



Research Article

Hollow canola-wood thermoset composites from concept to completion: fabrication, performance, failure and reliability analysis



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Abstract

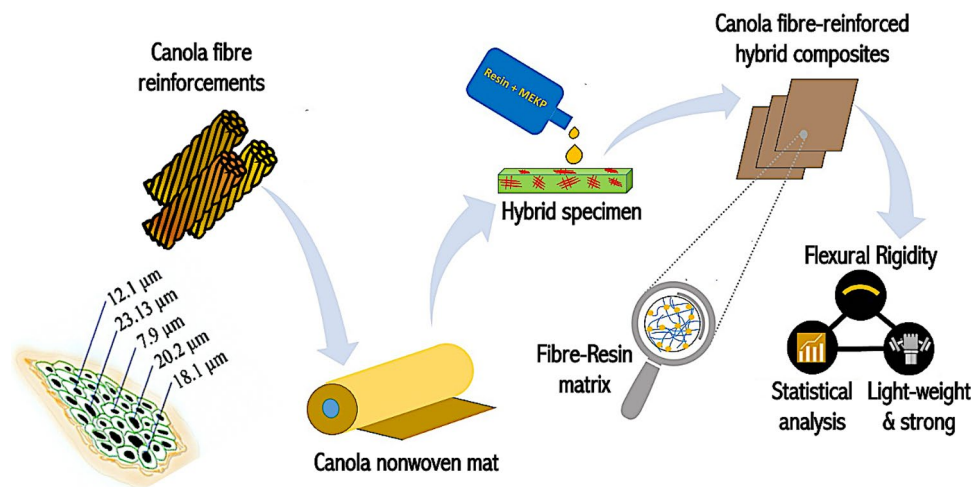
This study aims to reinforce wood particle composite structures with novel lightweight hollow Topas canola fibres (*Brassica napus* L.). The feasibility of methyl ethyl ketone peroxide (MEKP)-cured hybrid composite structures of canola and wood biomass is demonstrated in the current study. By employing a facile fabrication technique under ambient conditions, different hybrid canola composite structures are fabricated with randomly distributed fibre polymer reinforcements and synthetic resin matrix. Hybridization process involves the incorporation of hollow canola fibres and wood biomass to yield better mechanical performance including breaking strength, flexural rigidity, and strength to weight ratio. The produced hybrid composites can outperform pristine heavyweight wood composites, exhibiting superior flexural strength up to $6.4 (\pm 2.5)$ Pa·m⁴ of flexural rigidity and 728.35 (± 242) N of breaking load with the extra feature of lower wood content. The hybrid composite structure exhibited a modulus of elasticity up to 8.76 GPa exhibiting ~6% higher stiffness than the pristine wood composites. Also, the diameter (84.30 ± 38.04 μ m) of the canola fibres is coarse enough for composite fabrication. This confirms the effective contribution of canola fibres to the flexural properties of hybrid composite structures. Further, the two-parameter Weibull mathematical model is applied for failure ($R^2 \geq 0.92$) and reliability ($R^2 \geq 0.93$) analysis. The constructed probability distribution curves predict probabilistic composite failures against ranges of bending deflection. The intrinsic hollow interior of the novel canola fibre opens new doors for self-healing natural fibre-based composite fabrication. Such lightweight composite structures can find promising applications in technical textiles, including the interior panels of automotive, aerospace, furniture, and the construction industry.

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Graphical abstract



Keywords Biomass · Hollow fibre · Canola · Wood · Composite · Lightweight · Weibull distribution

Abbreviations

CS	Composite set or specimens
SEM	Scanning electron microscope
VARTM	Vacuum assisted resin transfer molding
MEKP	Methyl Ethyl Ketone Peroxide
UPE	Unsaturated polyester
VER	Vinyl ester
WC	Wood crumbs
CTC	Crop Technology Centre
ASTM	American Society for Testing Materials
FR	Flexural rigidity
BL	Breaking load
MOE	Modulus of elasticity

1 Introduction

Technical textiles are defined as the “textile materials and products manufactured primarily for their technical and performance properties rather than their aesthetic or decorative characteristics” [1]. The technical textiles industry is the fastest growing sector of the functional textile and electronic garment world with an annual growth rate of 4% since 2008 [2]. Lightweight textile fibres and yarns are of growing interest for technical textile industries, and along with nonwoven fabrics and fibre reinforced flexible/rigid composite structures, make up the majority of the technical textile market. Twelve major markets in the domain of technical textiles have been illustrated in Fig. 1.

Lightweight is a buzzword for aerospace, automotive, construction, and biomedical applications. The use of lightweight natural fibres could remarkably reduce

consumption of composite reinforcements, energy consumption, and thus the overall cost of purchase and operations. Automotive and construction industries have already started investigating the use of natural fibres to promote sustainability and reduce carbon emissions [3]. The low-density profile of bamboo fibres has been used in designs for bamboo fibre-based composites for industrial application [4]; a coir (coconut) fibre-based composite formulation for automotive interior panels has been proposed [5]. To reduce the carbon footprint, Joshi et al. [6] have reviewed the prospects of replacing synthetic fibres with plant-based fibres. Although there are debates among the scientific and engineering communities over the preference between synthetic and natural fibre applications, the research community has been making concerted efforts to use natural fibre-based composite structures because of the low cost, ease of availability, zero health hazard, negligible wear during processing, biodegradability, and low density offered by lignocellulosic textile fibres like jute, flax, hemp.

Supplementary reinforcement materials to partially or completely substitute wood content in wood-based panels like particle boards, fibre boards or plyboards has long been a research topic for the scientific and engineering community. Woody and non-woody agricultural biomass continue to be investigated over the last decades for use in particleboards by completely replacing the wood content with a wide range of industrial crop residues including coir fibres [7], walnut shell [8], flax shiv [9], cotton carpels [10], kenaf [11], maize husk and cob [12], reed and wheat straw [13], wheat straw [14], hazelnut husk [15], eggplant stalks [16], oil palm trunks [17]. Although in many instances these

Technical textile application areas

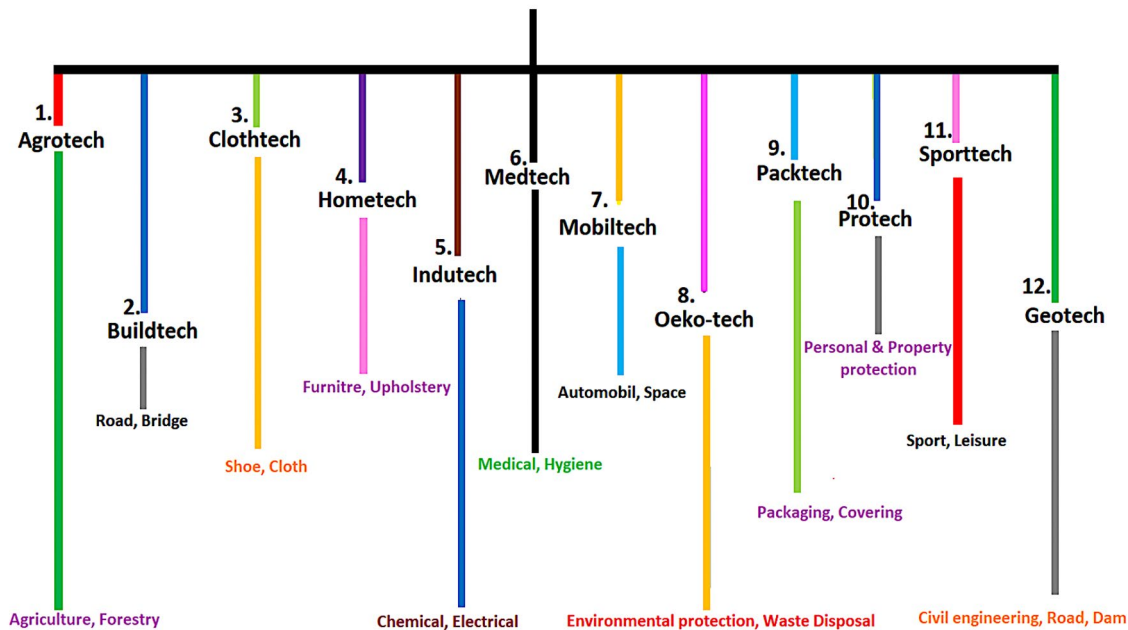


Fig. 1 Different application areas of technical textiles

100% natural or plant-based biomass manufactured particleboards meet the minimum criteria of a particleboard, a hybrid mixture of natural biomass and wood particles offers a comparatively superior mechanical performance, which was demonstrated in hybrid composites using varying proportions of jute-wood [18], peanut hull-wood [19], sunflower stalk-wood [20], and kiwi pruning-wood [21]. The current study took a similar approach to manufacture hybrid composites using biomass of industrial oil-plants and wood particles and compared their flexural performance against 100% pristine wood particle composites. The resulting hybrid composite structures may be used for furniture applications or interior panels for automotive, aerospace, and construction industries.

