Integration and mechanical characterization of PALF and jute fibre in epoxy polymer to fabricate structural hybrid biocomposites

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A report submitted in partial fulfillment of the requirement for the degree of Bachelor of Science in Mechanical Engineering

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Candidate's Declaration

This is to certify that the work presented in this report, titled, "**Integration and mechanical characterization of PALF and jute fibre in epoxy polymer to fabricate structural hybrid biocomposites**", is the outcome of the investigation and research carried out by me under the supervision of **Prof. Dr. Mohammad Ahsan Habib, PhD**

It is also declared that neither this thesis nor any part of it has been submitted elsewhere for the award of any degree or diploma.

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RECOMMENDATION OF THE BOARD OF SUPERVISORS

The thesis titled "**Integration and mechanical characterization of PALF and jute fibre in epoxy polymer to fabricate structural hybrid biocomposites**" submitted by **Tajwar Azim Baigh** (Student No: **180011104)** & **Fairooz Nanzeeba** (Student No: **180011214)** has been accepted as satisfactory in partial fulfillment of the requirements for the degree of BSc. in Mechanical Engineering.

BOARD OF EXAMINERS

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Acknowledgment

A marathon is the result of the accumulation of thousands of steps. This project, for us, was the equivalent of running a marathon. From trying to find out research gaps to work on, to ordering our equipments, to failing our first experiment to finally making the biocomposites. Between juggling the project with our studies, we learned more about real life and discipline than we could have ever imagined. First and foremost, we thank Almighty Allah (SWT) for giving us the opportunity and the drive to keep on working when things were tough. We are deeply grateful to everyone who has provided us support and mentorship, enabling us to successfully complete our thesis within the allotted time.

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Abstract

The field of materials science has made significant advancements in green technology through the development of biocomposites, driven by environmental and sustainability concerns. The purpose of this study is to fabricate hybrid biocomposite of thermoset polymer epoxy, reinforcing it with PALF and jute fabric that are used in the textile industry to analyse their mechanical properties as well as water absorption characteristics to compare the results with pure pineapple leaf reinforced polymer and pure jute reinforced polymer composites. Hybrid composites with 4 different stacking sequences and two pure composites were fabricated by wet lay-up technique followed by vacuum bagging technique following the latest ASTM standards. The findings reveal that combining jute with PALF improves tensile and flexural characteristics, outperforming pure PALF composites and slightly less than pure jute composite. The highest tensile strength was observed in JFRP (35.16 MPa), and the highest tensile strength for the hybrid composite was observed to be 32.16 MPa. The highest tensile modulus in hybrid composite 4P5J – 2 (1.315 GPa). Hybrid composite 4P5J – 4 exhibited the highest elongation (15.94%). Among the hybrids, 4P5J – 1 demonstrated the best flexural strength (44.36 MPa), equivalent to pure jute composites with a 78.57% increase in flexural modulus. In terms of impact strength, PFRP performed best, but the hybrids still outperformed JFRP. The choice of stacking sequence significantly impacts composite fabrication outcomes. These results suggest a greener future for the development of hybrid composites reinforced with natural fibres from textile industries for uses in the automobile sector, specifically in making levers such as brake and accelerator pedals. It also shows a way of using waste materials of the textile industries as natural reinforcements for fabricating biocomposites.

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Chapter 1

Introduction

Over the last half – century, research and engineering have shown great emphasis to fibre – reinforced polymer-based structural materials over monolithic materials due to their unique advantages of high strength to weight ratio, non – corrosive property and high fracture toughness. These composite materials consisted of high strength fibres such as carbon, glass and aramid, and low strength polymeric matrix, now have dominated the aerospace, leisure, automotive, construction and sporting industries. Unfortunately, these fibres have several major drawbacks such as (i) non-renewable, (ii) non-recyclable, (iii) high energy consumption, (iv) health risk during manufacturing process and (v) non - biodegradable. Recently, due to a strong emphasis on environmental awareness worldwide, it has brought much attention in the development of recyclable and environmentally sustainable natural composite materials (Cheung et al., 2009). Natural materials, while being simple components with poor intrinsic mechanical properties sometimes possess extraordinary properties and perform their own function due to their unique microstructure (Cho et al., 2017). Evolutionary selection further made sure that structural materials such as natural fibres and biocomposites attain superb levels of efficiency and performance (O'Brien et al., 1998)

1.1 Natural fibres

World 2022 Natural Fibre Production was estimated at the end of November at 32.9 million tonnes, essentially unchanged from 2021 (*December-2022-The-Global-Market-for-Natural-Fibers-2022-1*, n.d.). The wastage of agriculture and forests (30-40%) can be used to make different value-added items (Jalil et al., 2021). By 2024, the market for natural fibre-reinforced composites is predicted to reach $10.89 billion (F. M. Khan et al., 2022). Natural plant fibres are used frequently because they are inexpensive, strong, light, have a high specific strength and modulus, are biodegradable, and provide superior thermal and acoustic insulation (Hasan et al., 2021; T. Li et al., 2018; Mohammed et al., 2015; Pickering et al., 2016). Additionally, some plant fibres' mechanical attributes can even rival those of glass fibres (Kalali et al., 2019; Ying-Chen et al., 2010).

Figure 1 shows a hierarchy of several natural fibres that were used to make fibre – reinforced composites (Chen et al., 2014; Dittenber & Gangarao, 2012; Faruk et al., 2012; "Handbook of Natural Fibres," 2020; Kalia et al., 2009; Y. Li et al., 2000; W. Liu et al., 2005; Pickering et al., 2016; Prakash et al., 2011; Siakeng et al., 2018; Ying-Chen et al., 2010).

1.1.1 Harvesting and processing

Primary plants are produced for their fibres (jute, hemp, kenaf, elephant grass & sisal), while secondary plants are the ones where the fibres are by – products (pineapple leaf fibre (PALF), coir, corn husks & oil palm).

There are several stages of fibre or fabric production. They are: (i) plant growth, (ii) harvesting, (iii) fibre extraction (iv) Processing (v) Supply. A definition of each stage is given in Table 1. Factors such as: species, location, type of cultivation, climate, ripeness, type of extraction, transportation and storage conditions, can affect fibre quality (Dittenber & Gangarao, 2012). Even after careful harvesting and processing, the practical yield of fibres is not 100%.

Table 1: Stages of fibre production *(Dittenber & Gangarao, 2012)*

Stage	**Definition**
Plant growth	• Planting seeds • Crop cultivation
Harvesting	• Extraction of plants from soil or water using hand tools or machinery
Fibre extraction	• Mechanical decortications, dew retting or water retting which separates the useful fibres from the non – useful biomass (Faruk et al., 2012)
Processing	• Wash and dry • Cutting into short fibres • Conditioning (chemical, physical treatment etc) • Spinning and weaving (if fabric)
Supply	• Storing of fibre or fabrics in a designated space • Transportation of fibre or fabrics

1.1.2 Properties of natural fibres

The major constituents of natural fibres are cellulose, hemicellulose, and lignin. The elementary unit of a cellulose macromolecule is anhydro-D-glucose, which contains three alcohol hydroxyls (–OH). These hydroxyls form hydrogen bonds inside the macromolecule itself (intramolecular) and between other cellulose macromolecules (intermolecular) as well as with hydroxyl groups from the air. Therefore, PALF and jute, like all vegetable fibres are hydrophilic in nature and their moisture content can reach about 5-17% (Bledzki et al., 1996; Dittenber & Gangarao, 2012; Lotfi et al., 2021). The moisture content of natural fibres is dependent on the percentage of non – crystalline parts and the void content inside the fibre (Faruk et al., 2012).The structure and properties of these natural fibres depend upon several factors such as age, source, microfibrils, straightness, diameter, and chemical constituents

(Faruk et al., 2012). Natural fibres can differ from each other in cellulose content and spiral angle.

A natural fibre itself can be described as a multi – walled composite in which the unidirectional microfibrils made of cellulose act as the reinforcing element in a matrix of hemicellulose and lignin (Y. Li et al., 2018). The lumen is a hollow channel in the middle of the fibre axis, over which the primary and secondary cell walls wrap itself in the shape of the lumen. The secondary cell walls are made up of microfibrils, which consists of repeated crystalline and amorphous regions. The physical and chemical properties of natural fibres are very important in predicting the resultant property of the composite that it is reinforcing. Due to their small cross-sections, natural fibres are not suitable for direct use in engineering applications. Therefore, they are embedded in matrix materials to create fibre composites, with the matrix acting as a binder to hold the fibres together and transfer loads to the fibres. To effectively develop and promote the use of natural fibres and their composites, it is essential to have a thorough understanding of their physio-mechanical properties. Several reports have been published on the structures and properties of these natural fibres but a great deal of additional research is still needed in this area. Table 2 and Table 3 shows the physical and chemical properties of natural fibres based on the average of scientific papers, reviews and case studies.

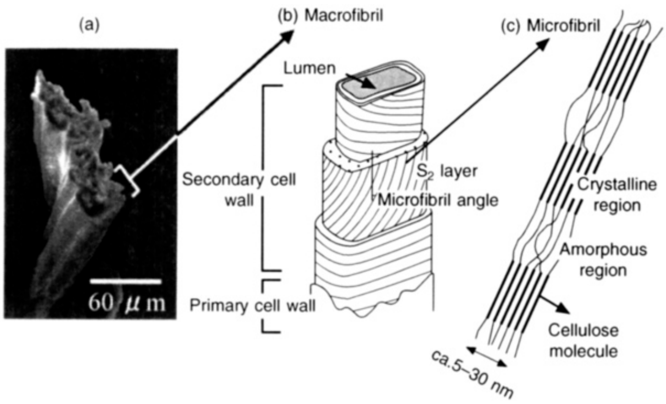


Figure 1: (a) SEM of a natural fibre (b) Schematic of a macrofibril (c) Schematic of a microfibril (Adapted from Green composites: Polymer composites and the environment, Chapter: 4) (Nishino, 2004)

