WOOD-BASED PREPREG FOR COMPOSITE LAMINATES

M. Mohammadabadi

Assistant Professor
Department of Sustainable Bioproducts
Mississippi State University
Starkville, MS 39762-5724
E-mail: mm5132@msstate.edu

V. Yadama†

Professor
Department of Civil and Environmental Engineering
Composite Materials and Engineering Center
Washington State University
Pullman, WA 99164
E-mail: vyadama@wsu.edu

L. V. Smith

Professor
School of Mechanical and Materials Engineering
Washington State University
Pullman, WA 99164
E-mail: lvsmith@wsu.edu

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Abstract. A wood-based prepreg was formed using vacuum-assisted resin transfer molding (VARTM) and a low-viscosity thermoplastic resin. Wood strands were assembled to make a porous mat for resin injection. The resin filled most of the cavities inside the wood cells resulting in a void volume fraction of 7%. The Young's modulus and strength of the saturated wood strands were 38% and 124% higher, respectively, than those of wood strands prior to resin infusion. Flat laminates were produced by thermoforming prepreg plies at 180°C and 830 kPa, for 25 min. The Young's modulus and strength of flat 12-ply laminates were 73% and 20% higher, respectively, than a wood-strand panel produced using compression resin transfer molding (CRTM) and epoxy resin. Wood prepreg shows promise as an alternative to traditional wood composite forming processes, with the potential to simplify the manufacture of complex shapes, while improving the properties of the natural material.

Keywords: Wood prepreg, liquid thermoplastic resin, vacuum-assisted resin transfer molding, thermoforming; natural fiber panels, compression molding.

INTRODUCTION

Synthetic fibers are strong and stiff with lower density than metals and are in use in almost every type of advanced engineering structure from aerospace, marine, and automotive to sport and biomedical (Masuelli 2013). However, in addition to being expensive, they do not degrade at the end of their life. Although a small fraction of these synthetic composites are crushed into powder and used as filler or incinerated to obtain energy in the form of heat, most of them are not recycled and end up in land-fills (Mitra 2014). Environmental issues have motivated governmental actions in the form of environmental regulations to protect the environment for future generations. To increase the biodegradation and recyclability of products, and reduce the use of petroleum sources, natural fibers have received considerable attention from both academia and industry. Since natural fibers are renewable, degradable, carbon negative, nonabrasive, less emission of toxic

* Corresponding author
† SWST member

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fumes, and abundant, there has been an increase in natural fiber composites (NFCs) research (Saheb and Jog 1999; Westman et al 2010; Ahmad et al 2015; Pickering et al 2016) for a variety of applications including aerospace (John et al 2008; Haris et al 2011; Boegler et al 2015) and automotive (Drzal et al 2001; Huda et al 2008; Hill et al 2012; Verma and Sharma 2017). Due to these advantages, NFCs are a realistic alternative to synthetic composites that meet the requirements of the automotive industry for both exterior and interior applications (Holbery and Houston 2006). However, one challenge with NFC utilization in the automotive sector is the capital requirements and risk averse philosophy associated with high production processes (Hill et al 2012). Also, typically thermoset resins have been used to produce NFCs for automotive applications due to poor fiber-matrix interaction.

Among the natural fibers, wood has the highest annual production (Antov et al 2017). Healthy forests play a vital role in meeting the climate change/global warming challenge through carbon sequestration in trees and wood products. The practice of thinning is an effective method to improving the overall health and value of a forest, mitigating fire risk, and optimizing forest management regimes. However, the biggest challenge with thinned materials is that they are generally left on the forest floor or stacked and burned (Hunt and Winandy 2002). Therefore, high-value markets can not only efficiently use these low-quality materials, but can also recover the cost of thinning and management processes.

Liquid molding technology (Nedanov and Advani 2000; Umer et al 2007), commonly used for synthetic fibers, provides an opportunity to develop a sustainable manufacturing infrastructure. Properly developed, it has the potential to convert underutilized lignocellulosic fiber from forest thinning for hazardous fuel reduction and fast growing short-rotation plantations into net shape composite products for niche markets such as the automotive, marine, and aviation industry. Resin transfer molding (RTM) (Fong and Advani 1998; Rouison et al 2004, 2006; Verrey et al 2006), vacuum-assisted resin transfer molding (VARTM) (Grimsley et al 2001; Kang et al 2001; Dai et al 2004), and compression resin transfer molding (CRTM) (Bhat et al 2009; Idicula et al 2009; Verleye et al 2011) are some variations of liquid molding technology for production of composites with complex geometries and large curvatures. Liquid molding, such as RTM, has been used in the aerospace and automotive industries due to its cost-effectiveness and dimensional stability. While natural (jute and sisal) and synthetic fibers (glass and carbon) are commonly used, the authors have shown that low-cost wood strands can be formed with controlled orientation and consolidated using RTM to yield high-performance thin flat panels (Yang 2014; Gartner et al 2022).