In the quest for developing low density and green material-based composites, researchers often use hollow fibres to reduce the overall weight of the composite structures [22, 23]. Kumar et al. [4] recently designed a natural fibre-based light-weight high-strength composite for aerospace and automotive application using hollow glass microspheres. The application of hollow fibres also enables the application of healing agents through the hollow interior to develop functional self-healing composite panels [26]. Aerospace industries are exploring hollow fibres for self-healing applications. However, a naturally available hollow fibre could be a good choice over synthetic glass fibres to reduce environmental carbon footprint. Canola, a new generation cellulosic textile

fibre extracted from the biomass of *Brassica napus* L. oil-plants, was recently shown to possess an inherently hollow structure [27, 44]. Furthermore, Shuvo et al. [27] revealed that canola is the most lightweight textile bast fibre having a fibre density of 1.34 g/cc [27], which is demonstrably lower than other major textile fibres like cotton (1.54 g/cc) [28], hemp (1.48–1.49 g/cc) [29], flax (1.54 g/cc) [28], and jute (1.44–1.50 g/cc) [29], thus making it an excellent choice for lightweight composite structures. Topas canola fibres exhibit a satisfactory level of breaking tenacity (12.59 g-force/tex) [27], which is quite close to jute (18 g-force/tex) but lower than flax (23.4 g-force/tex) and hemp (27 g-force/tex) [29]. Hence, the study aimed to reinforce wood particle composite structures with the recently discovered novel hollow canola fibres. Hybrid composite structures were fabricated by combining these natural polymers (canola) with wood particles for a comparative analysis with pristine wood composites of 100% wood particle content. To the best of our knowledge, this is the first study focusing on fabricating hollow canola fibre reinforced composite structures.

Unsaturated polyester (UPE) and vinyl ester (VER) resins were utilized to produce Topas canola and wood particle reinforced hybrid composites. In Sect. 2 of this current study, experimental materials (2.1) and characterization techniques (2.2–2.4) were introduced. Subsequent attempts were made to manufacture composite structures using canola nonwovens with different synthetic

resins and dust of wood crumbs followed by a comparative statistical study among their flexural rigidities. At the end of this section, the experimental design flowchart (2.5) summarizes the overall process starting from fibre production to characterization of canola composites. Section 3 discusses several findings and observations recorded during the experiment, such as the fibre diameters (3.1), flexural strength (rigidity, resistive loading, and stiffness) of composite structures (3.3–3.5), and the prospect of hollow canola fibres for composite fabrication (3.2). A two-parameter Weibull distribution was introduced (3.6) at the end of this section for probabilistic prediction about the failure deflection length of the composite structures. Several research works used this model to predict mechanical properties of fibres; examples include predicting the tensile strength of alkali treated fibres by Roy et al. [30] or survival probability analysis of jute fibre against tensile behavior by Sayeed and Paharia [31]. Section 4 gives concluding remarks and future work directions.

2 Experimental

2.1 Materials

Seeds (Fig. 2a) of Topas cultivar of canola (*Brassica napus* L.) were germinated inside a growth room (Fig. 2b) (day temperature: 22 °C; night temperature: 17 °C). Soil named “Sunshine® Professional Growing Mix”, from Sun Gro Horticulture, Canada, was used for growing plants. At the two-leaf-stage, the plants were transplanted to larger plastic pots (14.60 cm in height) and placed inside the greenhouse. The atmospheric condition of the greenhouse (temperature: high 25 °C, low 22 °C, relative humidity 40–50%, light cycle: 16 h light, 8 h dark) was controlled by Argus Control System Ltd., Surrey, BC Canada. Flowering stage of canola plants (Fig. 2c) appears prior the harvesting stage of the dead plants that are ready for biomass collection process. Harvesting time is the most labor-intensive period and require much attention. Once the harvesting period

arrived, the dead plants (Fig. 2d) were cut and collected on the 116th day inside the greenhouse [32].

Following harvesting, plant samples were brought to the Textile Laboratory in the Department of Biosystems Engineering at the University of Manitoba. Canola fibres (Fig. 3a) were extracted from the plant stems (Fig. 3b) by water retting at room temperature based on the water-retted canola fibre extraction process detailed in our previous work [32].

The extracted canola fibres were flexible with an average length of 7 cm. The density and strength index of this lignocellulosic fibre was 1.36 g/cc and 2.63 lb/mg, respectively. Typically, cellulose content of the canola fibres is 61.3% [24]. Further, industrial softwood crumb particles were used in its pristine form without any modification for composite fabrication. Unsaturated polyester resin (UPE) and vinyl ester resin (VER) were supplied by AOC, LLC Chemicals, Guelph, Ontario, Canada (Altek General Purpose Laminating Purpose: H834-RDE-23; Lot: G180). The curing agent methyl ethyl ketone peroxide (MEKP) was supplied by Structural Composite Technologies Ltd., Canada (Product Trade name: NOROX MEKP-925H; Specification: 185,547; CAS-N0: 1338–23-4 Mother supplier: United Initiators, Inc., Helena, AR, USA).

2.2 Methods

Nonwoven fabric (Fig. 4) was prepared using extracted fibres of Topas biomass. Next, canola composites were manufactured using canola nonwoven fabric specimens with different polymer resin matrix systems (UPE/VER), and recycled dust of wood crumbs (WC). A constant polymer matrix solution of 150 ml solution was used in preparing all the composite structures, where 145.5 ml resin (UPE/VER) was added to 4.5 ml of MEKP. The recipe for formulating the 3% MEKP assisted resin solution was based on the 3% MEKP assisted hemp-reinforced composites developed by Qui et al. [33]. A Tension Frame (Model 2017) was used to conduct a three-point bending test according to ASTM D3410 (2016) [34] to analyze the breaking load and flexural

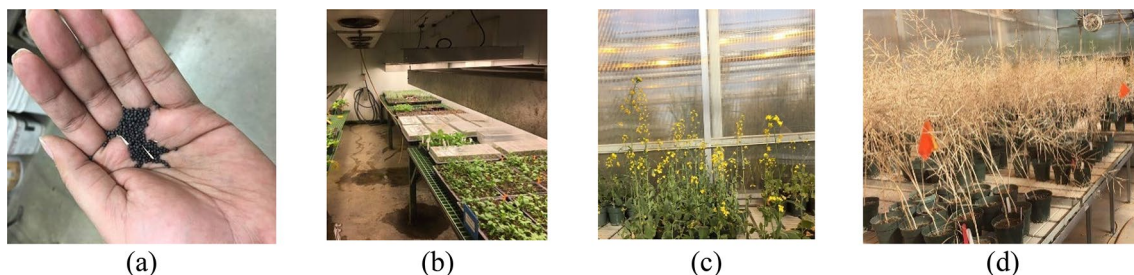


Fig. 2 Canola biomass collection process: **a** canola seeds, **b** germination inside a growth room, **c** flowering of canola plants inside a greenhouse, **d** harvest-ready plants (source of biomass)

Fig. 3 Extracted raw canola fibres **a** from the water-retted plant stems **b**



rigidity of the composite structures. Canola fibre diameter was measured using a FibreShape machine.

2.2.1 Nonwoven mat and composite fabrication

A facile energy-saving route was developed for fabricating the canola fibre reinforced composites to keep the energy consumption to a minimum. The fabrication technique involved two steps: (i) nonwoven mat preparation by mechanical-press into a steel mold, and (ii) applying the MEKP-polymer resin matrix to the reinforcements (canola and wood) (Fig. 4). The composite specimens were dried at ambient temperature, accelerated by the curing agent. Before application into the composite structures, the canola and wood fibres were conditioned in a controlled environment (temperature at 20 °C and relative humidity at 65%). The current design employed high-pressure molding instead of hand-compression for surface consistency and focused on ambient drying to save energy as used by Velmurugan et al. [35] in a recent research work. Five (5) different composite sets (CS# 1–5) were investigated in this current study with different polymer fractions using 3% MEKP mixed resin matrix, as detailed in Table 1. Nine specimens per composite set ($N=9$) were tested in the current research work. 3 sets of pristine composites—which were made without changing the fibre or particle property in any way—are represented by CS# 1 (made of 100% wood particles) and CSs# 2–3 (made of 100% canola fibres). CSs# 4–5 represent the hybrid composites (made of wood and canola fibres).

Canola fibre extraction process is not only labour intensive but the fibre yield (fibre weight: plant stem weight) is also very low, around 6–10%. Hence, the amount of fibre was limited compared to the available wood polymer fraction. As shown in Table 1, the weight fraction of wood was around 10 times higher than canola fibre in the hybrid composite. The idea was to investigate the impact of least affordable canola

fibres, as reinforcements, in the composite structures. Any increase in mechanical properties using the least amount of canola fibre would ensure the influence of canola fibre in a hybrid composite structure and further increase in fibre content would certainly result in better strength and stiffness of the composite.

Although controlling the weight of wood particle was easy, keeping the weight of the nonwoven under control was difficult as it is intrinsically anisotropic. Canola, being a nature-originated fibre, varied in magnitude in different directions of measurements in the nonwoven fabric, contributing towards a slight fluctuation (of canola to wood ratio) between the weight fractions of UPE (~ 1:10) and VER (~ 1:8) resin-impregnated hybrid composites (as shown in Table 1).