Fibre name	**Fibre length (cm)**	**Density (g/cm^3)**	**Tensile strength (MPa)**	**Specific tensile strength (MPa/g cm^{-3})**	**Elongation at break (%)**	**Young's modulus (GPa)**	**Specific young's modulus (GPa/g cm^{-3})**	**Moisture absorption**	**Degree of crystallinity (X-ray)**
Abaca	—	1.5	400	—	3-10	12	—	15	—
Alfa	35	1.4	188 – 308	134 – 220	1.5 – 2.4	18 – 25	134 – 220	—	—
Bamboo	0.15 – 0.4	0.6 - 1.1	800 - 1200	—	10-20	15 - 20	25	8.9	—
Banana	30 – 90	1.35	500	—	1.5 – 9	12	9	—	—
Bagasse	1 - 30	1.25	290	—	1.1	17	18	8.8	—
Caraua	3.5	1.4	500–1150	—	3.7–4.3	11.8	39	—	—
Coir	0.8 - 33.7	1.2 – 1.32	95 – 230	110 – 180	15 – 37	4 – 6	3.3 – 5	10	25 - 33
Cotton	1 – 6	1.5 – 1.6	287 – 597	190 – 530	3 – 10	5.5 – 12.6	3.7 – 8.4	8 – 25	—
Flax	.5 - 90	1.4 – 1.5	345-1500	230 – 1220	1.2 – 3.2	27.6 - 80	18 – 53	7	44
Harakeke	.4 - .5	1.3	440–990	338 – 761	4.2 – 5.8	14 – 33	11 – 25	—	—
Hemp	.5 – 5.5	1.5	550 – 900	370 – 740	1.6	70	39 – 47	8	44
Isora	—	1.2 – 1.3	500 – 600	—	5 – 6	—	—	—	—
Jute	.2 – 12	1.1 - 1.34	393 - 800	300 – 610	1.5 – 1.8	10 – 78	7.1 – 39	12	52- 60
Kenaf	—	1.5	223 – 930	—	1.5 – 2.7	15 - 53	24	—	—
Nettle	—	—	650	—	1.7	38	—		
Oil Palm	—	0.7–1.55	248	—	25	3.2	2	—	—
Pineapple	90 – 150	0.8–1.6	180 – 1627	—	1.6 - 14.5	1.44 – 82.5	35	13	—
Piassava	—	1.4	134 – 143	—	7.8 – 21.9	1.07 – 4.59	2	—	—
Ramie	6 – 25	1.5	220 – 938	270 – 620	2 – 3.8	44 – 128	29 – 85	12-17	—
Sisal	90	1.33 – 1.5	507 – 855	362 – 610	2 – 2.5	9.4 – 28	6.7 – 20	11	—
Wool	3.8 – 15.2	1.3	400 – 600	38 – 242	10 – 20	15 – 20	1.8 – 3.8	13 - 18	—
Silk	Continuous	1.3	100 – 1500	100 – 1500	15 – 60	5 – 25	4 – 20	—	—
Ixtle (or Pita)	—	1.02 - 1.023	13.93 - 22.75	—	—	20.86 - 32.16	—	—	—
Henequen	—	1.11 - 1.13	430 – 570	—	3.7 – 5.9	8.8 - 17.04	11	—	—

Table 2: Physical properties of natural fibres (Asim et al., 2015; Dittenber & Gangarao, 2012; Faruk et al., 2012; “Handbook of Natural Fibres,” 2020; M. Li et al., 2020; Mittal & Chaudhary, 2018; Pickering et al., 2016; Prasad et al., 2020; Torres-Arellano et al., 2020; Varghese et al., 2020; Zin et al., 2018)

Fibre name	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Pectin (%)	Ash (%)	Moisture content (wt %)	Waxes	Microfibrillar Angle °
Abaca	56–63	7–9	15–17	1	0.5	5–10	3	—
Alfa	45.4	14.9	38.5	—	—	—	2	
Bamboo	26 – 43	25.3	16.8	—	—	—	—	—
Banana	63 – 67.6	5	10 – 19	—	—	8.7 – 12	—	—
Bagasse	55.2	25.3	16.8	—	—	—	—	—
Cabuya	80	17	—	—	—	—	—	—
Caraua	73.6	7.5	9.9	—	—	—	—	—
Coir	36 - 43	40 - 45	0.15 - 24	3.3 - 4	—	8	—	30 – 49
Cotton	83 - 99	<2	5.7	0 – 1	—	7.85 – 8.5	0.6	—
Flax	71	2.2	18.6–20.6	2.3	—	8–12	1.7	5 –10
Hemp	57–77	3.7 – 13	14–22.4	0.9	0.8	6.2–12	0.8	2 – 6.2
Henequen	77.6	13.1	4 – 28	—	—	—	0.5	—
Ixtle (or Pita)	46 – 48	—	17 – 20	11 – 12	—	—	—	—
Isora	74	23	—	—	—	—	1.09	—
Jute	61–71.5	12–26	13.6–21	0.2	0.5–2	12.5–13.7	0.5	8
Kenaf	31–57	15–19	21.5–23	3 – 5	2–5	—	—	—
Nettle	86	—	10	—	—	11 – 17	4	—
Oil Palm	65	12.7	—	—	—	—	—	42 – 46
Pineapple	81	12.7	—	—	—	14	—	14
Piassava	28.6	45	25.8	—	—	—	—	—
Ramie	68.6–76.2	0.6–0.7	13–16.7	1.9	—	7.5–17	0.3	7.5
Rice husk	35 – 45	20	19 - 25	—	—	—	14 –17	—
Rice straw	41 – 57	8 – 19	33	—	—	—	8 – 38	—
Seed	43–47	21–23	24–26	—	5	—	—	—
Sisal	47–78	7–11	10 –14.2	10	0.6–1	10–22	2	10–22
Wheat straw	38 – 45	12 – 20	15 – 31	—	—	—	—	—

Table 3: Chemical composition of natural fibres (Asim et al., 2015; Dittenber & Gangarao, 2012; Faruk et al., 2012; “Handbook of Natural Fibres,” 2020; M. Li et al., 2020; Pickering et al., 2016; Prasad et al., 2020; Torres-Arellano et al., 2020; Varghese et al., 2020; Zin et al., 2018)

1.2 Matrix

The type of resin matrix used affects the compressive strength, interfacial shear strength (IFSS), impact strength, and durability of the natural fibre – reinforced composites (NFRCs). The durability of the composite is influenced by several factors including the type of resin, existing stress during resin processing, microcracking, thermal and fatigue cycling. The intrinsic qualities of the resin also play a role in the extent of interaction between the fibre and resin. The properties of the resin are affected by curing temperature and exposure. Fully cured composites show improved mechanical properties and resistance to solvents and hydrophilic nature, while partially cured composites have poor mechanical properties (Teles et al., 2015).

Matrix can be of mainly two types: thermoset and thermoplastic. The shape, appearance, environmental tolerance and overall durability of a fabricated composite is heavily influence by the matrix, into which the fibres are impregnated to act as the main load bearing component (Faruk et al., 2012). It also protects the reinforcement materials from mechanical damage and environmental damage (Fowler et al., 2006). Some widely used matrices are shown in Table 4. In order to make a highly functional composite, the matrix/fibre interfacial and resin bulk properties are critical factors that needs to be controlled (N.L. Hancox, 1981).

Table 4: Commonly used matrices to make composites *(Alvarez et al., 2004; Arib et al., 2006; Barkoula et al., 2010; Berzin et al., 2014; Fung et al., 2003; Hauenstein et al., 2016; Ilyas et al., 2021; L. Liu et al., 2009; W. Liu et al., 2005; Malkapuram et al., 2009; Mansor et al., 2018; A. K. Mohanty et al., 2000; Moustafa et al., 2020; Panthapulakkal & Sain, 2007; Ray, Sarkar, & Bose, 2002; Ray, Sarkar, Das, et al., 2002; B. K. Sarkar & Ray, 2004; Sathish et al., 2021; Shubhra et al., 2013; Ye et al., 2023; Ying-Chen et al., 2010; Zhao et al., 2020)*

	Synthetic	**Biobased**
Thermoplastic	Polypropylene	
	Polyethene	Starch
	Polystyrene	Polylactic acid (PLA)
	Polyvinyl chloride	Polyhydroxyalkanoate (PHA)
		Polyhydroxybutyrate (PHB)
		Polycaprolactone
Thermoset	Polyester	Polybutylene succinate (PBS)
	Epoxy	Polybenzoxazine
	Phenol formaldehyde	Polycarbonate
	Vinyl ester	Soy – based resin
	Phenolic resin	

1.3 Natural fibre reinforced polymer – based structural biocomposites (NFRCs)

In recent years, natural plant fibre-reinforced composites have become increasingly popular as structural composites due to their green credentials and low-density characteristics (Dayo et al., 2017). A structural composite is a composite that is arranged in laminar form (stacked on top of each other). They usually have high tensile, compressive and torsional strength, low density and high stiffness. Laminar composite and sandwich panels are the most common type of structural composites. Another reason it is called structural composites is because, the composite’s mechanical properties, other than their constituent reinforcement materials, also depends on their geometry (William D. Callister & David G. Rethwisch, 2018).

Laminas of fibres can be stacked in many ways, the name of the orientation of the lamina along with the angle they make with reference axis (one of the edges being taken as 0 ˚) are shown in Table 5.

Table 5: Orientation of laminaa and the angle they make with the reference axis *(William D. Callister & David G. Rethwisch, 2018)*

Orientation of lamina	Angle with reference line
Unidirectional	0°
Cross – ply	90°
Angle – ply	$\pm 45^\circ$
Multi – directional	90°, $\pm 45^\circ$

The stacked laminas are then impregnated with matrix resin and then left to cure. There are several types of fabrication processes that can be done to make laminated composites. They are shown in Table 6.

Table 6: Methods of manufacturing high performance structural composites (Akampumuza et al., 2017; Lotfi et al., 2021; N.L. Hancox, 1981)

Name of process	**Definition**	**Mostly applied for**
Compression moulding	Individual prepreg plies are laid on top of each other, and then compressed under heat and left to cure.	Making large flat composites; infrastructure; car interior components
Vacuum bag/ autoclave moulding	Involves prepregs which are put under pressure through a vacuum and put into an autoclave for heating and curing purposes.	Fabricating fairly complex shapes such as structural aerospace components.
Mandrel wrapping	Prepreg material is wrapped onto a mandrel and then pressure and heat is applied to consolidate and cure the component.	High volume tubular hybrids; tapered tubes; parallel tubes.
Pultrusion	Reinforcing fibres are impregnated with resin in a continuous manner using a resin system and then pulled using a curing die that incorporates the desired shape of the composite.	Hybrids in the shape of rods, profile sections and tubular sections in continuous length; round bars and rod stocks; complex profile shapes and hollow sections
Filament winding	The reinforcing fibres are wounded over a mandrel and then put into a path of neat resin. Curing is done with or without the mandrel by the application of heat.	Hollow components (cylindrical or spherical) such as pressure and storage tanks, pipes and rocket cases.
Adhesive bonding	Usually a follow – up for another moulding process, mostly autoclave moulding. The skins are then put on the side of the onto the composite using an adhesive film. Then, the whole assembly is vacuumed bag again and put into an autoclave for curing.	Certain aerospace components that have reinforcements on the side of the core.
Vacuum resin infusion	The reinforcements are stacked and then vacuum bagged with one line connected to a sealed container and another line connected to a resin system. During vacuuming, the resin from the resin system is pulled into the mould and impregnates the reinforcements. Excess resin is pulled out from the mould to the sealed container on the other side.	Complex profiles and shapes, i.e., automotive components such as car dashboard, doors and handles; skiing equipment; boats etc.
Hand lay – up	Hand placement of reinforcements in the mould and then applying resin. The wet composite is then rolled, brushed and squeezed and then left to cure.	Low – cost tooling
Automated fibre placement	The process of hand – lay up is automated and the lay – ups are done using a robotic hand	Customized parts and bulk manufacturing

Compared to a matrix, fibres are stiffer and stronger. The fibre may be continuous, unidirectional, woven and/or laminated. It might be because there are imperfections in the bulk material, whereas there is very little chance of internal problems in fibre. Due to their low density and superior mechanical qualities, fibres with an aspect ratio (l/d) > 1000 are preferred among all natural fibres for use as reinforcement in polymer systems (Jahan et al., 2012). Both natural and synthetic fibres can be utilized as reinforcement in fibre reinforced polymer composites. The general advantages and disadvantages of using natural fibre as reinforcements are shown in Table 7.