One method to increase the use of NFCs and enable sustainable forest management is to adapt underutilized wood into material forms that have successfully been used with synthetic composites. Synthetic fiber (ie carbon or glass fiber) thermoset or thermoplastic prepregs are growing in popularity among all segments of the composites industry at 10% per year since 2002 (Stewart 2009). The demand for thermoplastic composites is strong as they can be recycled and their market size is estimated to grow from USD 22.2 billion in 2020 to USD 31.8 billion by 2025 (Garofalo and Walczyk 2021).

Considering theses aspects, however, the composite industry lacks a natural fiber-based prepreg, analogous to a synthetic fiber prepreg (such as carbon fiber prepreg), with a thermoplastic or a thermoset matrix. The innovation described in this paper is a natural fiber prepreg using wood strands that has been developed with a thermoplastic resin for use as feedstock for fabricating laminated composite materials, flat or profiled, by compression molding for a variety of applications including automotive interior and exterior panels. Wood strands have an advantage over other natural fibers and wood fiber as they enable production of higher performance composite panel products with complex geometries and large curvatures for more demanding applications as needed in the automotive and aerospace industries. Lightweight and renewable materials are of interest to several industries including aerospace and automotive; the automotive industry is pivoting toward bio-based materials for interior and exterior body parts. Meeting the requirements, such a prepreg can be introduced by current consumers of the synthetic prepreg to their fabrication line without any disturbance to the manufacturing process. Due to development of low-viscosity liquid thermoplastic resin (Kinvi-Dossou et al 2019), in this study, a thermoplastic wood-based prepreg was developed using a VARTM process. Processing parameters to thermoform flat laminates from the prepreg were investigated. Production process of wood strand prepreg and a composite laminate using prepregs is depicted in Fig 1.

MATERIALS

Thin wood strands measuring 146 × 19 × 0.36 mm were produced from small diameter trees (ponderosa and lodgepole pine logs ranging in diameter from 191 to 311 mm) and dried to approximately 1% MC. Wood strand mats were assembled using strips of tacky paper (Super 77 Multipurpose Adhesive by 3M), as shown in Fig 2(a). The preform was placed under peel ply and vacuum bagged using VARTM as shown in Fig 2(b). Spiral tubes under the bagging film on both ends of the preform were attached to the resin inlet and outlet for an even flow of resin. A low-viscosity resin (Elium® 150, Arkema, Prussia, PA) was mixed with 3% initiator (Luperox® AFR40 benzoyl peroxide) prior to injection. Elium® is a thermoformable, infusible, and recyclable acrylic thermoplastic resin and has high mechanical properties and compatibility with conventional thermoset processes (Nash et al 2018; Arkema 2021). Fiber-reinforced Elium® resin can be thermoformed with heat and pressure as the resin undergoes a radical polymerization to produce a thermoplastic matrix after injection and the curing process. Resin-injected prepregs were then allowed to cure at room temperature. At room temperature, the resin system has a viscosity of 100 cps, open time of 20 min, and cure time of 40 min. A finished wood prepreg with an average thickness of 0.43 mm is shown in Fig 2(c) and (d).

EXPERIMENTS

Strands and Prepregs

To determine the level of resin saturation, unprocessed plain wood strands were compared with the prepreg under a scanning electron microscope (SEM). The effect of VARTM was determined by comparing the mechanical properties of unprocessed strands to those that are resin saturated (shown in Fig 3[a] and [b]) using the mechanical test coupons described in Table 1. To examine the effect of fiber discontinuity, the mechanical properties of the prepreg were evaluated by testing large samples cut in the longitudinal direction as shown in Fig 3(c).