The research design aimed to investigate whether a lightweight composite structure could be fabricated having flexural strength comparable to the heavyweight 100% pristine wood composite. To test the hypothesis (i.e., flexural strength as function of fibre/wood content), substantial amounts of wood fraction were replaced with a low-fraction of canola fibre (< 4%) (Table 1) and observed the impact of the canola on the weight and flexural property of the new hybrid (wood-canola) composites. Consequently, the overall canola-wood composite sets (CSs) were lighter compared to the 100% wood composites. Similar approaches of experimenting with varying proportions of wood and fibre contents can be found in different research works including Ahmad et al. [36], who analyzed the influence of weight % of bamboo and wood fiber on composite performance; Chung et al. [37] who characterized the performance of composite structures having varying proportions of bamboo and wood. The pristine composite structures (CSs# 1, 2, 3) show the comparative mechanical strength among each other as well as with the hybrid composite structures (CSs# 4, 5).

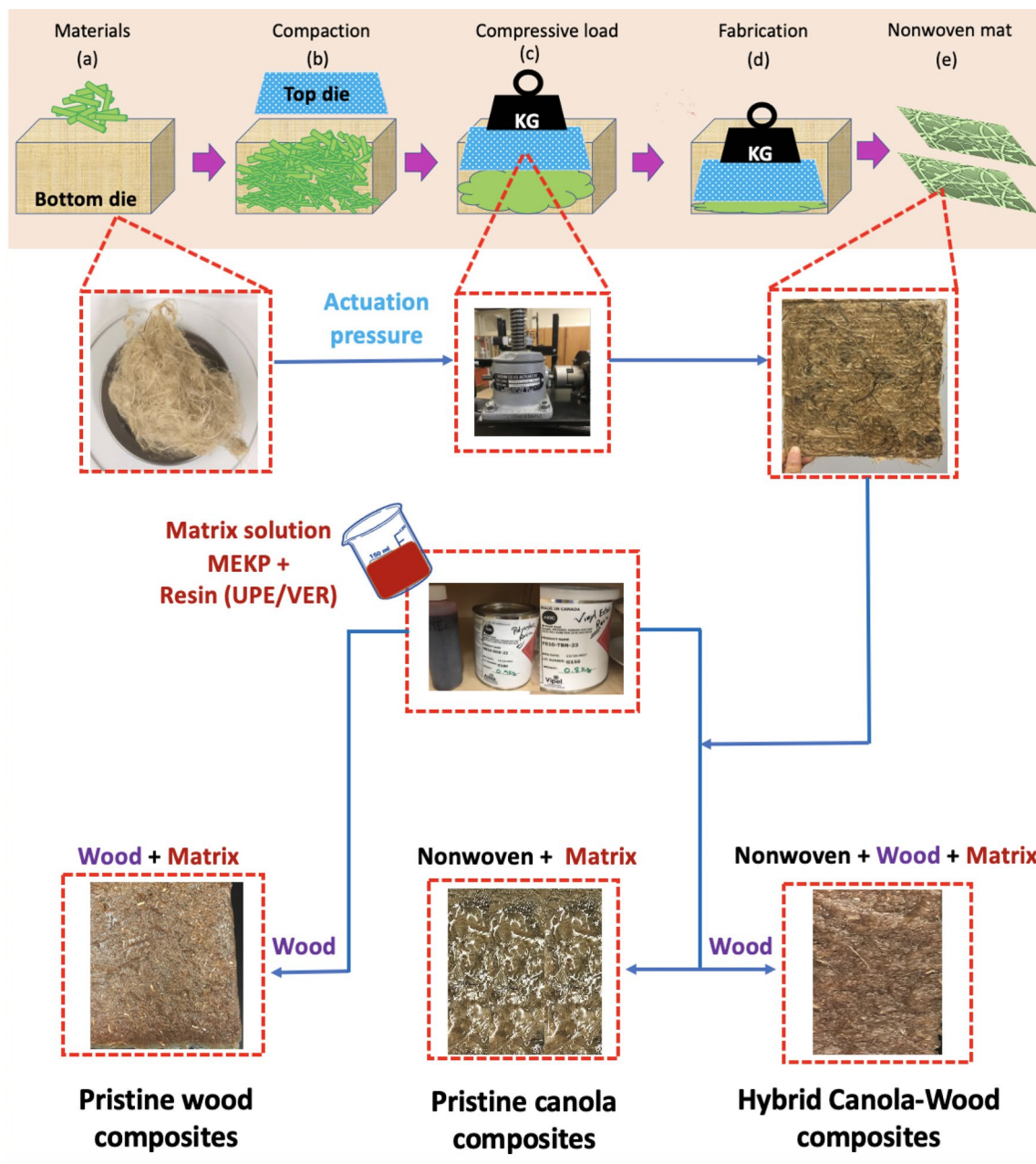


Fig. 4 Mechanics of the nonwoven mat and composite structure preparation at ambient temperature using thermosetting resins (unsaturated polyester / vinyl ester) and curing agent MEKP

Table 1 Five composite sets (CS # 1–5) and their biomass content %

Composite sets # (N=9)	Composite constituents	Reinforcement			Resin matrix	Response
		Canola (g)	Wood (g)	Ratio (canola: wood)		
CS# 1	Pristine Wood+UPE	–	8	–	3% MEKP mixed resin solution	Flexural performance
CS# 2	Canola +UPE	0.4	–	–		
CS# 3	Canola +VER	0.4	–	–		
CS# 4	Hybrid Canola+Wood+UPE	0.4	4	1: 10		
CS# 5	Canola+Wood+VER	0.5	4	1:8		

2.2.2 Flexural strength analysis of the composite structures

Composite strength was measured using three-point bending method according to ASTM D3410/D3410M-16 [34] by a Tension Frame (Model 2017). The specimens were first loaded into the Tension Frame and a compressive load was applied on the specimen at a rate 5 mm/min until the specimen broke down. The flexural rigidity of the specimen was calculated using the following mathematical formulas (Eqs. 1, 2, 3):

$$\text{Flexural Rigidity (F.R.)} = IE(\text{Pa m}^4) \tag{1}$$

$$\text{Moment of Inertia (I)} = \frac{h^3b}{12}(\text{m}^4) \tag{2}$$

$$\text{Apparent Modulus of Elasticity(E)} = \frac{PL^3}{48I\delta}(\text{Pa}) \tag{3}$$

where, F.R. = Flexural rigidity (Pa-m⁴), I = Moment of inertia (m⁴), E = Apparent modulus of elasticity (Pa), P = Breaking load (N), δ = Deflection (mm), L = Specimen length (m), b = Specimen width (m), h = Specimen thickness (m).

2.3 Fibre diameter measurement

Fibre diameters play a significant role in the load-bearing capacity of fibre-reinforced composites. Hence, the diameters of canola fibres were measured using a FibreShape machine (Innovative Sintering Technologies Ltd, Switzerland) [27].

2.4 Failure and reliability analysis using two-parameter Weibull distribution

Due to the intrinsic nature of natural fibre materials, lots of variations exist among the fibre reinforcements, which may ultimately lead to variations of quality in the composite structures. Hence, a failure analysis of this canola fibre reinforced composite structure was further conducted using Weibull probability distribution curve using Weibull equation (Eq. 4).

$$F = 1 - e^{\left(\frac{-\sigma_f}{\sigma_o}\right)^m} \tag{4}$$

where m = Shape parameter (slope);
 σ_f = Applied deflection length; ;
 σ_o = Scaling parameter or characteristic strength; ;
 F = Probability of failure of variable σ_f(F > 0)
 (F = 0.5 means 50% failure whereas 0 means no failure); ;
 m = σ_o = Constant for specimens
 (m also known as Weibull modulus)

Reformulating by taking natural logarithm,

$$\Rightarrow \ln \left(\ln \frac{1}{1 - F} \right) = m \ln (\sigma_f) - m \ln (\sigma_o) \tag{5}$$

By linearizing,
 Y = mX + b if,

$$Y = \ln \left(\ln \frac{1}{1 - F} \right);$$

$$X = \ln (\sigma_f);$$

$$b = -m \ln (\sigma_o)$$

Furthermore, using this Weibull mathematical model, reliability analysis can also be calculated by Eq. 6:

Since, R + F = 1; (R = Probability of survival of variable σ_f; R is also known as reliability)

$$R = 1 - F \tag{6}$$

Alternatively, R can be measured using the obtained values of the two-parameters (m and σ_o) by applying the following Eq. 7:

$$R = e^{\left(\frac{-\sigma_f}{\sigma_o}\right)^m} \tag{7}$$

There are different ways of calculating the value of F which is the function of σ_f i.e., F(σ_f) such as by using Eqs. 8 or 9. In this current study, Eq. 8 was applied following the work of Quinn and Quinn [38].

$$F = \left(n - 0.5 / N \right) \tag{8}$$

$$F = \left(n - 0.3 / N + 0.4 \right) \tag{9}$$

where, n = Rank of nth data point, N = Total number of specimens tested.

2.5 Experimental design flowchart and data analysis

Figure 5 shows the experimental stages of the current research-work. Microsoft Excel software (Version- 2013, Microsoft Corporation, USA) and Analyse-it (Analyse-it Software, Ltd., UK) were used to conduct statistical analysis, t-test (α=0.05), investigate significance of variation, scatterplot, and correlation coefficient (r) analysis.