Table 7: Characteristics of natural fibre reinforced polymer composites (Cheung et al., 2009; Dittenber & Gangarao, 2012; Faruk et al., 2012; Y. Li et al., 2018; Pickering et al., 2016; Yang et al., 2019)

Advantages	**Disadvantages**
• Low density • High specific strength and stiffness • Non – corrosive • Renewable and sustainable source of reinforcement • High thermal and acoustic insulation • Growing these fibres extracts CO_2 from atmosphere • Lower cost than synthetic fibres • Lower risk of manufacturing • Low emissions of toxic fumes when exposed to heat and incineration	• Low strength and stiffness • Variability • Needs extensive pretreatments that is both costly and hazardous • High moisture absorption • Lower fabrication temperature, hence matrix options are limited

1.3.1 Hybridization

When reinforcing fibres are mixed intimately with a matrix, which must be able to infiltrate the fibres and then be solidified, the resulting material is known as a hybrid. The process of making this material is known as hybridization (N.L. Hancox, 1981). Hybrid composites are used to describe in which two or more types of fibres are used to reinforce a common matrix. Hybridization has become a very popular technique in polymer technology, which allows designing products with higher mechanical properties, while minimizing the effects of moisture absorption and poor fibre/matrix bonding by reinforcement with two different natural fibres or with natural and synthetic fibres (Asim et al., 2017; Siakeng et al., 2018; Zin et al., 2018). Hybridization is also done due to the following reasons (M. Li et al., 2020):

- To balance the areas of weakness of one fibre by incorporating another fibre that can compensate for those areas of weakness by having better properties.
- Replacing part of a high – costing reinforcement with a lower cost fibre without having too much effect on the performance.
- Achieving sustainability by incorporating natural – based reinforcements.
- Providing flexibility in fabricating composites with mechanical and chemical properties that are customized for one application.

The positive or negative impact of hybridization can be determined by finding out the Hybrid effect using Equation (1) (Yang et al., 2019)

$$P_{Hybrid} = P_1V_1 + P_2V_2 \qquad (1)$$

The different classes of hybrids are shown in Table 8.

Table 8: Types of hybrids (N.L. Hancox, 1981)

Type	**Name**	**Definition**
A	Dispersed fibre	Mixture of two or more continuous fibre aligned but randomly dispersed throughout a matrix
B	Dispersed fibre ply	Alternating layer of two or more fibre lamina (ply)
C	Fibre skin and core	Outer skins made of one or two types of fibre laminates over a core of another type or same type of fibre laminate
D	Fibre skin/non – fibre core	Outer skins are made or one or two types of fibre laminates over a honeycomb, wood or metal core.

1.4 Motivation

With concerns about the environment increasing, reducing carbon footprint has become crucial. There has been an increasing use of natural materials, biodegradable and recyclable polymers and their composites in various engineering applications, with the increasing usage of non-naturally decomposed solid wastes.

Greater environmental awareness, issues with waste disposal, and the degradation of petrochemical resources are the main drivers behind the creation of natural composites. In recent years, the expense of biopolymers has become really high for the business to continue. The price variations for each natural fibre are brought on by crop variability as well as the challenges of handling, storing, and processing fibres (Dittenber & Gangarao, 2012). Biocomposites must be improved to compete with glass fibre composites since surface alterations are necessary yet expensive (Dittenber & Gangarao, 2012; A. K. Mohanty et al., 2001).

The main motivation of this study is to fabricate a laminated hybrid biocomposite with natural fibres that are abundant in Bangladesh. Our aim is to stimulate the area of research of utilizing local cheap raw materials for different applications.

1.5 Objective of the study

Focusing on the mechanical performance of fabricating an epoxy – based NFRC, the objective of this study is:

- To fabricate a fibre reinforced composite of high quality with a combination of wet lay – up and vacuum bagging process.
- Do mechanical characterization and water absorption test to find industrial applications in the fields of automotive, marine and aerospace sector.

1.6 Significance

This thesis will aim to update an existing library of a research topic which is: fibre reinforced composites. It will also provide insights on the influence of blending several fibres to make woven mats and the influence of weaving patterns in the mechanical performance of the final fabricated composite. Furthermore, the thesis will aim to find replacement of conventional synthetic structural components in the automotive, aerospace and marine sectors with the aim to increase sustainability.

1.7 Thesis outline

Chapter 1 contains the current scenario of structural materials and a general overview of the key concepts needed to understand the thesis. Chapter 2 will provide a review of the motivation of using cotton – blended woven PALF and Jute as reinforcement, as well as why epoxy was used as a matrix. Furthermore, several factors that influences a structural composite is discussed, along with how it influenced our decisions during the project experiment. Chapter 3 will show the background of the research methodology along with the experimental procedure, starting from the sourcing of material, fabrication of the structural composites and the tests that has been selected for characterization. Chapter 4 will report the creation and characterization of the natural fibre reinforced hybrid. Finally, in Chapter 5, the key findings of our undergraduate research and future directions are summarized.

Chapter 2

Literature Review

The following literature review is a summary of previous experimental, analytical and numerical studies of PALF reinforced epoxy – based composites, jute reinforced thermoset – based composites, and natural fibre reinforced epoxy – based hybrid composites. It also discusses the various factors that affects the mechanical and chemical properties, surface morphology of hybrid composites.

2.1 Natural fibre epoxy – based hybrids

2.1.1 Epoxy

The diverse and adaptable nature of epoxy has made it a suitable material for various industrial uses, including laminated circuit boards, encapsulating electronic components, coating surfaces, potting, reinforcing fibres, and as adhesives (Saba et al., 2016). Epoxy is a type of thermosetting material used as a matrix or resin. It contains at least one epoxide group in its molecule, also known as oxirane or ethoxyline, which is considered the building block of the epoxy polymer (Pascault & Williams, 2013; Yu et al., 2009). Most commercially available epoxy resins are oligomers of diglycidyl ether of bisphenol A (DGEBA), which, when combined with a hardener, undergoes a curing process and becomes a thermosetting polymer (Roşu et al., 2002; Zhang et al., 2012). There are two types of epoxy families: non-glycidyl and glycidyl epoxies, with glycidyl epoxy resins further categorized into glycidylamine, glycidyl – ester, or glycidyl – ether. Non-glycidyl epoxies are also known as cyloaliphatic or aliphatic resins (Zhang et al., 2012). The most commonly used glycidyl epoxy resins are synthesized by reacting bisphenol A (BPA) with epichlorohydrin molecules. Another type of epoxy commonly used in certain industries is novolac – based epoxy resin. Epoxy resins are combined with various co-reactants, curing agents, or hardeners, such as anhydrides, amines, and amides, with amine-based hardeners being the most common. Compared to polyester or vinyl ester resins, epoxy resins require a higher amount of curing agent in the resin-to-hardener ratio (1:1 or 2:1) (Alamri & Low, 2012), and their cured systems have low fracture toughness, inherent low impact resistance, unaltered shape after curing/polymerization, and reduced resistance to crack initiation and propagation, (Comas-Cardona et al., 2005; Mirmohseni & Zavareh, 2010; Njuguna et al., 2007).

2.1.2 Hybrid thermoset – based natural fibre composites

The combinations of various natural lignocellulosic composite have always been a topic of research as it features a number of improvements. It offers a wide range of outcomes and qualities that are exceedingly challenging to obtain by a single sort of reinforcement. Thermoset matrix is typically utilized for the fibre because it promotes good interaction between the matrix and the fibres, which improves mechanical performance (Fung et al., 2003; Idicula, Malhotra, et al., 2005). Thus, a hybrid composite is made from a combination of two different fibre types that have been reinforced in a matrix. Fibres are blended based on their individual strengths to create composite materials that are more effective.

Numerous studies are being conducted to replace synthetic fibre fully or partially with natural fibres. Hybridization of natural fibres like sisal, jute, pineapple, hemp, in glass fibre offers excellent reinforcing (Ahmed & Vijayarangan, 2006; Panthapulakkal & Sain, 2007). Pothan *et al* investigated the effect hybridizing woven banana fibre and GF in polyester matrix (Pothan et al., 2005). Hybridization of GF with natural fibres such as oil palm fibre was also investigated (Sreekala et al., 2005). NFRC with a hybrid system of short carbon and kenaf fibres was also fabricated (Anuar et al., 2008).

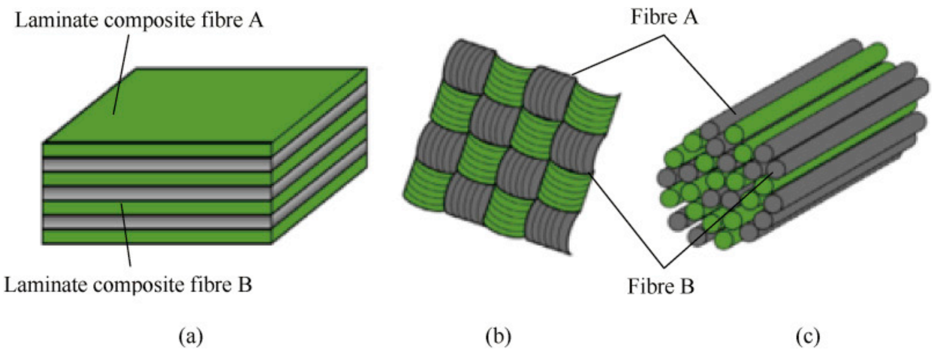


Figure 2: Fibre reinforce thermoset polymer hybrid composites (Supian et al., 2018)

Several works in hybridizing natural fibres in matrices have been conducted. Mixed, random orientation of banana/sisal hybrid fibre reinforcement with polyester composite was examined by Idicula *et al*. (Idicula, Neelakantan, et al., 2005). The composite made of banana and sisal fibres in a 3:1 ratio was seen to the highest computed transformational stress between the fibres and matrix and the lowest impact strength. Pineapple leaves are frequently used in conjunction with disposable chopsticks (Chiang et al., 2012; Hung et al., 2013). PLA and PBS were combined with recycled disposable chopstick fibres and pineapple leaf fibre (2.3-3.9 mm).