Since fiber content is a key factor for prepregs and composite materials, the following procedure was used to determine fiber, resin, and void volume fractions of the wood prepreg. The weight difference of the wood strand preform (Fig 2[a]) before resin injection (M_F) and the prepreg after cure (M_C) (Fig 2[c] and [d]) were used to obtain the resin weight (M_R). Wood strands are composed

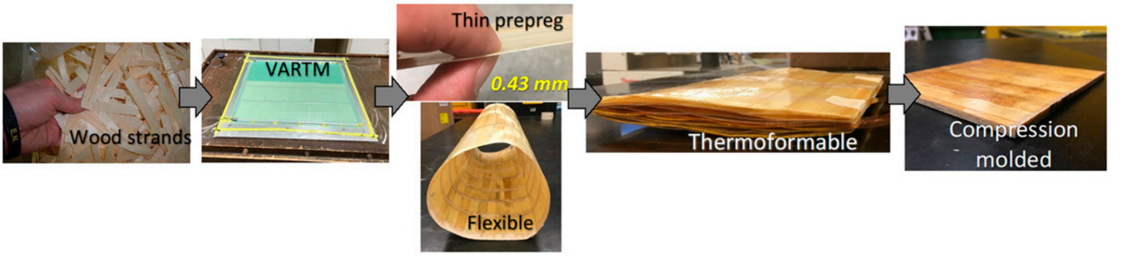

Figure 1. A brief pictorial summary of wood strand prepreg and composite laminate process.

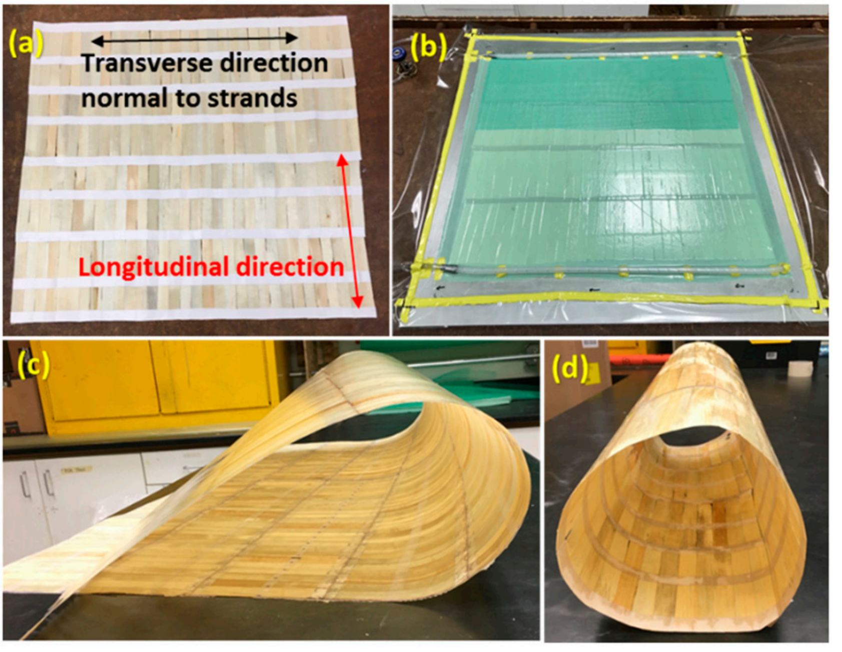

Figure 2. Prepreg development (a) wood strand mat indicating longitudinal and transverse directions (b) preform under vacuum bagging (c, d) thin and flexible prepreg.

of wood fibers that include the cell wall material (assumed to include the interfibrillar and cell wall void as well) and the fiber lumen (void in the fiber core). The wood fiber volume, V_F, was found from the wood cell wall density, ρ_W, (1524 kg/m^3; Kellogg and Wangaard 1969) as

$$V_F = M_F/\rho_W \tag{1}$$

Knowing the volume of the composite (V_C) by measuring the dimensions of the wood prepreg, the void volume, V_V, was found from

$$V_V = V_C - V_F - V_R = V_C - \frac{M_F}{\rho_W} - \frac{M_R}{\rho_R} \tag{2}$$

where V, M, and ρ are volume, mass, and density, and subscripts C, F, R, W refer to composite, fiber, resin, and wood cell wall, respectively. Knowing the volume of fiber, resin, and void, the volume fractions were computed with respect to the volume of the composite.

