS strength is considered a more representative experimental value of the interlaminar shear strength (ILSS) than IPS strength, which has a more complex stress state [23] [24]. The vitrimer composite SBS data potentially indicate poorer interlaminar adhesion after hygrothermal exposure, and thus a more susceptible fibre-matrix interface than conventional thermoset. Since hygrothermal effects induce accelerated plasticization and hydrolysis in transesterification vitrimers, the resulting degradation can promote increased void formation and microcracks at the fibre-matrix interface [18]. Contributing factors may also include more void nucleation sites produced by partial dissolution of the vitrimer zinc catalyst, even after thorough mixing.

4. Conclusions

While vitrimers have emerged showing promise in providing a sustainable alternative to thermoset composites, their intended operating environments must be considered. Current challenges with their industrial viability include the need for further research understanding their fundamental material properties and behaviour, especially in extreme conditions. In this work, a study was conducted to investigate the hygrothermal effects on a transesterification-based vitrimer system, and benchmark mechanical properties to thermoset. A greater susceptibility of vitrimer neat resin and composite to property degradation was observed after hygrothermal exposure, indicated by data for neat resin tensile, composite IPS, and SBS. Effective moisture equilibrium time for vitrimer neat resin and composite samples took considerably longer compared to their thermoset counterparts. A higher equilibrium content was noted for the vitrimer, with 2.8% absorbed for vitrimer neat resin compared to 1.1% for thermoset, and 1.0% absorbed for vitrimer composite versus 0.37% for thermoset composite. Vitrimer neat resin samples were shown to have a considerable decrease in tensile strength after exposure (-20.7%), while the change was negligible for thermoset (-0.9%). Also, vitrimer composite samples decreased in IPS strength (-23.5%), while thermoset composite did not (+1.2%). While both materials experienced property degradation in SBS strength, a more pronounced decrease was exhibited in vitrimer composite samples (-40.4%), versus in thermoset composite (-21.1%). Regarding the adoption of vitrimers in the aerospace industry, there is a need to prove their environmental durability in a wider array of tests such as flame, UV, and chemical resistances. Future studies may look to investigate further in these areas.

5. Acknowledgements

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6. References

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