Natural fibre composites are becoming more important due to environmental laws, sustainability theories, and ecological, social, and economic awareness (Faruk et al., 2012). In comparison to conventional materials, natural fibres have higher particular qualities including impact resistance, stiffness, flexibility, and modulus. Other advantageous characteristics are low cost, low density, less vibration damping, and improved recovery (Sgriccia et al., 2008). Other desirable qualities include widespread availability, the lack of associated health risks, and comparatively lower abrasion resistance (Lei et al., 2007). This thesis will explore the usage of natural fibre laminates as reinforcements in epoxy matrix to achieve the mechanical qualities, the ideal ratio and content of the hybrid fabric.

2.2 Natural fibres and their significance as reinforcement in hybrids

2.2.1 Jute reinforcement

Jute (*Corchorus Olitorius*, genus: *Corchous*) are usually grown in tropical nations, and it is one of the strongest and least expensive fibres in the world. Countries such as Bangladesh, India and China provide the best condition for the growth of jute (Faruk et al., 2012). Jute, which includes roughly 30–40 Capsularis species, is a member of the Tiliaceae family. The two jute species, Corchorus Capsularis (white jute) and Corchorus Olitorius, are the most often cultivated (tossa jute). The olitorious occurs in yellow, grey, and brown types, whereas the capsularis jute is naturally white in colour (Gopinath et al., 2014). Although jute is naturally associated with the Mediterranean, the best types for its cultivation are presently provided by Bangladesh, India, China, Nepal, Thailand, Indonesia, and Brazil (Faruk et al., 2012; Thiruchitrambalam et al., 2010). Jute fibre is produced globally in quantities ranging from $2300 * 10^3$ to $2850 * 10^3$ tonnes (Faruk et al., 2012; Mwaikambo & Ansell, 2002). In Bangladesh, regions like Faridpur can make up to 2 -3 kg of jute per day, selling at BDT 1400 – 1500 ($13.20 - $14.40) per meter. Jute contains a high amount of stiff natural cellulose compared to other lignocellulosic fibres (Corrales et al., 2007). Jute fibre has better mechanical properties than many natural or synthetic fibres as discussed by (Sayem & Haider, 2020).

The effect of incorporating jute as a reinforcement in several bio – based and synthetic matrix for better mechanical and dynamic properties have always been an active field of research. The composite strength produced when jute fibre is added to the matrix material would be stronger than the matrix material but weaker than the jute fibre (Maity et al., 2012; Sayem et al., 2020; Shahinur et al., 2016). Furthermore, in a study conducted by Khan *et al*, they showed that jute plants absorb 15 tons of carbon dioxide (CO_2) from the environment and release 11 tons of oxygen during their 120-day lives (O_2) (J. A. Khan & Khan, 2015), showing a very high potential for reducing the carbon footprint of structural components if they are incorporated with jute. Furthermore, Alves *et al*. studied that jute fibre composites provide improved environmental performance, according to a life-cycle evaluation research comparing them to glass fibre composites (Alves et al., 2010).

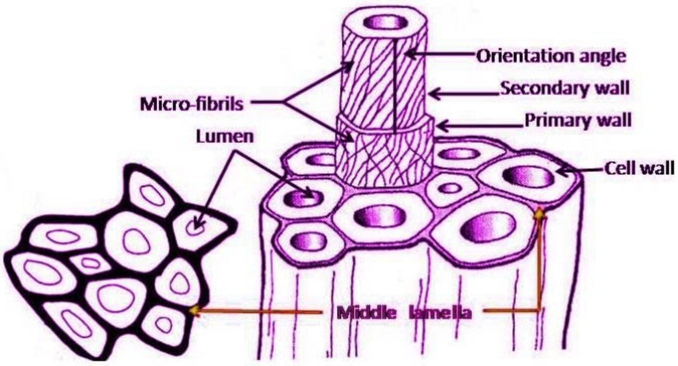


Figure 3: Cross sectional view of jute fibre (Mia et al., 2017)

Jute reinforced composites can be prepared using both thermoset and thermoplastic matrix. The fabrication of such composites can be done by simple methods such as hand lay-up (Seki, 2009). Incorporating jute fibre in thermoplastic resins such as polyhydroxybutyrate, and polyamide has shown to improve its tensile strength, bending strength and impact strength by 50%, 30% and 90% respectively. Using jute in thermoset matrices such as epoxy also showed higher tensile strength and flexural strength compared to pure epoxy matrix (Biswas et al., 2015) and has shown great potential in replacing conventional structures. Raghavendra *et al,* produced a jute/epoxy composite by hand lay-up method, studied their moisture absorption characteristics, and evaluated their tensile and flexural strength when subjected to saline, mineral, and sub-zero temperature water (Raghavendra et al., 2017). In a study conducted by Rahman *et al,* the impact of jute volume fraction and fibre orientation on the mechanical and thermal properties of jute – polyester composites were investigated. It was shown that, with a maximum volume percentage of 30%, the composite was good enough to replace wood-based structures and had the potential to be utilized as a thermal insulation material thanks to better mechanical qualities that were acquired with an appropriate combination of fibre orientation and fibre volume percentage (F. Rahman et al., 2022). Gowda *et al*. evaluated the hand-laid, untreated jute fabric – polyester composite and assessed its hardness and tensile, compressive, flexural, impact, and in-plane shear strengths (Munikenche Gowda et al., 1999). They also came to the conclusion that jute-polyester composites are stronger than wood composites. Hence, it can be concluded that jute reinforced thermoset composites has the potential to replace conventional wood structures such as door frames, cupboards, bookshelves etc. Other applications of woven jute reinforced thermoset composites includes: interior of cars & airplanes, as well as roofing and fences (Dobah et al., 2016; Hu et al., 2012).

Surface modification is a critical factor in putting jute into the market as a potential reinforcement. In a study conducted by Mohanty *et al* (A. K. Mohanty et al., 2000) they showed that altering the surface of jute/Biopol and jute/PA composites affects their mechanical properties and ability to biodegrade. They discovered that modifying the surface led to significant improvements in the composites' tensile, bending, and impact strength. These enhancements exceeded 50%, 30%, and 90%, respectively, and were similar to the strength values of pure Biopol sheets. The impact of various modifications on jute fibre reinforced PP composites was investigated through multiple studies. These studies examined the impact of modifying the matrix (Doan et al., 2006), exposing the composites to gamma radiation (Haydaruzzaman et al., 2009), improving interfacial adhesion and how it affects creep and dynamic mechanical behaviour (Acha et al., 2007), using a silane coupling agent (Hong et al., 2008; Wang et al., 2010), and incorporating natural rubber (Zaman et al., 2010). Sever *et al*. utilized various techniques such as alkali treatment, micro – emulsion silicon, fluorocarbon treatment, and compression moulding technology to modify jute fabrics before producing a composite of jute and unsaturated polyester (Sever et al., 2012). These treatments effectively enhanced the tensile, flexural, and interlaminar shear strengths of the composites, with fluorocarbon treatment yielding the most significant improvement.

Long jute fibre (LFT) reinforced thermoplastic composites have experienced significant growth in recent years, and are now widely accepted as structural materials with a growing presence in the automotive industry. These composites offer excellent mechanical properties and stiffness-to-weight ratio. LFT polypropylene composites are currently the most popular type, providing performance that falls between that of short fibre-reinforced polymer and glass-mat thermoplastic composites (Uawongsuwan et al., 2015). A variety of properties of jute/plastic composites were analysed and investigated in other studies. These properties include thermal stability, crystallinity, modification, trans-esterification, weathering, durability, fibre orientation, and their impact on frictional and wear behaviour. Additionally, studies were conducted on the eco-design of automotive components and the use of alkylation in the composites (Alves et al., 2010; Dwivedi & Chand, 2009; MIR et al., 2010; Samal et al., 2001; Sarikanat, 2010; S. Sarkar & Adhikari, 2001; B. Singh et al., 2000).

With regards to the above – mentioned information, mercerized long jute fabric is chosen as the first reinforcement of our natural fibre epoxy – based hybrid composite.

2.2.2 PALF reinforcement

Pineapple (*Ananas comosus*) is a tropical plant native to Brazil. Pineapple is a non- climacteric and third most widely cultivated after banana and citrus. Its yield per hectare varies from 40-60 tonnes depending upon the variety (Basu & Roy, 2007; Todkar & Patil, 2019). Pineapple fibres are a part of the group of natural fibres, classified as leaf fibres. The raw materials of pineapple fibres (PALF) are pineapple leaves, which are heavily unutilized (Debnath, 2016) Per shoot, there are approximately 40-50 leaves or 2.3 Kg of pineapple leaves as agro waste which are not suitable for consumption, yield a fibrous material made up of ligno-cellulose. This material is composed of 67.12–82% cellulose, 9.45–18.80% hemicellulose, and 4.40–15.40% lignin (H. Rahman et al., 2022). Numerous modern studies (Azlin et al., 2022; Mahjoub et al., 2014; Nimanpure et al., 2019; Pickering et al., 2016; H. Rahman et al., 2022; Sanjay et al., 2016; Sayem et al., 2020; Shubhra et al., 2013; Summerscales et al., 2010; Todkar & Patil, 2019) have reported the use of these fibres as reinforcements for composites in both thermoplastic and thermoset plastic matrices. This application is utilized for the development of both value-added products and structural components. PALF has been also used as reinforcement to make composites such as biodegradable plastics (Bajpai et al., 2014; W. Liu et al., 2005; Varghese et al., 2020) and natural rubber (Lopattananon et al., 2006). The reason for this is because pineapple fibres have good mechanical properties due to their high crystalline content compared to other vegetable fibres (N. Reddy & Yang, 2005; Satyanarayana et al., 2007; Sena Neto et al., 2013; Tomczak et al., 2007).

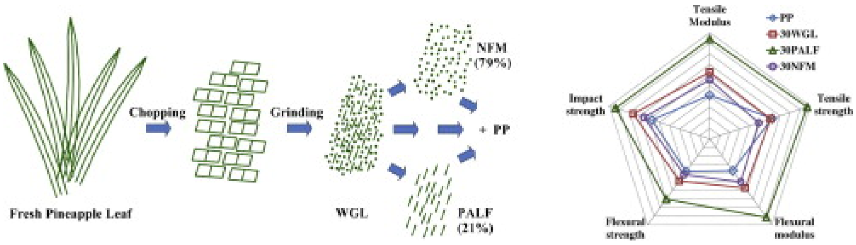


Figure 4: Pineapple fibre production procedure *(Kengkhetkit & Amornsakchai, 2014)*

Approximately 13 million tonnes of agro waste are produced from pineapple agriculture every year (*Https://Www.Ananas-Anam.Com*, 2023). So, on a large scale, there is a huge volume of post-harvest waste to be handled (Chollakup et al., 2011). Among natural fibres extracted from plant leaves, PALF possesses a very high percentage of cellulose content and low microfibrillar angle (A_m), which is the helix angle between the cellulose fibrils and the axis of the secondary cell wall's cell axis (Rowell & Rowell, 1996). The low microfibrillar angle, along with the high cellulose content gives PALF its characteristic high tensile properties, (Asim et al., 2015; Sena Neto et al., 2015). Among various fibres, PALF also finds apex application in the textile industry due to many reasons such as abundantly available, cheaper, good thermal and acoustic insulation, excellent tensile strength, and high toughness (*Https://Www.Ananas-Anam.Com*, 2023).