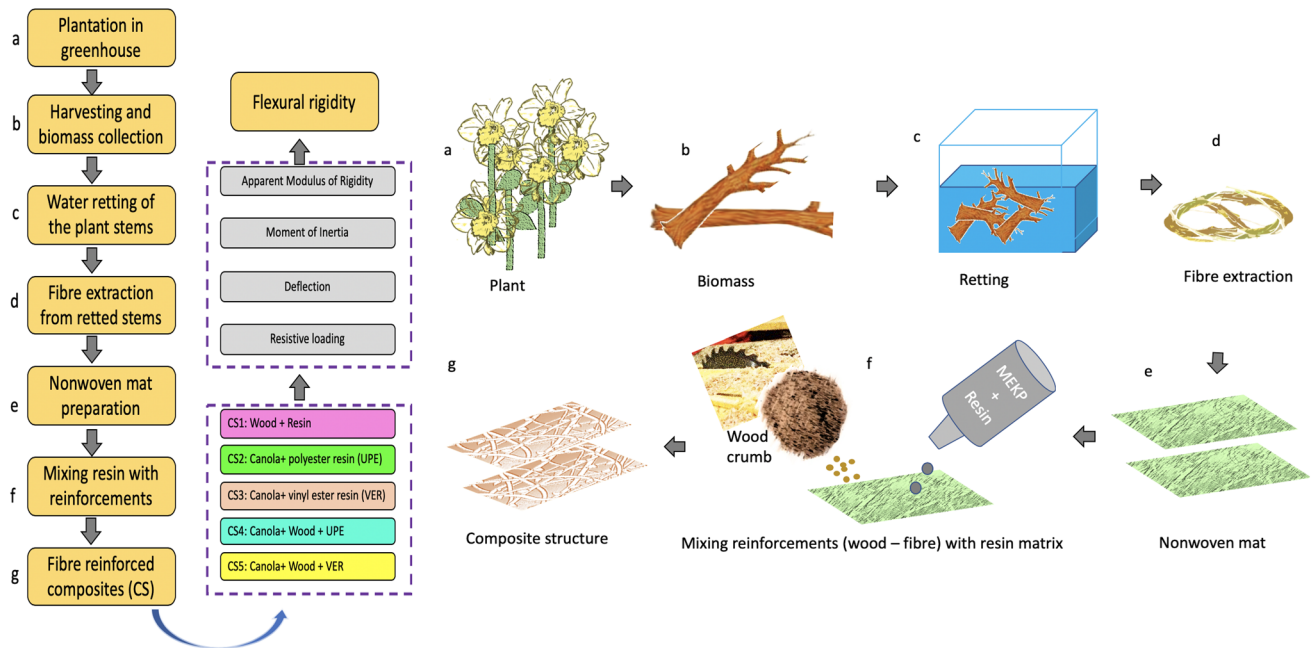


Fig. 5 Experimental design of the current research work. **a** Plantation and harvesting of Topas cultivars of canola (*Brassica napus* L.). **b** Biomass collected post-harvest. **c** Retting of stems in fluid medium. **d** Fibre extraction for optimum fibre yield. **e** Nonwoven mat prepared using extracted lignocellulosic fibres. **f** Synthetic

resin (unsaturated polyester and vinyl ester) mixed with suitable reinforcements (wood crumb and fibre-nonwovens) for composite fabrication. **g** Composite flexural strength evaluated for performance evaluation and statistical analysis

3 Results and discussions

3.1 Fibre diameter distribution in the nonwoven mat

The composite structures derive their strength from the inherent mechanical properties of the fibre reinforcements. Fibre diameters affect the flexural performance of fibre-reinforced composites as they play a critical role in the interphase layer (intermediate between fiber

and matrix). FibreShape provided the minimum, maximum, and average diameter of the canola fibres used for preparing the nonwoven mat: 23.82, 282.34, and 84.30 (± 38.04) microns, respectively (Fig. 6). The total counted objects were 1082, where the mean solidity and convexity parameters were 0.51 and 0.97. In the FibreShape scanner, the size, shape, and optical parameters were weighted by the Geodesic length according to ISO 9276-1 and ISO 9276-2. The presence of fine and coarse fibre diameters gave rise to entanglement between fibres, which assisted in fabricating the

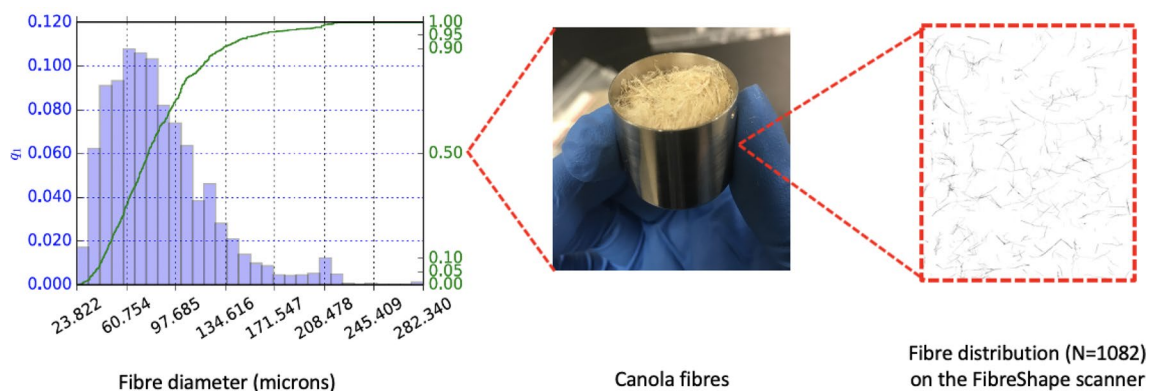


Fig. 6 Fibre diameter (microns) distributions for canola fibres (N=1082)

anisotropic configuration of the nonwoven mat. According to the study of Obukuro et al. [39] flexural strength of a composite structure increases with fibre diameter. In the current study, the combination of coarse and fine fibres influenced the flexural strength of the composite structure, with the former playing the dominant role compared the latter ones. Fibre diameter less than 16 microns makes inefficient fibre impregnation into synthetic resin matrix [39], which was absent in this case since the fibre diameter started with 23.82 microns. The thicker canola fibres (> 16 microns) were densely packed within the nonwoven mat. As a result, the resin distribution throughout the nonwoven surface was not difficult ultimately strengthening the interfacial adhesion between the fibre polymers and the resin matrix for improved composite structure and flexural performance. Obukuro et al. [39] found that flexural strength increased in fibre reinforced composites having fibre diameters ≥ 20 microns, making the canola fibre a suitable choice for fibre reinforced composite structures.

3.2 Hollow canola reinforced light-weight thermoset composites

UPE and VER based thermoset composite structures were fabricated using two different agricultural biomasses, such as the woody biomass fraction and the hollow non-woody canola biomass fraction (as identified by the SEM micrographs). SEM micrographs reveal the intrinsic hollow structure of canola fibres illustrated in Fig. 7. After water retting, the fibres were extracted from the stem (Fig. 7a). The SEM micrographs revealed that the fibres (Fig. 7c) of different diameters (Fig. 7b) e.g., 12.1 μm , 23.13 μm , 7.9 μm , 20.2 μm , 18.1 μm were bundled together. From this fact, the current study infers that canola fibres could display attractive lightweight composite structures. To the best of our knowledge, this is the first study on polymeric composite fabrication using natural hollow textile fibres extracted from any natural biomass. This inner porous layer not only supports the strong wood fraction layer but also complements the overall flexural performance of the composites.

Like canola, the wood fraction also contains cellulosic components. As a structural component, wood biomass also contains xylan (arabino-4-O-methylglucuronoxylan).