Composite Laminates

To laminate layers of prepreg into a composite laminate, the prepreg thermoforming temperature must be higher than its glass transition temperature (T_g) and lower than its thermal degradation temperature. Dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA) tests were conducted to find these temperature limits. For the DMA test, both single prepreg plies and 6-ply laminates were evaluated, while 12 g prepreg samples were used for the TGA analysis.

To achieve good bonding between the prepreg plies, a suitable temperature and pressure for thermoforming were determined. Twelve plies of prepreg were hot-pressed at different temperatures and pressures to make flat laminates and tested using the shear block test (ASTM 2021) to evaluate the bonding between prepreg plies. To have specimens with the desired thickness for shear block tests, samples cut from laminates were sandwiched between two layers of a wood-strand panel. The midplane of the 12-layer-prepreg

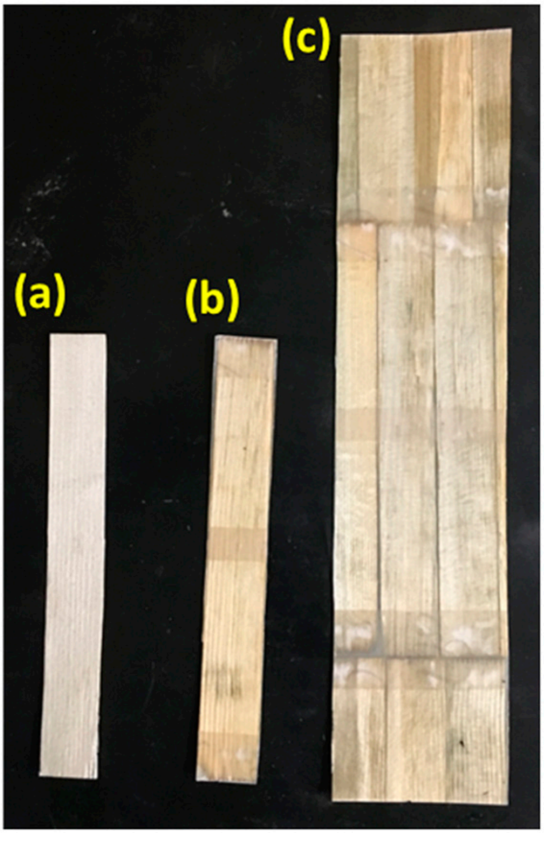

Figure 3. Tensile coupons (a) plain strand, (b) resin-saturated strand, and (c) large coupons having fiber discontinuity cut from wood prepreg in longitudinal direction.

laminate was the plane subjected to shear by compression loading.

Composite laminates consisting of 12 plies of wood strand prepreg were produced under the predetermined conditions for mechanical testing. Specimens cut from these laminates were submitted to tensile, bending, and water absorption (WA) and thickness swelling (TS) tests (ASTM 2020). The dimensions of these specimens are given in Table 1.

RESULTS AND DISCUSSION

Strand Impregnation

The average thickness of the prepreg was 0.43 mm with COV of 5%. Three samples, fully resinated, partially resinated, and plain strands shown in Fig 4(a) were evaluated under SEM to see how resin permeates throughout wood strands. Partially resinated samples were cut from regions where one side of the strand was wetted by resin whereas the other side was dry. This method resulted in good quality prepreg and partially resinated only occurred in two prepreg samples in regions close to the resin outlet. Empty lumens can be seen in Fig 4(b) for plain strands. It can be seen how fiber lumens of fully saturated strands shown in Fig 4(d) and (e) have been filled with resin. For partially resinated strands, where the resin could reach just one side of the strands, only half of the fiber lumens have been filled as shown in Fig 4(c). Unlike thermoplastic polymers such as polypropylene and polyethylene which only encapsulate the wood particles (López et al 2018) when used in wood thermoplastic composites, SEM images demonstrate that the low-viscosity thermoplastic resin used in this study can not only encapsulate the wood fibers,

Table 1. Dimensions of specimens used for mechanical testing (all are in mm).

Material	Test	Details such as size, shape, and direction		#	Length: Total	Length: Active[a]	Width	Thickness
Plain strand	Tensile	Rectangle-longitudinal		15	146	96	19	0.40
Prepreg	Tensile	Strand size	Rectangle-longitudinal	33	146	96	19	0.43
		Large Coupon	Rectangle-longitudinal	35	255	166	51	0.45
Laminate	Tensile[b]	Dog bone-longitudinal		6	255	166	39[c]	5
	Bending	Rectangle-longitudinal		5	185	140	51	5
	WA-TS	Square		5	153	—	153	5

[a] Active length is the length between two grips for tensile and two supports for bending specimens.
[b] Fillet radius was 76 mm and gauge length was 51 mm.
[c] This dimension is width of the reduced section.