The primary advantages of using these fibres as reinforcements in polymer composites are listed as follows: (1) low density, (2) low cost, (3) nonabrasive nature, (4) high filling level possible, (5) low energy consumption, (6) high specific properties, (7) biodegradability, and (8) generation of rural/agricultural-based economy (Mishra et al., 2004). It also has high onset oxidation temperatures 240 – 272◦C, making it a good choice for most commercial polymers (Sena Neto et al., 2015). There are many factors that influences PALF composites' mechanical and thermal performance, such as: fibre length, fibre content (Chollakup et al., 2011; Mishra et al., 2001). It was shown by Chollakup *et al* that, for thermoplastics PP and LDPE – based PALF composites, the PALF reinforcement improved the strength and stiffness of the composite without any surface modifications. When comparing the tensile and flexural properties by varying the fibre content in PALF reinforced composites, it was shown that a 30% wt fibre content showed optimum mechanical performance (Mishra et al., 2001).

Hence, PALF is chosen as the second reinforcement used in the natural fibre reinforced epoxy composite hybrid.

2.3 Factors affecting natural fibre hybrids

The properties of natural fibres and natural fibre composites are subject to variation, which can be dependent on several factors such as the type of fibre, cultivation environment, timing of harvest, method of extraction and processing, and source from which it is obtained (Wan Nadirah et al., 2012). These variables play a crucial role in determining the mechanical properties of natural fibre reinforced composites (NFRCs), and therefore, should be carefully considered to determine their impact on the final composite properties. Other factors such as surface treatment, fibre blending and weaving patterns also influence the properties of the biocomposite.

2.3.1 Variety of the natural fibre

In order to be considered as the primary fibre for reinforcement, the fibre's modulus must meet a certain threshold when combined with the polymer matrix. Researchers characterized PALF by conducting experiments using three Malaysian varieties- Josapine, Sarawak, and Moris Gajah in order to identify the best option (Munawar et al., 2015). The tensile strength of Sarawak, Moris Gajah, and Josapine was observed to increase in that order, with Josapine exhibiting the highest elasticity modulus (18.94 Gpa), followed by Sarawak and Moris Gajah. To enhance the mechanical properties of PALFs, the cross-sectional fibre diameter should decrease, while the crystallinity index should increase. However, these two factors are inversely proportional, so it is essential to obtain an optimal fibre diameter to achieve a higher crystallinity index and enhance mechanical properties (Sena Neto et al., 2013).

2.3.2 Length of fibre

Compared to short fibres, long fibres can be easily directed in a particular direction. Short fibres, on the other hand, are randomly oriented, which causes composite samples to behave in a brittle manner (Ramli et al., 2017). Long fibres, due to their controlled orientation, behave like ductile materials. The fracture modulus of the short PALF sample was 25 times higher than that of the long PALF sample. The long PALF sample had higher tensile strength and flexural strength than the short PALF composite. Furthermore, it was observed that as the fibre content increased, a reduction in elongation at break was observed due to the decreasing fibre length (Chollakup et al., 2011). This highlights that composite samples become stiffer and lose their flexibility during elongation as short fibre loading increases (Jose et al., 2016; Teles et al., 2015).

2.3.3 Fibre orientation

Potluri et al. (Potluri et al., 2018) examined how fibre orientation affects mechanical properties, with longitudinal orientation producing higher properties. PALF was tested with PP and LDPE, varying fibre length and loading. Long fibres had effective load carrying with some breakages, while short PALF resulted in more pull – out and poor adhesion causing splitting and debonding. LDPE with long fibres was better, but short PALF with PP at 25% weight was superior due to multi-directional orientation (Chollakup et al., 2011; Lopattananon et al., 2006).

2.3.4 Voids and porosity content in the composite

The presence of voids in composite is caused by the difference in polarity between the fibre and resin, which occurs due to the untreated fibres' incompatibility with the resin, particularly in hybrid composites. During impregnation, air or other volatile substances become trapped in the composite, leading to fibre pull – out, debonding, and premature failure (Asim et al., 2018). These voids act as stress concentrators, resulting in a decrease in fatigue strength (Alkbir et al., 2016; Graupner et al., 2016; Shih et al., 2014).

2.3.5 Surface treatment of natural fibres

The morphology, as well as density, cell wall thickness also has strong effect on the physical properties of the fibres. The poor adhesion between the fibre – matrix interphase of the hydrophilic biofibres and hydrophobic thermosetting polymers results in inadequate stress transfer between the fibre and matrix. The presence of non-cellulosic elements, such as pectin, hemicellulose, wax and lignin on fibre surface limits the interlocking of the reactive functional group of the fibre with the matrix. This impedes full utilization of the fibre strength, thus limiting the scope and scale of application and reducing fatigue life (Kalia et al., 2011) .Several surface treatments are currently present that can modify the fibre surface by removing the non-cellulosic elements. They are generally characterized into two parts: physical and chemical treatments. Chemical methods are the widely-used and the most convenient method of improving interface properties of organic fibre – based biocomposites (Gurunathan et al., 2015).

2.3.5.1 Acetylation

Acetylation treatment is a process by which the reactive hydroxyl group (-OH) in the cell wall takes part in esterification by reacting with an acetic anhydride, forming an acetylated lignocellulosic. The fibre wall swells and interlocking between the fibre and matrix is improved. This method introduces acetyl function group in the fibre and produces by-product acetic acid (Amiandamhen et al., 2020; Rowell, 2004) .

$$\begin{aligned} Cell\,Wall - OH + CH_3C(=O) - O - C(=O) - CH_3 \\ \rightarrow\; Cell\,Wall - O - C(=O) - CH_3 + CH_3C(=O) - OH \end{aligned}$$

Zaman et al. in 2021 (Zaman & Khan, 2021) has shown that banana empty fruit bunch fibres treated through acetylation demonstrated significant improvement in the mechanical properties, interfacial shear strength and water absorption properties than alkali – pretreated and untreated banana empty fruit bunch fibre – polypropylene (PP) matrix composites. Another study on acetylation treated plain woven banana fabric/PVA composite (Kivade et al., 2022) demonstrated improved interfacial behaviour, storage modulus, loss modulus, a damping factor, impact strength but reduced water absorption rate and biodegradability compared to untreated plain woven banana fibre. A higher tensile strength was demonstrated in Acetyl Treated kenaf – bast fibres in comparison to Untreated kenaf – bast fibres for all fibre volume fractions in (S. I. A. Razak et al., 2014).

2.3.5.2 Silane treatment

Silane treatment makes use of silane-coupling agents, to form silanols, (Amiandamhen et al., 2020) on the surface of the fibre in reaction with moisture.

$$Fiber - OH + R - Si(OH)_3 \rightarrow Fiber - O - Si(OH)_2 - R$$

This enhances the matrix – fibre adhesion and improves mechanical properties. In addition to improving the mechanical properties of the composites, silane treatment reduces water absorption significantly in natural fibre composites.(Fathi et al., 2019; Y. Liu et al., 2019). While exploring the usability on a brake pad, Vijay et al (Vijay et al., 2021) demonstrated noticeable improvement in the interfacial bonding, crystalline index, thermal stability, hardness and mechanical strength of silane treated *Leucas Aspera* fibres in thermosetting polymer matrix.

2.3.5.3 Benzoylation

Often as a follow-up to alkali treatment, benzoylation further improves the mechanical properties of lignocellulosic fibre – based composites through further decreasing the hydrophilicity of the natural fibre, promoting the interfacial adhesion and surface interlocking between the fibre and the hydrophobic matrix.

$$Fiber - O - Na + C_6H_5 - COCl \rightarrow Fiber - O - C_6H_5 + NaCl$$

Recent studies performed on hybrid composites of sugar-palm fibre showed that benzoylation treatment showed a positive increase in the mechanical properties, interfacial adhesion, initial and final degradation temperature, flammability resistance, thermal stability, dynamic mechanical, impact and post impact properties. (Izwan et al., 2021; Safri et al., 2019, 2020). Benzoylation has been reported to increase the tensile properties, generally attributed to aiding in the interlocking of surfaces in sisal fibre(Manikandan Nair,' et al., n.d.; Sreekumar et al., 2009), vetiver fibre (Vinayagamoorthy, 2019) and bamboo fibre (Kushwaha & Kumar, 2011) based biocomposites.

2.3.5.4 Graft co – polymerization

Graft co-polymerization is a widely used method that is advantageous in the context that the graft co-polymers do not impart any non-bio-degradable property on the fibre. Vinylic monomers are grafted onto the cellulose backbone of the organic fibre. On the surface a co-polymer is produced having the characteristics of both a grafted polymer and fibrous cellulose. In cellulosic biofibres graft copolymerization of methylacrylate, acrylonitrile, acrylic acid and methyl methacrylate widely have been studied in literature(Kaith et al., 2010; X. Li et al., 2007; Thakur et al., 2011). Composites prepared from fibres treated by grafted co-polymerization showed improved mechanical and thermal properties (Kaith et al., 2010; Thakur et al., 2011)

2.3.5.5 Alkali treatment (Mercerization)

Although the chemical treatments stated are widely used and have shown varying degrees of effectiveness, the most common and the most primitive chemical treatment is alkaline treatment (mercerization). This form of treatment removes the non-cellulosic substances such as hemicellulose, pectin, lignin and wax from the fibre surface, promoting surface roughening and thus improving interphase adhesion. This process is commonly performed though soaking the untreated bio – fibres in aqueous solution of alkali, commonly NaOH of varying concentrations.

$$Fiber - OH + NaOH \rightarrow Fiber - O - Na + H_2O$$

The effect of mercerization on different natural fibres based biocomposites are shown in Table 9 below.

Table 9: Effect of alkali treatment on different natural fibres used as reinforcement in composites.