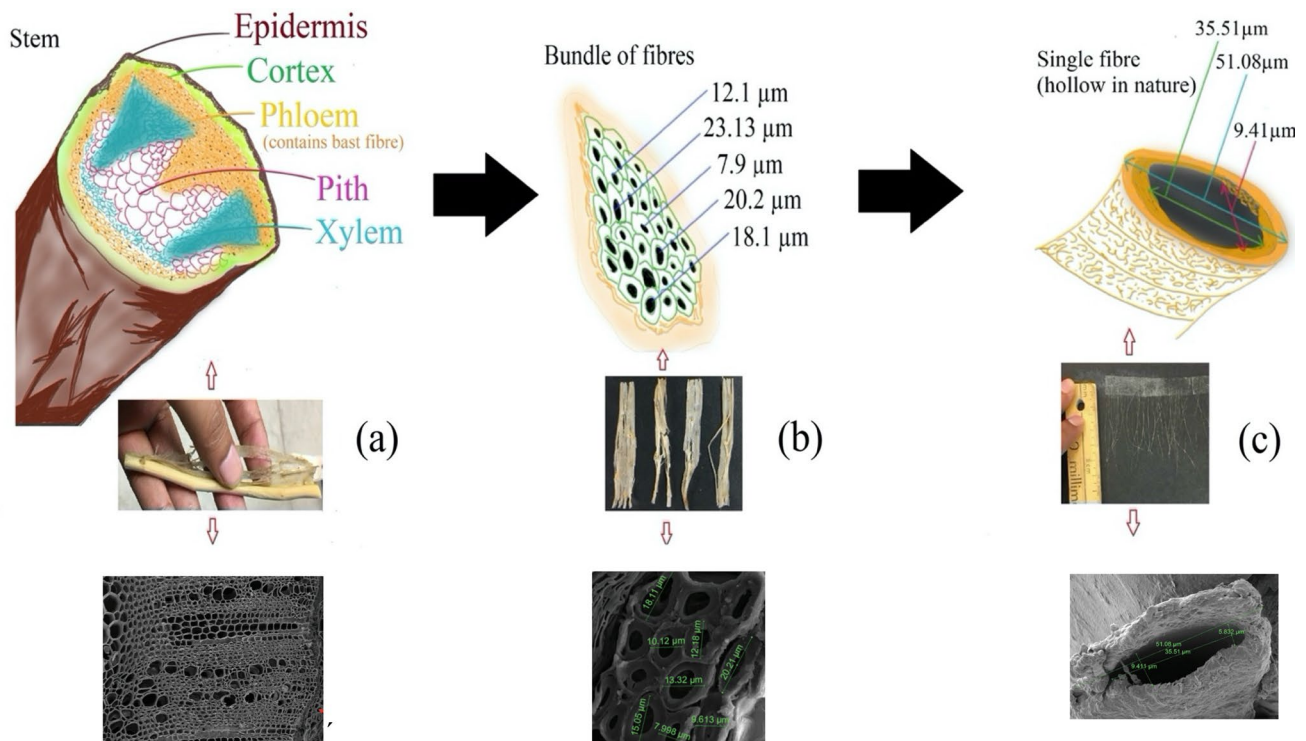


Fig. 7 Images of canola specimens with SEM images. **a** Canola biomass (stem). **b** Extracted fibres from the stem. **c** Inherent hollow canola fibres. Cross-sectional view from the corresponding SEM micrographs confirm the intrinsic structural hollowness of canola fibres with different size or shape on plant stem, fibre-bundle com-

prising hollow fibres of different diameters from extracted from stem exterior, and individual fibre taken at a magnification level of $200\times$ **a**, $1600\times$ **b**, and $3000\times$ **c**, respectively (SEM images are reproduced with permission [27]. Copyright 2019, SAGE Publications)

The introduction of woody fraction imparts improved performance to the overall composite structure since the woody biomass is a good resistant of hydrolysis reaction [40]. Further, the wood fraction also acts as a strong reinforcement alongside the fibre fraction. The fibre-matrix interfacial bonding was associated with the available hydroxyl groups [41] and the inherent hollow structure of the canola fibres was responsible for the improved lightweight structure of the thermoset composites. Figure 8 displays the hypothesis towards a general overview of the chemical mechanism involved during fabricating the canola-wood composites based on the participating elements: (i) cellulosic canola fraction; (ii) wood fraction; (iii) the thermosetting UPE resin; and (iv) catalyst MEKP (methyl ethyl keton peroxide) as the curing agent at ambient temperature.

Aerospace industries are heavily investigating hollow composite structures including the application of embedded hollow glass fibres with self-healing agents to develop self-healing composites and laminates [26]. The current study opens a new door for fabricating functional and self-healing canola based natural composite structures by embedding healing agents containing hollow reinforcements. The intrinsic hollowness of canola fibres would allow the healing agents to flow and fill the cracks to prevent any damage propagation. Future work can investigate the possibility of fabricating self-healing functional natural fibre composites by harnessing the hollow structure of canola fibres. Such natural functional composites would extend the service life and will promote recycling and sustainability.

3.3 Flexural rigidity and resistive load of the canola reinforced composites

Decortication involves a mechanical procedure for extracting the restrictive layer of technical bast fibres overlying the plant stems. This process is widely used for industrial bast fibres, such as flax, hemp, and so forth. Unlike the mechanical decortication process, solvent-retted fibre tissues have better surface uniformity and evenness since the fibres are not damaged during the decortication process. By using this technique, canola fibres with minimal inhomogeneity were obtained for composite structures. Also, as mentioned earlier, the breaking tenacity of canola fibres are lower compared to flax or hemp. Due to the lower tenacity, the mechanical decortication process could eventually damage the canola fibres and reduce the load-bearing capacity in the composite structure. Besides, the lightweight hollow canola fibres may not survive the decortication cycles and could end up producing short fibres and lowering the overall fibre yield (%), required for successful composite designing process. Consequently, fibre damage and short fibre associated with the mechanical extraction technique could counteract the benefits of the procedure. Nevertheless, clear advantages concerning the overall flexural performance arise upon the introduction of solvent-retted hollow canola fibres. Hence, using the solvent-retting technique, it is possible to produce canola fibre reinforced lightweight composite structures with a breaking load and flexural rigidity above 700 N and 6.5 Pa·m⁴, respectively. Surprisingly, relatively stronger peaks of flexural rigidity were observed for composite structures fabricated with the solvent-retted canola fibres than the composites with 100% wood particles. Table 2 represents the flexural rigidity (FR) and breaking load (BL) of the five different composite structures (CS #1, CS #2, CS #3, CS #4, CS#5) prepared from canola fibres and wood

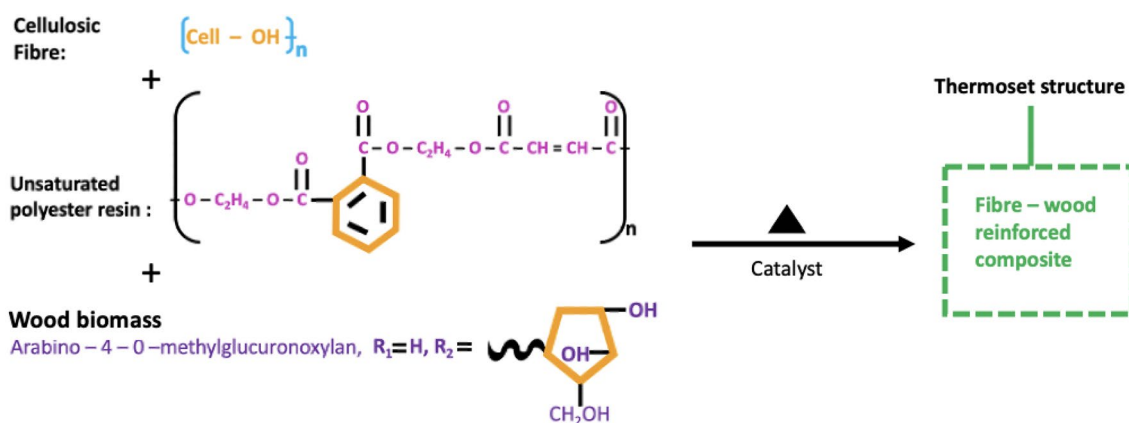


Fig. 8 A general overview of the chemical reaction involved in synthesizing the cellulosic natural fibre-wood reinforced composites using unsaturated polyester resin and methyl ethyl keton peroxide catalyst

Table 2 Flexural rigidity and breaking load of the five manufactured composite-sets (CSs) (CS1: wood-polyester resin; CS2: canola-polyester resin; CS3: canola-vinyl ester resin; CS4: wood-canola-polyester resin; CS5: wood-canola-vinyl ester resin)

Composite sets #	Polymer constituents	Breaking load (N)	Flexural rigidity (Pa·m ⁴)
CS# 1	Wood + UPE	585 ± 130	5.9 ± 3
CS# 2	Canola + UPE	92 ± 15	1.04 ± 0.3
CS# 3	Canola + VER	121 ± 13	1 ± 0.2
CS# 4	Canola + Wood + UPE	549 ± 99	6.4 ± 2.5
CS# 5	Canola + Wood + VER	728.35 ± 242	5.6 ± 2.7

crumbs using the polymer matrix systems; Table 3 demonstrates their statistical analytics.

Inelastic damage took place during the flexural loading. The pristine wood reinforced composites resulted in a relatively lower flexure performance compared to the hybrid canola reinforced composites. The incorporation of canola fibres made this remarkable difference in mechanical performance at a reduced wood consumption. This superior strength to weight ratio of the lightweight canola composites is attributed to few major factors including the mechanical property of the solvent-retted canola fibres, the aspect ratio, diameter, density, and fibre volume-fraction of the produced composite structure. The lightweight composite structure was in direct linear response to the lower density of the fibre. Canola composites with better dimensional stability were achieved by incorporating the solvent-retting technique to produce canola fibre reinforcements with minimal damage. As a result, the solvent-retted canola fibre – in this research work – provided good reinforcement properties and successfully carried the applied compressive stress, and thereby, exhibited satisfactory breaking load. The fibre aspect ratio (i.e., the higher length to diameter ratio) of the canola fibres enhanced the fibre entanglements, facilitating a strong inter-connected fibre network in the nonwoven fabric, which ultimately optimized the mechanical properties of the canola fibre reinforced composite structures. The strength and stiffness of polymer-matrix composites would increase with an increase in the fibre content (%) available as reinforcements. The diameter of the Topas fibres lies in between

23.82 and 282.34 µm, which is comparatively lower than other bast fibres like hemp (25–500 µm) and flax (40–600 µm) [25]. Further, the density of canola is lower than other bast fibres like flax and hemp. As a result, for a given area and weight, Topas canola fibre is capable of constructing a superior packing density inside the non-woven mat and composite due to its lower density and diameter, which, in theory, gives rise to higher fibre volume fraction. Such a configuration provides a higher surface area to spread the fibre-resin interfacial loads. Consequently, the closely packed canola fibres exhibited superior mechanical properties. The canola fibre diameter improved the overall fibre-resin interfacial adhesion, allowing the canola composites to withstand the applied load and preventing its fracture until the flexural performance exceeded the threshold displayed by the pristine wood-resin composites. From a mechanical viewpoint, both UPE and VER resin matrix system can be applied with the expectations of good flexure performance in the canola reinforced composites. Observed variations between the UPE- and VER-mixed hybrid canola composites were not significant in terms of flexural rigidity.