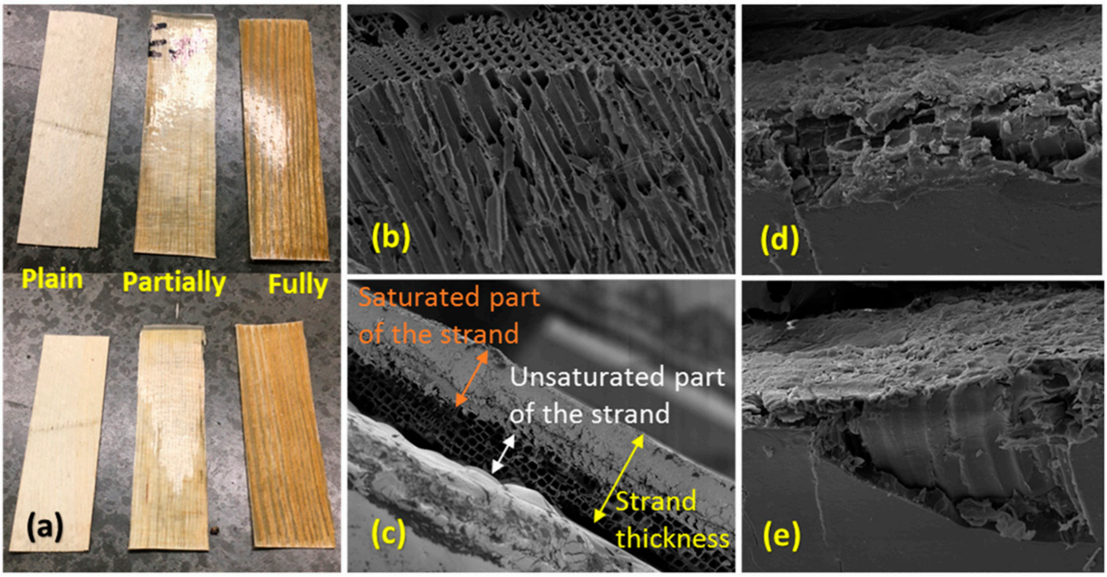

Figure 4. Scanning electron microscope evaluation (a) both sides of plain, partially, and fully resinated, (b) plain strands, (c) partially resinated, (d, e) fully resinated strands.

but can also penetrate the fiber lumens within the strands of the prepreg. In addition, the effect of penetration of high-density polyethylene (HDPE) in different wood species (lodgepole pine, grand fir, and Douglas fir) was evaluated using vacuum bagging (Gacitua 2008), and the results showed that the penetration parallel to resin flow varies between 0.04 and 0.1 mm for both earlywood and latewood. However, the low-viscosity thermoplastic resin in this study was able to penetrate the whole thickness of the wood strands (0.4 mm), which was normal to resin flow.

Experimental Mechanical Properties of Prepreg

Saturated strands, cut from prepreg, along with plain strands, were submitted to tensile tests to compare the effect of VARTM on strand mechanical properties, as shown in Table 2. The average Young's modulus and strength of saturated strands increased by 38% and 124% compared with those of plain strands, respectively.

Since the wood strands in the prepreg are not continuous, larger specimens (shown in Fig 3[b]) were cut from the prepreg in the longitudinal direction and tensile tested. Because of the discontinuity between the strands, the mechanical properties of these specimens given in Table 2 are lower than those of saturated strands having no discontinuity.

Analytical Mechanical Properties of Prepreg

Fiber, resin, and void volume fractions of the prepreg were found to be 25%, 68%, and 7%, respectively. Knowing these properties, the rule of mixtures (Gibson 2016) can be used to predict ideal continuous fiber material properties of saturated strands by

$$E_{SS} = \nu_F E_F + \nu_R E_R \text{ and } U_{SS} = \nu_F U_F + \nu_R U_R \quad (3)$$

where E and U are Young's modulus and strength, ν is the volume fraction, subscript SS refers to saturated strand, subscript F refers to fiber, and subscript R refers to resin. The stiffness and strength of plain strands depend on the strand fiber volume fraction (ν_{PS}) as

$$E_{PS} = \nu_{PS} E_F \text{ and } U_{PS} = \nu_{PS} U_F \quad (4)$$

Table 2. Mechanical properties of Elium®, plain strands, saturated strands, and coupons cut from wood prepreg (COV is mentioned in parenthesis).