Fibre Type	**Mercerizing Agent**	**Concentration**	**Temperature and Time**	**Effect on Mechanical Properties**	**Reference**
Sisal	NaOH	2M	Ambient Temperature, 2h	Improvement of fracture stress and Young's Modulus. Reduction of toughness and fracture strain	(Kim & Netravali, 2010)
Ramie	NaOH	15%	Ambient Temperature, 2hr	Improvement of Tensile Strength, fracture strain. Reduction of Young's Modulus	(Goda et al., 2006)
Bamboo	NaOH	10%,15%,20%,25%	Ambient Temperature, 1hr	Improvement of Ultimate Tensile Stress, Tensile Modulus with increasing alkali concentration, up to 20%, depreciation of mechanical properties for further increase of alkali	(Das & Chakraborty, 2009)
Jute	NaOH	5 %, 10% and 20%	Ambient Temperature, 30,60 and 90 minutes	Tensile and Bending Strengths increases with NaOH concentration, but decreases for concentration more than 10%- and 90-minutes soaking time. Highest values of tensile and bending stresses were found when the concentration was 20% and soaking time was 60 minutes. Impact strength reduced sharply with the increase of NaOH concentration.	(J. A. Khan et al., 2012)
Pineapple Leaf	NaOH	3% and 7%	30˚ and 60˚, 2hr	Drastic improvement of Tensile Strength and Reduction of Stiffness.	(Jaramillo-Quiceno et al., 2018)

The concentrations of NaOH which causes excess delignification and swelling results the fibre to become weaker, exhibiting inferior mechanical properties is dependent on the type of fibre and its lignin content. Table 10 illustrates the concentration of NaOH that causes excess delignification in organic fibres, resulting in loss of strength.

Table 10: Effect of excess alkali treatment that causes excess delignification of natural fibres

Fibre Type	NaOH concentration	Treatment Duration	Temperature	Reference
Pineapple	10%	1hr	30 C	(Mishra et al., 2003)
Bamboo	25%	1hr	Ambient Temperature	(Das & Chakraborty, 2009)
Jute	20%	90 minutes	Ambient Temperature	(J. A. Khan et al., 2012)

Overall, mercerization process remains an accessible, primitive and widely adopted method to improve the mechanical properties of the fibre reinforced composites by improving the interphase contact between fibre and matrix.

2.3.5.6 Others

Apart from the chemical treatments stated above, other chemical treatments namely isocyanate treatment(Karmarkar et al., 2007; Kokta et al., 1990; Qiu et al., n.d.), Maleic Anhydride grafted Poly Propylene (MAPP) coupling agents(Madhavi et al., 2021; S. Mohanty et al., 2004; Nayak & Mohanty, 2010), permanganate treatment(Imoisili & Jen, 2020; *Role of Potassium Permanganate and Urea on the Improvement of the Mechanical Properties*, n.d.) and peroxide treatment (N. I. A. Razak et al., 2014) on lignocellulosic fibres with their implications have been widely explored in the literature.

2.3.6 Blending of natural fibres

The blending of different textile fibres is a common practice used to obtain fabrics with desirable properties (Duckett et al., 1979). Natural fibres and their blends with classic fibres bear valuable properties, so at present there are various products made of these fibres. Blended yarns are better than classic yarns in terms of their absorption and discharge of moisture, non-irritating, antibacterial, and anti-allergic characteristics, as well as protection against the sun's harmful ultra-violet rays and other valuable properties. These blended yarns are useful for clothing, underwear, socks, hygienic textile products, and composites. In the cotton spinning process, blending aims to produce yarn with acceptable quality and reasonable cost (Svetnickienė & Čiukas, n.d.). In the cotton spinning process, blending has the objective of producing yarn with acceptable quality and reasonable cost. A good quality blend requires the use of adequate machines, objective techniques to select bales and knowledge of its characteristics.

As awareness grows regarding the significance of blended products in the textile industry, along with the increase in production costs, it has become increasingly crucial to achieve economic and high-quality blends using different types of cotton (Azzouz et al., n.d.). Blending fibres in the textile industry involves combining various fibres that have distinct properties to enhance or achieve specific qualities in the resulting yarn or its processing performance. Fabrics made from blended yarn may have superior characteristics compared to those made from a single fibre (Duru Baykal & Babaarslan Rızvan Erol, n.d.). The process of blending cotton with other fibres aims to improve various properties in fabric products, such as drape, comfort, durability, and dyeability. If a new fibre can successfully blend with cotton, it would be a significant breakthrough in the field of textiles. Blending different types of fibres is a widely used approach to enhance both the performance and aesthetic qualities of textile fabric.

Blended yarns made from natural and man-made fibres have a significant advantage in combining the desirable properties of both fibre components, such as comfort with easy care properties. These advantages lead to an increased variety of products, which results in a stronger marketing advantage (Ning Pan et al., 2000). Prediction of the mechanical properties of blended yarns using proposed theoretical and mathematical models were also been studied by numerous authors (Kemp & Owen, 1955; Pan & Postle, 1995; Ratnam et al., 1968; Xu & Ma, n.d.).

The blending mechanism of different natural fibres is still a topic of further research as there are still some gaps in knowledge. For instance, Prakash et al. conducted a study which found that the quality of a 50/50% blend of bamboo and cotton yarn was comparable to that of a 100% cotton yarn. This could be attributed to the fact that the mechanical properties of the blended yarn are primarily determined by the properties of the individual fibers and the method of their combination. In the case of a 50:50 bamboo-cotton blend, the properties of the blended yarn are an average of the properties of the individual fibers. Therefore, the properties of the 100% cotton yarn and the 50:50 bamboo-cotton blended yarn may be similar.

It’s important to note that other factors such as the fibre quality, spinning process, and plying process may also affect the final properties of the yarn. Also, the yarn appearance and handle may vary depending on the ratio of the fibres and the spinning process but the properties should remain similar.

2.3.6.1 PALF-Cotton

While PALF has shown excellent physical properties, its larger diameter makes it have a poor choice for making woven yarn. In order to improve the quality ratio, blending of finer fibres such as cotton can be a solution as it would provide more fibres per section (Jalil et al., 2021).

Pineapple leaf fibre (PALF), similar to that of cotton fibres, is relatively inexpensive and abundantly available (Todkar & Patil, 2019). Due to the advantages of PALF and their chemical similarity to cotton, a fabric made of PALF and cotton blend was used in this experiment.

2.3.6.2 Jute – Cotton

Blending jute with cotton fibre may be an acceptable way of jute diversification by which value added products can be produced. Jute fibres have several advantages like a lustrous golden appearance, high tenacity and good properties. Hence the techniques of blending and softening could as utilize to upgrade the quality of jute and thus form a new class of jute-based fabrics having an expanding market within and outside the country (Ahmed Ullah et al., 2016). In a study on the blending of jute with cotton, the study revealed that, the characteristics of blended yarn fabric can be used as fully cotton fabric which may reduce the dependability on importable cotton fibre. On the whole, it may be said that, not only depending on the cotton fibre, but also jute-cotton blending may reduce the dependability on 100% cotton yarn (Ahmed Ullah et al., 2016). Another study showed that the thermal conductivity of a composite made of a blend of jute and cotton rose as the number of layers increased, with the exception of the 50/50 jute-cotton combination, providing potential of being used as thermal insulation with high structural properties (Hu et al., 2012).

It is safe to say that the blending of jute with cotton may have applications in many areas of industry due to its economic benefits as well as its improved performance. Hence, we incorporated a blend of cotton in our jute fabric.

2.3.7 Effect of weave patterns

In contrast to individual fibre or bundle fibre, the fibres are highly entangled with one another in woven fabrics, and the resulting composites exhibit higher strength. Shahinur *et al.* showed the results of incorporating several layers of jute fabrics with polyester using the hand lay-up method. It was evidently seen that as the number of layers in the composite increased, so did the tensile strength and modulus (Shahinur et al., 2022). The fabrication of multilayer thermoplastic composites uses a variety of woven fabrics with highly interconnected threads, such as plain, satin, and twill. Their mechanical properties depend on fabric weight, fabric structure, yarn count, types of fibre, processing technique and also the type of matrix used. The cross-section of a six-layer plain-weave jute fabric composite was studied by Sayem *et al*. where the layers entirely immersed within the layered matrix (Sayem et al., 2020). Jute nonwoven fabric was also used with thermoplastics as a promising reinforcement since it is more flexible, lightweight, and affordable than traditional woven fabrics (Rawal & Sayeed, 2014; Sayeed et al., 2014).

2.4 Applications of hybrid natural fibre epoxy – based composites

Due to the numerous uses in anything from home goods to automobiles (Akampumuza et al., 2017; Pickering et al., 2016; B. S. Reddy et al., 2020), aerospace (Pickering et al., 2016) and also marine sectors (Kappenthuler & Seeger, 2021), the biocomposite industry has recently attracted a lot of interest. Comparative analysis of Natural Fibre Based epoxy composite has also deemed that they can display better performance automotive and aerospace products (Saba et al., 2016).

Table 3 shows various applications where natural fibres were used to make different components of several industries such as infrastructure, packaging, clothing, sports, fashion, automobiles, marine and aerospace.

	Fibres	Applications	References
Natural fibre composite applications	Hemp fibre	• Construction work • Textiles • Cordage • Paper and packaging • Furniture	(Balea et al., 2019),(Ardanuy et al., 2011)
	Oil palm fibre	• Building materials- window, frames, roofing, siding	(Ardanuy et al., 2015)
	Wood fibre	• Window frame • Panels • Decking • Railing system • Door shutters	(Madsen & Gamstedt, 2013)
	Flax fibre	• Tennis racket • Bicycle frame • Fork • Seat post • Snow boating • Window material • Aeronautical industry • Automotive industry	(More, 2022)
	Rice husk fibre	• Building panels • Bricks • Proof cement • Window frame • Railing system • Roofing units	(Verma et al., 2013)
	Bagasse fibre	• Window frame • Cement Panels • Decking • Railing system • Fencing	(Devadiga et al., 2020),(Prasad et al., 2020)
	Sisal fibre	• Construction in industries- panels, doors, shutting plate, roofing etc.	(Balea et al., 2019)
	Kenaf fibre	• Packaging material • Mobile cases • Bags • Insulations • Ropes, canvas	(Akil et al., 2011)
	Stalk fibre/ Banana fibre	• Building panel • Bricks • Fire resistance fibre boards • Constructing drains and pipelines	(Verma et al., 2013)
	Cotton fibre	• Furniture in industries • Textile and yarn • Goods and cordage	(Radoor et al., 2022)
	Coir fibre	• Building panels • Flush door shutter • Storage tank • Packing material	(Verma et al., 2013)
	Jute fibre	• Building panels • Packaging • Chip board	(Chandekar et al., 2020)
	Ramie fibre	• Industrial sewing thread • Packing material • Fishing net	(Sathish et al., 2021)

Table 11: Application of different natural fibre

Chapter 3

Methodology

3.1 Research methodology

The following procedures were followed through the whole experiment:

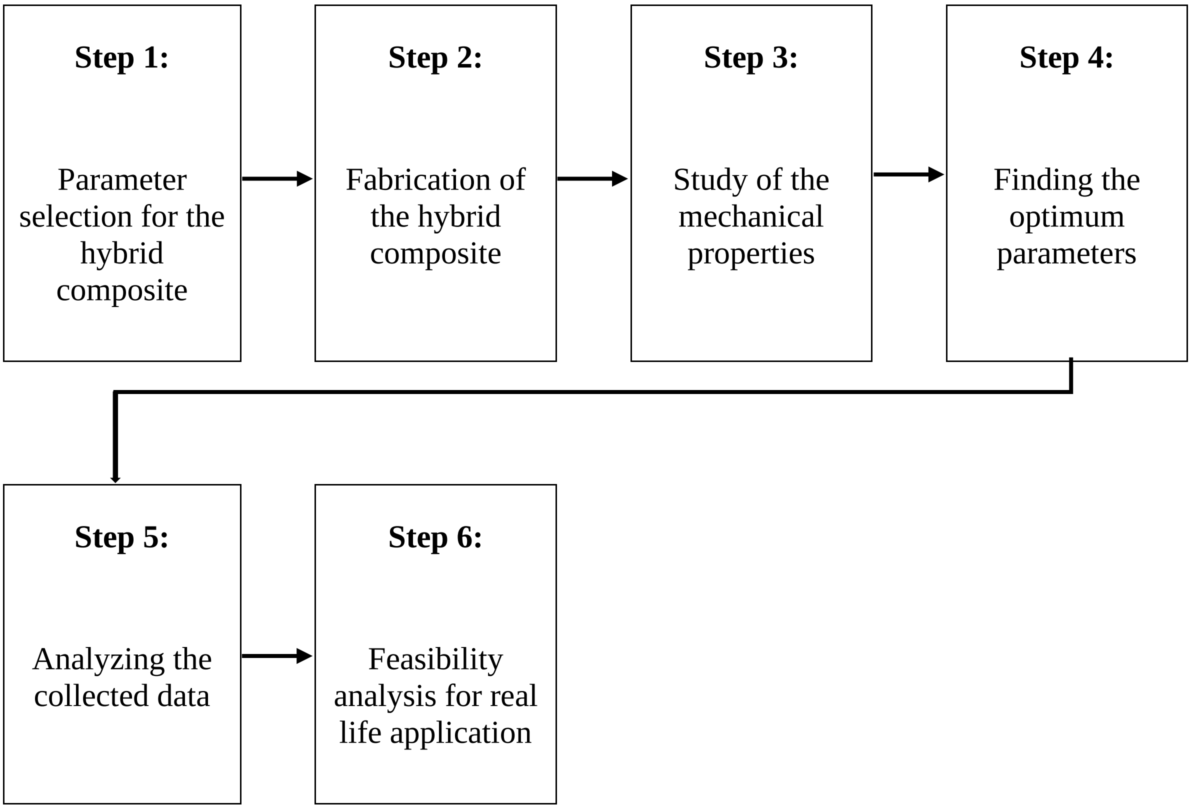


Figure 5: Research Methodology

3.2 Theoretical background

3.2.1 Vacuum bagging and wet – lay up

This method can be used to create composite materials that are both natural and synthetic. A releasing agent is sprayed on the surface, and thin plastic sheets are placed at the top and bottom to prevent the mould from adhering to them. The resin and hardener mixture were then poured over the reinforced fibre, which had been laid out on top of a plastic sheet (peel ply), and spread evenly with a brush or scrape. To get rid of the extra matrix, a second layer of fibre was added on top of the first and rolled. Up till the desired thickness is reached, this process is repeated. The top of the mould was covered with plastic sheet, and pressure was then applied. After around 24 hours, the mould was prepared (Gogna et al., 2019).

In order to create void-free composites in big and complicated moulds, such as wind turbine blades, boat hulls, the frontal sections of a high-speed railway engine, and other offshore constructions, vacuum bagging process is incorporated as a follow – up procedure. The whole process of vacuum bagging and wet lay – up is shown in Figure 3.

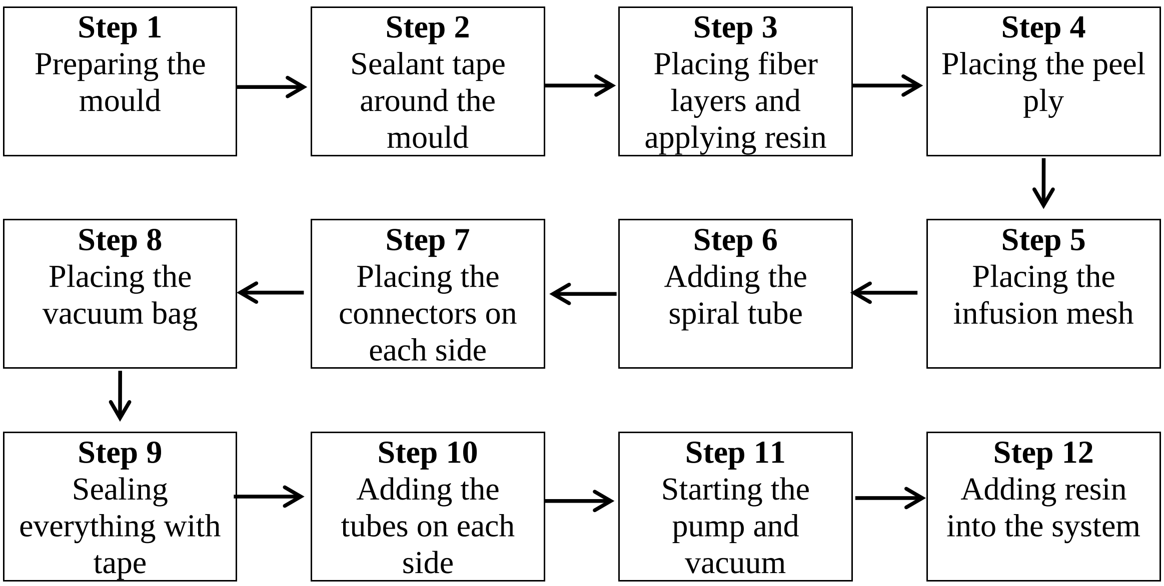


Figure 6: Flowchart showing the experimental procedure for fabricating the natural fibre reinforced hybrid

3.3 Experimental procedure

3.3.1 Materials

3.3.1.1 Reinforcements

A radar plot showing some key mechanical performance taken from Table 2 is shown in Figure 7.

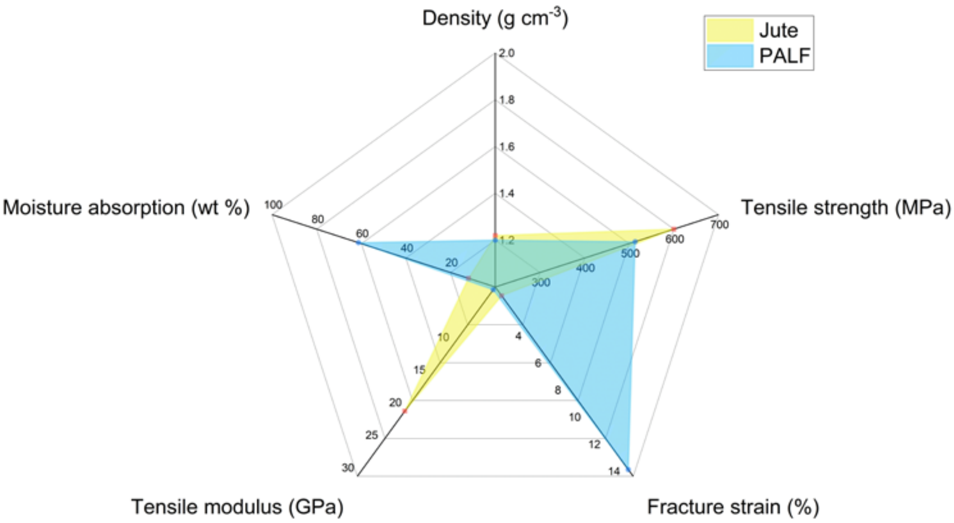


Figure 7: Radar plot of jute fibre and PALF in terms of (a) Density, (b) Tensile strength, (c) Fracture strain, (d) Tensile modulus & (e) Moisture absorption

Jute fibre

Figure 8: Woven jute-cotton fabric

Jute is a potential fibre among all natural reinforcing materials because of its reasonable price and easy access in the required form. It is stronger and more elastic than synthetic polymers, which are typically employed as matrices (Munikenche Gowda et al., 1999). The jute-fabric used in this experiment was sourced from Faridpur. The fibre content ratio in the collected material is 55/45 jute/cotton blend.

Properties	
Fibre orientation	Twill (Woven)
GSM (Gram per Square Meter)	250
Extraction process	Mechanical extraction
CSP (Count Strength Product)	--

Table 12: Properties of woven jute-cotton fabric (collected from Classical Handmade Products BD Ltd)

Pineapple fibre

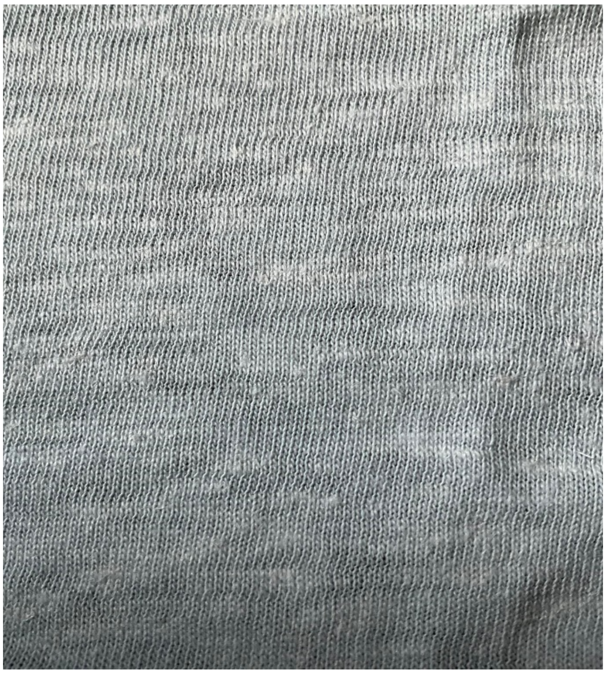


Figure 9: Woven PALF-cotton fabric

PALF, or pineapple leaf fibres, have excellent mechanical qualities. Pineapple fibres has greater mechanical qualities because of its high cellulose content and relatively low microfibrillar angle. The pineapple fabric used in this experiment was sourced from Madhupur, Tangail. However, we have collected them from Classical Handmade Products BD Ltd. The fibres content ratio in the collected material is 20/80 pineapple/cotton.