The current study demonstrated that mean BL and FR of the wood-UPE resin composite structures (CS #1) are 585 ± 130 N (min: 441.79 N, max: 695.14 N) and 5.9 ± 3 Pa·m⁴ (min: 3.25 Pa·m⁴; max: 9.27 Pa·m⁴). Similarly, the mean BL and FR of the canola-unsaturated polyester resin (CS #2), canola-vinyl ester resin (CS #3), canola-wood-unsaturated polyester resin (CS #4), and canola-wood-vinyl ester resin composite structures (CS #5)

Table 3 Significance of variation analysis among the pair of means of flexural rigidity (FR) of the composite structures using t-test

Group 1	Group 2	p-value*	Significant variation
Canola + UPE	Canola + VER	0.34	Negative ($p > \alpha$)
Canola + UPE	Wood + UPE	0.03	Positive ($p < \alpha$)
Canola + VER	Wood + UPE	0.03	Positive ($p < \alpha$)
Wood + UPE	Canola + Wood + UPE	0.42	Negative ($p > \alpha$)
Wood + UPE	Canola + Wood + VER	0.44	Negative ($p > \alpha$)
Canola + Wood + VER	Canola + Wood + UPE	0.36	Negative ($p > \alpha$)

*p-value was obtained after conducting t-test for testing the significance of variation between two means, assuming equal variances at $\alpha = 0.05$

were found to be 92 ± 15 N (min: 74.58 N, max: 103.59 N), 1.04 ± 0.3 Pa·m⁴ (min: 0.74 Pa·m⁴; max: 1.26 Pa·m⁴); 121 ± 13 N (min: 104.37 N, max: 136.11 N), 1 ± 0.2 Pa·m⁴ (min: 0.87 Pa·m⁴; max: 1.13 Pa·m⁴); 549 ± 99 N (min: 460.24 N, max: 654.73 N), 6.4 ± 2.5 Pa·m⁴ (min: 4.39 Pa·m⁴; max: 9.20 Pa·m⁴); and 728.35 ± 242 N (min: 448.67 N, max: 876.38 N), 5.6 ± 2.7 Pa·m⁴ (min: 2.79 Pa·m⁴; max: 8.24 Pa·m⁴), respectively. It was seen that CS #3 displayed the lowest and CS #4 highest mean FR among all five CSs. It can be also seen that FR of a canola composite structure is always higher when canola nonwoven fabrics are used with the UPE resin system (CS #2, #4) than the VER resin systems (CS #3, #5) with or without wood crumbs (FR of CS #2 > CS #3 and FR of CS #4 > CS #5). Further, CS #4 provided a higher FR than CS #1 and FR of CS #5 is nearly equal to CS #1. The pristine canola composites (CS #2, #3) exhibited lower FR and BL compared to the pristine wood composite structures (CS #1). Hence, adding the wood fraction with the pristine canola composites can significantly increase the flexural strength; alternatively replacing wood fraction with fibre polymer fraction increases the flexural strength, as exhibited in the hybrid CSs #4 and #5. The improved strength observed from hybrid composites could be attributed to the increased fibre polymer fraction. The superior breaking tenacity (12.59 g-force/tex) of the canola fibre could have contributed towards the increased mechanical strength of the hybrid composites. Further, the fibre aspect ratio could have played a positive role in increasing the strength of the hybrid composites, which produced a robust inter-fibre bridging to constrain the crack propagation and provide lateral confinement in the hybrid composites with a reduced wood fraction. Owing to the bridging action among the canola fibres, the hybrid composite structures maintained good structural integrity, contributing towards a strong flexural rigidity and breaking load. As a result, the ductility of the hybrid composites might have been better compared to the pristine composites. However, the assumption should be further refined in future works. It was interesting to see that CS #4 exhibited better flexural property compared to CS #5 – a behaviour which may be explained by the entanglement of fibres. Increased fibre content increases the void content, which in turn decreases the mechanical strength of fibre reinforced composite structures [42]. Consequently, the flexural property can be improved with increasing wt% of the canola content and decreasing wt% of the wood content in a canola-wood composite structure. The current study also speculates that a better wetting behavior of UPE resin with canola fibre played a dominant role in bolstering the flexural properties of the composite structures than the VER resin matrix. Hence, it could be concluded that the choice of resin system is a critical factor for fabricating canola-wood composites, possibly due to the difference

of molecular weight between the resin matrix systems. Kim et al. [43] reported similar observations regarding the influence of resin system on the flexural properties of wood-cotton composites.

Table 3 displays the significance of variations among the mean of the flexural rigidity (FR) of the five different composite sets (CSs). It was found that there is no significant variation among CS #1, CS #4, and CS #5 or between CS #2 and CS #3. Therefore, it was displayed that, if the amount of wood content is reduced and replaced by a single layer of canola non-woven fabric (accounting for 2.5–3.5% canola fibre content), not only the overall weight of the composite structure is reduced (CS #4, #5) but also a comparable FR to that of 100% wood composites (CS #1) can be achieved. This observation was confirmed by the t-test comparisons reported in Table 3. Further, it was also interesting to observe that by replacing about half of the wood mass inside a composite structure (CSs# 1, 4, 5), a similar and superior mean breaking load can be achieved (Table 2).

3.4 Stiffness (or MOE: modulus of elasticity) of the composite structures

Canola fibres can be used with UPE and VER resins without any prior pre-treatment process with good mechanical properties, provided that MEKP curing agent is used. Most of the natural fibre tissues had variability in length. However, the average length of the solvent-retted fibre unit was 7 cm, which was strongly bonded together. The interfacial bond within the solvent-retted fibre bundles could be probably higher compared to the interfacial bond between fibre and resin, contributing towards a strong mechanical performance. Solvent retting caused the fibre tissues to become more open due to the microbial activity in the retting bath. As a result, a strong interface between the fibre and resin matrices took place, and these cellulosic natural fibres acted as a good reinforcing agent for an effective modulus property.

The hybrid canola fibre-wood reinforced composite structures (CS# 4, 5) were marked displaying comparable stiffness (MOE) (GPa) properties to the composite structures fabricated with 100% wood biomass (CS #1) only. Figure 9 displays the comparative assessment of stiffness (or modulus) performance of the composite structures. The content (%) of wood was substantially low in the composite specimens CS# 4 and #5: overall average structural weight 15.57% and 28.38%, respectively, less than the wood-resin composite specimens of CS #1. However, it was an excellent observation that neither the low structural weight nor the low wood content (%) affected the flexural performance of the fibre reinforcement composite structures. In fact, the CS# 4 exhibited 5.94% higher stiffness

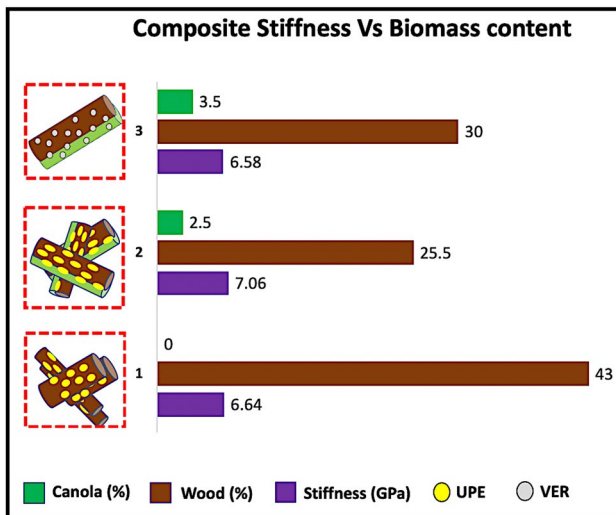


Fig. 9 Modulus of elasticity (MOE) or stiffness (GPa) of canola fibre reinforced composite structures for different mix proportions of biomass reinforcements: 1) wood-polyester CS #1; 2) wood-canola-UPE CS# 4; 3) wood-canola-VER CS #5)

than the wood-only composites, CS #1; whereas the difference was < 1% between the stiffness of CS #1 and CS #5. The mean values of the composite stiffness were reported $6.64(\pm 1.80)$ GPa, $7.06(\pm 1.70)$ GPa, and $6.58(\pm 1.93)$ GPa, for the CS #1 (wood content: 43%), CS #4 (wood content: 25.5%; canola content: 2.5%), and CS#5, (wood content: 30%; canola content: 3.5%), respectively. It can also be seen that the hybrid composites exhibited higher stiffness propensity with the UPE matrix system (CS #4) compared to the VER resin (CS # 5). Surprisingly, this reported MOEs of the canola-wood composites were superior to the reported MOE performances of wood-flax (2.72 GPa), wood-hemp (2.06 GPa), or shive-flax (4.02 GPa) structures [45]. The behavior of the improved flexural stiffness with a lower wood fraction of the hybrid composites is similar to that of the flexural rigidity. The advantage of improved stiffness could be a result of incorporating canola fibres. Since canola fibre has a lower diameter (than the major bast fibres, for example, jute, hemp, or flax), the surface area of fibre is high in the composite structure. As a result, canola fibre-reinforced composite structures presented better stress-bearing behavior, and thereby, stiffness at a lower wood fraction compared to the pristine composite structures. Further, we assume that the dependency of natural canola fibres on multiple factors contributed to the flexural behavior of the reinforced composites: aspect ratio, surface morphology, arrangement, treatment time, processing route, and choice of the resin matrix.