			Saturated strands		
	Elium® (Bair 2020)	Plain strands	Experiment	Rule of mixtures	Longitudinal prepreg specimens
Young modulus (GPa)	3.3	9.82 (28%)	13.58 (13%)	12.92	10.03 (20%)
Strength (MPa)	76	46 (45%)	103 (20%)	102	32 (47%)

where *PS* stands for plain strand and ν_{PS} is the average fiber volume fraction in plain strands which using Eq 1 and physical properties of the plain strands (dimensions and mass) was found to be 0.23. The measured and predicted saturated strand modulus and strength are compared in Table 2. Good agreement was found (within 5% for Young's modulus and 1% for strength) due to uniform resin penetration into the fiber lumens.

Thermoforming Variables

The glass transition and thermal degradation temperatures were found to be 132°C and 257°C, respectively. Since hemicelluloses, a primary constituent of the wood cell wall, begins to degrade at around 220°C (Waters et al 2017), a temperature of 200°C was used as the upper thermoforming limit. The effect of the processing temperature (140, 160, and 180°C), pressure (380, 555, 690, 830 kPa), and thermoforming duration (15 and 25 min) were evaluated using the shear block test (ASTM 2021).

A thermocouple was placed at the mid plane of 12-ply laminates to monitor temperature, which, on average, required 6 min to reach the target temperature. Laminates were left under pressure for an additional 19 min (25 min thermoforming duration). The laminates cooled under pressure to 80°C at which point the laminate was unloaded and removed from the press. The shear strength of six specimens cut from each laminate is reported in Table 3, which tended to increase with temperature and pressure (except for 555 kPa and 180°C). The fiber and matrix demonstrated good adhesion with increasing the temperature and pressure as specimens failed due to fiber failure rather than adhesion. Different types of shear failure are shown in Fig 5 (the bonding area of shear block specimens is indicated by dashed line).

Table 3 includes the effect of the cooling process and pressing time on shear strength. Laminates pressed at 180°C and 830 kPa were removed from the press prior to cool. For a 25 min press time the shear strength decreased by 56% by removing the cooling step. Reducing the pressing time from 25 to 15 min decreased the shear strength by 20%. These results show that bond strength depends strongly on the hot-press duration and the cooling process. In the following, laminates were formed at 180°C and 830 kPa for 25 min, and cooled to 80°C under the target pressure of 830 kPa as these specified thermoforming variables resulted in an excellent bonding between prepreg plies as shown in Fig 5(c).

Laminate Mechanical Testing

The results of laminate bending and tensile tests are given in Fig 6. The tensile Young's modulus of the wood prepreg laminate was 66% and 22% larger than the wood prepreg and the saturated strands, respectively, as shown in Fig 6(a). The laminate tensile strength (Fig 6[b]) was 150% higher than the prepreg and 22% smaller than the saturated strands. Fiber discontinuity and different densities between specimens cut form the prepreg and laminate likely cause the differences. The average density for wood prepreg was 1026 kg/m^3, while it increased by 12% and reached 1151 kg/m^3 for flat laminates.

The prepreg laminate is compared in Fig 6 with that of similar composites fabricated from wood strands but using different manufacturing techniques and resins (Gartner 2017; Mohammadabadi et al 2020). RTM with external pressure is known as CRTM and was used to manufacture wood-strand-based composites with Derakane 411-350 epoxy vinyl ester resin (Gartner 2017). The thickness and fiber volume fraction for this composite

Table 3. Maximum shear stress in samples made under different temperature and pressures and submitted to shear block test.

Laminates under target temperature for 25 min but under target pressure until cool down									
Target pressure (kPa)	380			555		690		830	
Target temp. (°C)	140	160	180	160	180	160	180	160	180
Shear strength (kPa) (COV %)	1793 (52)	2868 (34)	4675 (46)	2992 (74)	4199 (43)	3027 (32)	5716 (13)	4730 (45)	7308 (12)
Laminate under target temperature and pressure for specified pressing time with no cooling									
Target pressure (kPa)								830	
Target temp. (°C)								180	
Press time (min)								15	25
Shear strength (kPa) (COV %)								2551 (15)	3206 (30)

were 6.35 mm and 37%, respectively. Densifying the wood strand mat by applying pressure during resin injection and injecting resin with high pressure resulted in fiber compaction and higher fiber volume fraction and less void content compared with the prepreg laminate developed in this study. Another composite was produced by hot-pressing wood strands with a low percentage of phenol formaldehyde resin (8% of the oven-dry weight of the wood strands) (Weight and Yadama 2008a 2008b; Mohammadabadi et al 2020). The thickness and density of the hot-pressed wood strand composite were 6.35 mm and 640 kg/m^3, respectively. The fiber volume fraction of the composite was about 40%. Even though the laminate developed in this study had a lower fiber content

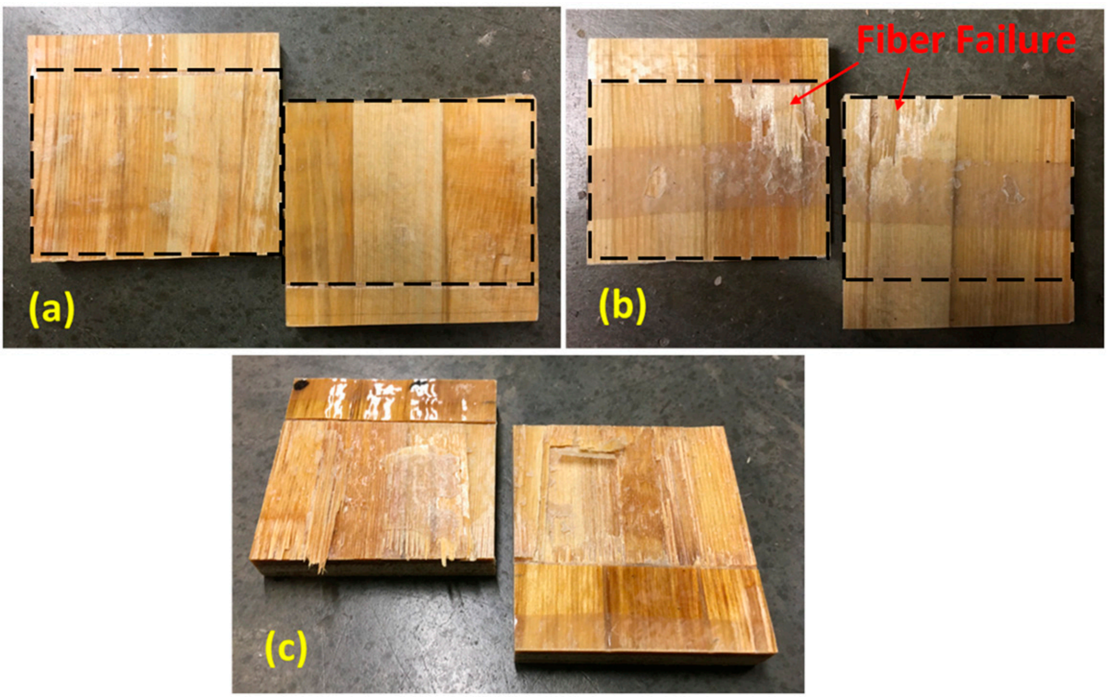

Figure 5. Different types of shear failure (a) pure adhesion failure, (b) partial fiber failure, and (c) pure fiber failure.