An exothermic curing technique was applied to polymerize the UPE and VER resin system while fabricating the canola fibre reinforced composites. This room temperature

curing process is cost-effective as it doesn't require any additional hot-pressing processing step. For the first time, the current study explored the possibility of using this curing technique with the natural canola fibres and successfully applied it with multiple synthetic resin systems. As a result, an easy processability was obtained in the current work. Further, the demand from OEMs for ambient temperature curing is increasing constantly. Fibre composite industries have also aroused considerable interest for ambient curing technique in recent time. In current research work, the UPE and VER resin systems were polymerized at ambient temperature using the MEKP curing agent. Once the curing process initiated, the heat transfer through the fibre-resin network quickly solidified the composite structures. Since the variation between the UPE- or VER-mixed composites wasn't significant, it is worth noting that the current ambient MEKP curing process can be used for canola with both the UPE and VER resin systems at a similar mechanical performance. Interestingly, Topas is a commercial cultivar of canola, which is being cultivated and harvested by the agricultural farming industries at a commercial scale. The experimental results also exhibited that the Topas cultivar is a good candidate – among different canola cultivars – that could undergo an exothermic chemical reaction without compromising the structural integrity or mechanical performance of the canola composite structures.

3.5 Mono-plot vectors and correlation coefficients among composite parameters

Interestingly, a strong correlation was observed between the flexural rigidity and deflection of the composite structures (canola-wood-resin). Pearson's coefficient $r = -0.77$ (with an R^2 value of 0.70) (Fig. 10) depicts a phenomenon – the higher the flexural rigidity, the lower its bending tendency. Further, the apparel modulus (stiffness) showed a positive correlation with resistive load ($r = 0.65$) as well as with the flexural rigidity ($r = 0.69$) of the composite structures.

Mono-plot was also constructed to verify and validate the correlations being demonstrated, which would reduce the space to represent the identical behavioral patterns among variables. Figure 11 displays that the deflection and flexural vector is closest to the periphery telling that these two variables account for much of the variance in the vector configuration, where the principal component one (PC1) and principal component two (PC2) in combination represent 94.2% of the total data-set variance. In a mono-plot, the correlation between two vectors is: positive if the angle between them is small; zero if 90° ; and negative if 180° . Since the angle between the two vectors (deflection and flexural rigidity) is closer to 180° , the relationship is

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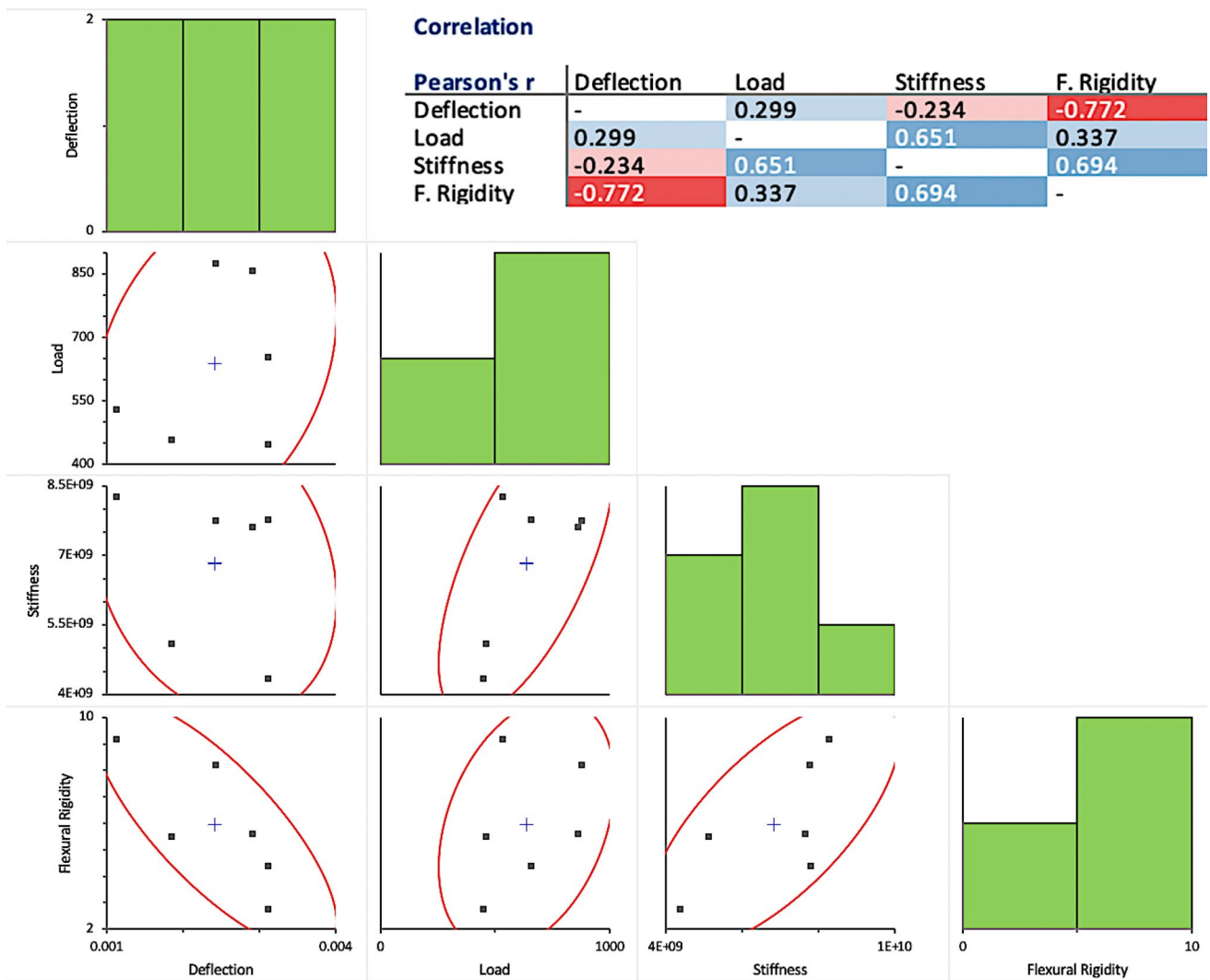


Fig. 10 Scatterplot and correlation coefficient showing the distribution and relationship between different mechanical parameters of the canola-wood-resin composite structures

inverse or negative, which is validated by the value of Pearson's coefficient r , as discussed above. Similarly, apparent modulus (stiffness) and flexural rigidity vectors are correlated positively as the angle between them is around 40° .

3.6 Weibull distribution analysis for probabilistic deflection of composites

A failure analysis of this canola fibre reinforced composite structure was further conducted using two-parameter Weibull distribution (Fig. 12a). The shape parameter (also known as Weibull modulus) of the composite structure was the slope, $m = 3.23$, which is quite typical for materials with natural variation (ranges around less than 7). The intercept was $b = -3.11$, a constant for the specimen.

Solving the intercept b , the value of σ_0 (scaling parameter) was found to be 0.38. A smaller shape parameter (m) would refer to a higher scattering in the dataset i.e., deflection length; whereas, a larger value of shape factor (m) would indicate a steeper slope referring a lower variability among the population dataset. Interestingly, the average deflection was found to be $2.33 (\pm 0.75)$ mm at failure ($N = 12$). If the average Weibull deflection length is in close proximity to the experimental average deflection length, then the approximate deflection length can be predicted from the fitted curve for analyzing failure probabilities at different percentiles.

The Weibull distribution graph gives a probabilistic performance for failure rate analysis of the engineered product, the canola composite structure in this case. The failure

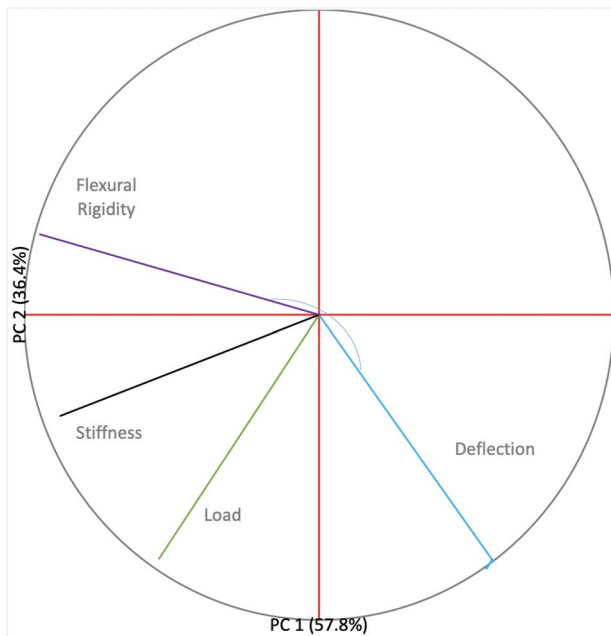


Fig. 11 Mono-plot of different variables (deflection, flexural rigidity, stiffness, load) of R Mode PCA canola-wood-resin composite structures

rate (F) refers to y-axis values and the corresponding failing deflection length (to its natural logarithm) is given by the x-axis values. For instance, 50% survival probability for specimens lies below the deflection length of 2.34 mm upon external loading, which could be calculated using Eq. 5. The average (at $F=0.5$) Weibull deflection length was found to be 2.34 mm. Hence, it can be seen that an excellent agreement has been obtained since the difference between Weibull and experimental average deflection length is only 0.01 mm ($= 2.34 - 2.33$). This result suggests that the two-parameter Weibull probabilistic distribution could be used for predicting the yield deflection to external loading for composite structures.

$$\ln \left(\ln \frac{1}{1 - 0.5} \right) = 3.23X - 3.11$$

$$\Rightarrow X = -0.85$$

$$\Rightarrow \ln(\sigma_f) = 0.85$$

$$\Rightarrow \sigma_f = 2.34 \text{ mm}$$

Statistical analysis of experimental data at 95% confidence interval also shows a good fit with Weibull distribution, having a value of $R^2 \geq 0.92$. The yield deflection at a level of 95% probability i.e., the failure deflection length achieves maximum values of 3.67 mm. Similarly, to avoid any failures above 1 percentile during composite fabrication, an engineer could design the composite in such a manner that the specimen would possess the inherent

resistive loading to restrict its deflection within 0.63 mm. In this way, an engineer or a scientist could make a prediction about the performance or failure. For instance, at what optimum deflection (mm) should an engineer design (the canola composite structures) in order to have the probability of failure of only 1 in 1 million specimens. The solution to this mathematical question is given below using the Eq. 5 of this current research work.

$$\ln \left(\ln \frac{1}{1 - \frac{1}{10^{-6}}} \right) = 3.23X - 3.11$$

$$\Rightarrow X = -3.28$$

$$\Rightarrow \ln(\sigma_f) = -3.28$$

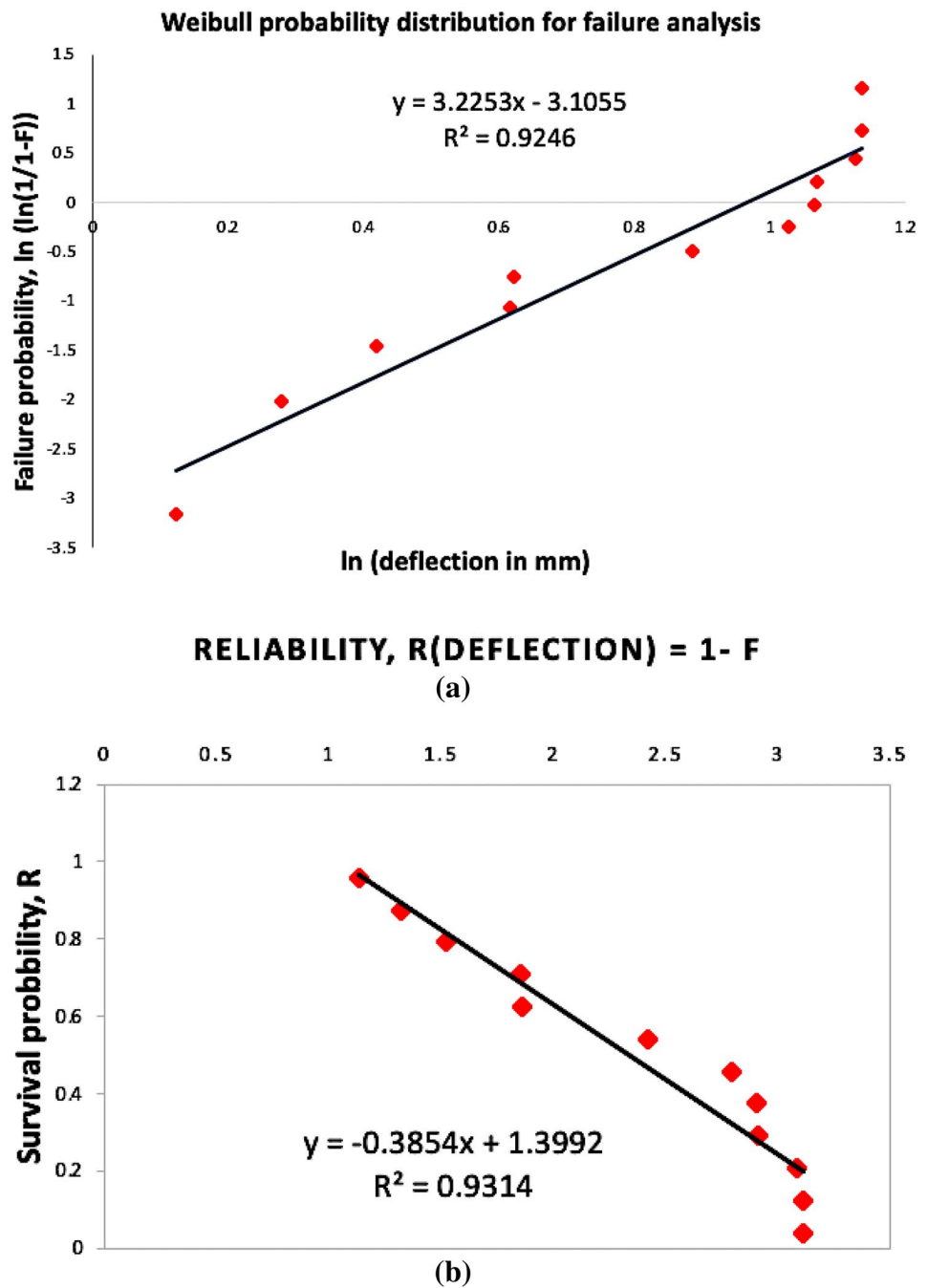
$$\Rightarrow \sigma_f = 0.04 \text{ mm}$$

However, this is a probability and the probability would be more approximate if a larger dataset could be used for reducing any trace of residual error, which is one of the focuses for future works. This probabilistic analysis is only applicable as long as the data fits this mathematical Weibull model. The model can be also used to predict the failure rate (F) (0–100%) at a given deflection length (X or σ_f) by solving for Y or by directly using Eq. 4 using the value of shape parameter (m) and scale parameter (σ_0). Furthermore, the probabilistic survival deflection length of the composite structures was analyzed using Eq. 6 as illustrated ($R^2 \geq 0.93$) in Fig. 12b. Markedly, the shape of both the failure and reliability curves resembled the findings of the current research works by Roy et al. [30] and Sayeed and Paharia [31].

4 Concluding remarks and future work

The research work hypothesized that the inclusion of canola as reinforcement with wood particles in a composite fabrication could increase the flexure properties more than wood particles alone. A facile composite fabrication process successfully demonstrated this to be so. This infers the suitability of applying novel canola fibre to produce lightweight composite structures with a reduced wood content, having superior flexural rigidity, breaking load, and bending stiffness. Canola fibre exhibits a minimum fibre thickness of 23.82 microns making it suitable for interfacial adhesion with resin matrix for composite fabrication. UPE resin demonstrated a better wetting behavior for the canola fibres compared to VER resin matrix system. UPE mixed hybrid canola-wood composite structures exhibited a mean flexural rigidity of $6.4 \text{ Pa}\cdot\text{m}^4$, outperforming the pristine wood composites that displayed a mean value of $5.9 \text{ Pa}\cdot\text{m}^4$. The demonstrated work is the first work on

Fig. 12 Weibull distribution for failure, **a** and probabilistic reliability, **b** analysis of the deflection length of canola-synthetic resin composite structures



canola fibre reinforced hybrid composites. The architectural hollow structure of canola also opens a new possibility of fabricating self-healing natural fibre composites by harnessing the inherent hollowness of canola fibres.

Future work should concentrate on SEM (Scanning electron microscope) micrograph analysis for a better understanding of the interfacial adhesion between the reinforcements (canola biomass, wood crumbs) and matrix system (UPE resin, VER resin). FTIR (Fourier-transform infrared spectroscopy) analysis may demonstrate the nature of interaction between chemical groups of

fibre-reinforcements and resin matrix; whereas, SEM analysis can exhibit the strength of this interaction. Further, Instron or similar machine can be used to investigate the tensile strength of the composites. However, it would also be interesting to investigate the effect of different variables (different resin-MEKP recipes, shelf time inside the fume hood, amount of reinforcements, different canola cultivars) on the performance of the canola composites. Advanced nonwoven manufacturing technologies, for example, needle-punching or carding and composite manufacturing processes like VARTM (vacuum assisted

resin transfer molding) or compression molding methods could be used for a faster and more efficient production process of canola nonwoven mat and composite structures at an industrial level. IEM (isocyanatoethyl methacrylate) can be used in treating the canola fibres prior applying the resins. Research works have indicated that IEC could improve the interfacial adhesion between cellulosic fibres and unsaturated polyester resin.

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Data Availability All data generated or analyzed during this study are included in this article.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethical approval The research performed by the author does not contain any studies on human participants or animals.

Consent for publication I, Ikra Iftekhar Shuvo (IIS), the author, hereby declare that that this is my study; I developed the manuscript titled 'Hollow Canola-Wood Thermoset Composites from Concept to Completion: Fabrication, Performance, Failure & Reliability Analysis'.

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