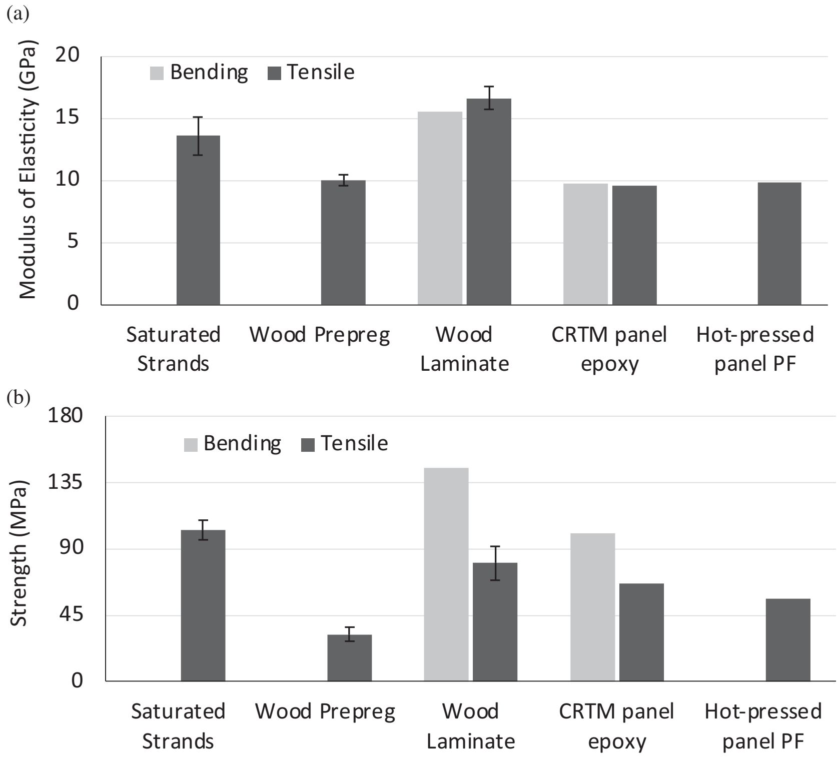

Figure 6. Results of mechanical testing (a) modulus of elasticity and (b) strength compared with other studies and similar materials.

compared with the other composites, its modulus of elasticity and strength in tension and bending were noticeably higher. For the bending test, the modulus of elasticity of the laminate was 60% higher than that of the CRTM panel and in tension, it was 73% and 69% larger than CRTM and hot-pressing panels, respectively. The modulus of rupture of the laminate was 44% higher than that of CRTM and its tensile strength was 20% and 42% higher than that of CRTM and hot-pressing panels, respectively. Fiber orientation is one reason for this difference as all wood strands were oriented in the longitudinal direction to make the prepreg as shown in Fig 2(a). For CRTM and hot-pressed panels, however, strands were oriented within ±35 degrees with respect to the longitudinal direction. Compared with CRTM specimens with a higher fiber volume fraction of 37%, the laminates yield better mechanical properties which could be due to higher mechanical properties of the thermoplastic resin or improved interaction between the thermoplastic resin and wood strands compared with the low-viscosity thermoset resins used for the CRTM specimens.

WA and TS of the wood prepreg laminate after 2 and 24 h are shown in Fig 7 and are compared with the wood strand composites produced (using CRTM and hot press) and tested by Gartner (2017) and Weight and Yadama (2008a, 2008b). The laminate and CRTM panel showed similar behavior in the presence of water. The wood prepreg laminates showed much better performance compared with hot-pressed panels as the low-viscosity resin during the VARTM process was mostly able to fill all the cavities or fiber lumens within the wood strands. These superior moisture resistant properties are important for automotive applications where shape stability and durability are critical.

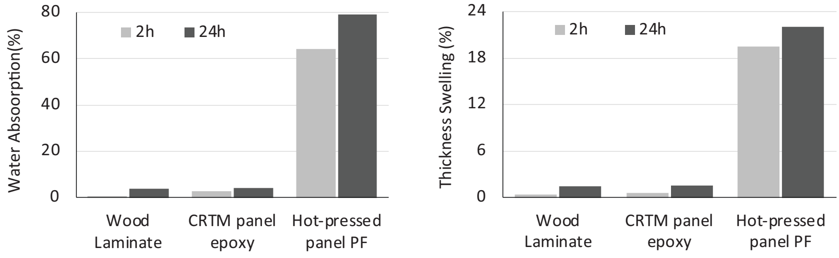

Figure 7. Comparison between water absorption and thickness swelling of wood prepreg laminate with other bio-based composites.

CONCLUSIONS

A natural fiber-based thermoplastic prepreg similar to a synthetic prepreg has been developed to manufacture molded and shaped interior panels for a variety of applications, including automotive. Wood strands from small diameter timber were used to develop a wood-based prepreg using a novel thermoplastic resin and VARTM technique. Unlike the traditional thermoplastic resins which penetrate only a few microns into the wood structure, the low-viscosity thermoplastic resin in this study easily penetrated the fiber lumens. This resulted in a low void volume fraction and a high-performance thin wood strand prepreg. The interlaminar shear strength of laminates produced from the wood prepreg increased with the thermoforming duration and cooling under pressure. Due to reprocessability of the wood-based prepregs, they can be used to produce flat or shaped composite laminates, with high strength, stiffness, water resistant, and dimensional stability.

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