Properties	
Fibre orientation	Knitted
GSM (Gram per Square Meter)	160
Extraction process	Mechanical extraction
CSP (Count Strength Product)	1300

Table 13: Properties of woven PALF-cotton fabric (collected from Classical Handmade Products BD Ltd)

Epoxy

Figure 10: Araldite resin AW 106 IN and Hardener HV 953U

In this work, the Epoxy resin – Araldite AW 106 IN is used as matrix, Araldite HV 953 U as hardener. Araldite AW 106 / Hardener HV 953 U is a multipurpose, industrial grade, two component, room temperature curing adhesive of high strength and toughness. Table 4 below contains the pertinent technical information.

	Araldite AW 106	**Hardener HV 953 U**	**2011(mixed)**
Color (visual)	Neutral	Pale yellow	Pale yellow
Specific gravity	ca. 1.15	ca. 0.95	Ca. 1.05
Viscosity at 25°C	30-50	20-35	30-45
Pot life (100 gm at 25° C)	-	-	Ca. 100 minutes
Lap shear strength at 23°C	-	-	>19 MPa

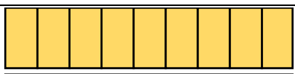
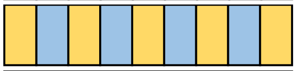
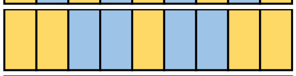
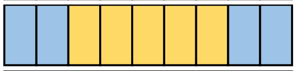
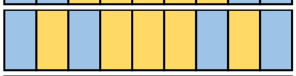
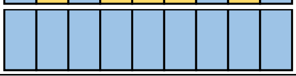
Table 14: General Properties of Araldite AW 106 IN resin/HV 953 hardener

3.4 Manufacturing procedure

3.4.1 Stacking sequence

The fabric plies are cut in the size of $250 \times 100\ cm^2$ from the jute – cotton and pineapple – cotton fabric. 4 PALF fabric mats and 5 jute fabric mats are taken for the making of the hybrid NFRCs. Then the fabrics, hardener, and resin were weighed. The density of the composites was measured with a mass balance (accuracy 0.01g) and dividing it with the volume of the composite. The varying stacking sequence, density, and fibre volume fractions are shown in Table 13.

Table 15: Design parameters of hybrid and non-hybrid NFRC with varying hybrid ratios and stacking sequence.

Laminate designation	Stacking sequence	Density (kg/m^3)	Plies number ratio (PALF/jute fibre)	Fibre volume fraction	Volume composition
JFRP		1.076	0/9	0.996	0/100
4P5J – 1		1.044	4/5	0.684	44.4/55.55
4P5J – 2		1.074	4/5	0.668	44.4/55.55
4P5J – 3		1.068	4/5	0.639	44.4/55.55
4P5J – 4		1.131	4/5	0.64	44.4/55.55
PFRP		1.181	9/0	0.810	100/0

3.4.2 Fabrication of the hybrid NFRCs

Hardener and epoxy were mixed in a bowl with the help of stirrer. Care should be taken to avoiding the formation of air bubbles, which can get stuck in a matrix, increasing the void content and decreasing the mechanical performance of that material. The pineapple-cotton fabric ply is placed on the die and the resin-hardener mixture was applied by wet lay-up process. Then the jute-cotton fabric ply was laid and rolled. Here rolling was done by a cylindrical copper rod. This process is repeated until nine (4 pineapple-cotton and 5 jute-cotton) alternating fabrics have been arranged. A polymer coating is put on top of the ply for good surface finish and then vacuum bagging was done. After ensuring no leakage is in the vacuum system, the specimen is left it for 12 hrs for curing and hardening. After curing, the specimens will be cut by using the laser cutter into dimensions conforming to ASTM standards. A schematic of the experimental set – up is shown in Figure 11. The experimental procedure is shown in Figure 12.

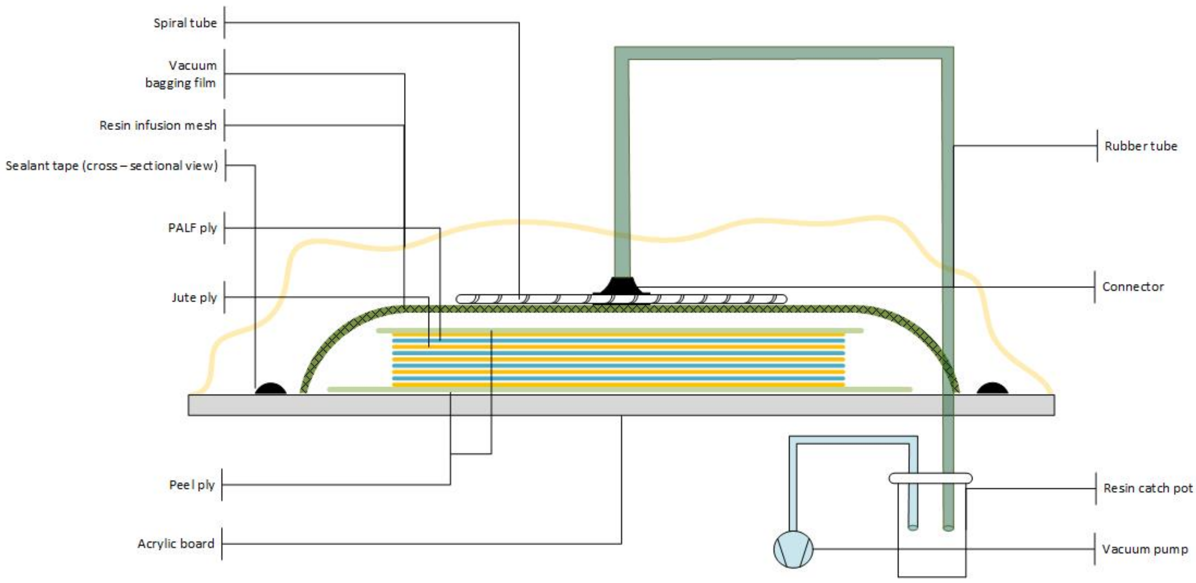


Figure 11: Schematic of experimental set - up used to create the natural fibre reinforced composites and its hybrids

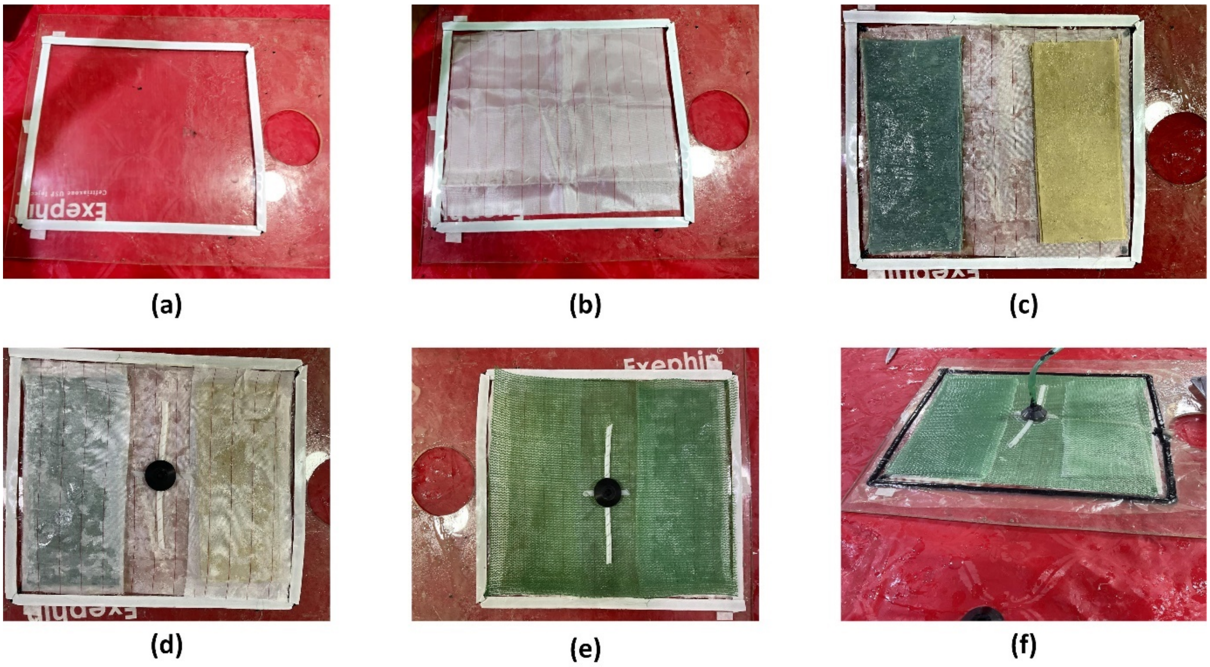


Figure 12: Fabrication of NFRCs (a) Using sealant tape to fix the boundary, (b) Applying peel ply, (c) Putting reinforcement pre – pregs, (d) Applying a second, smaller layer of peel ply, (e) Applying infusion mesh and (f) Peeling off sealant tape and putting vacuum bagging film, connect, spiral tube and rubber tube.

3.4.3 Specimen preparation

There are mainly two types of cutting methods in case of composites: conventional and unconventional cutting techniques. One of the conventional methods of cutting this type of composite is using a shear cutter. Shear cutters are not a dependable approach for cutting applications due to the application of unwanted pressure from the contact edge. Furthermore, the method has a limited ability to create intricate geometrical elements and, the whole shear cutting process has a lengthy processing time and significant difficulty in cutting thicker materials (Tamrin et al., 2020). Different frictional forces are produced in conventional machining which decreases their cutting efficiency.

(a) **(b)**

Figure 13: (a) Schematic of CO2 laser cutting & (b) Actual photo of CO2 laser cutting (Y. Singh et al., 2021)

In contrast to conventional techniques, laser cutting offers a potential alternative for cutting composites that can address their limitations. As a thermally dependent method, laser cutting has the ability to contour cut a wide range of materials. Due to its high cut quality and cost – effectiveness in large – scale batch processing, it has become a widely adopted non – traditional machining technique in industry. The many benefits of laser cutting, such as: ease of operation, minimal heat – affected zone, exceptional precision, low waste, minimal deformation, ease of automation, low noise, and durability, have made it a popular choice for modern manufacturing units, facilitated by ongoing technological advancements in laser cutting machines (Y. Singh et al., 2022)

In this experiment, CO_2 laser cutting was used in order to get higher precision on the cutting edges with accurate dimensions and lesser material waste. The composites were marked and drawn before cutting according to the pre – set ASTM standards chosen for tensile, flexural, impact and water absorption test. Then the composites were cut in the Quantiumlaser (model 6040) cutting machine maintaining all the safety precautions. The cutting parameters used are given in Table 10.

Table 16: Cutting parameters used during specimen preparation

Parameters	**Value**
Laser intensity Speed	90% 30 m/s

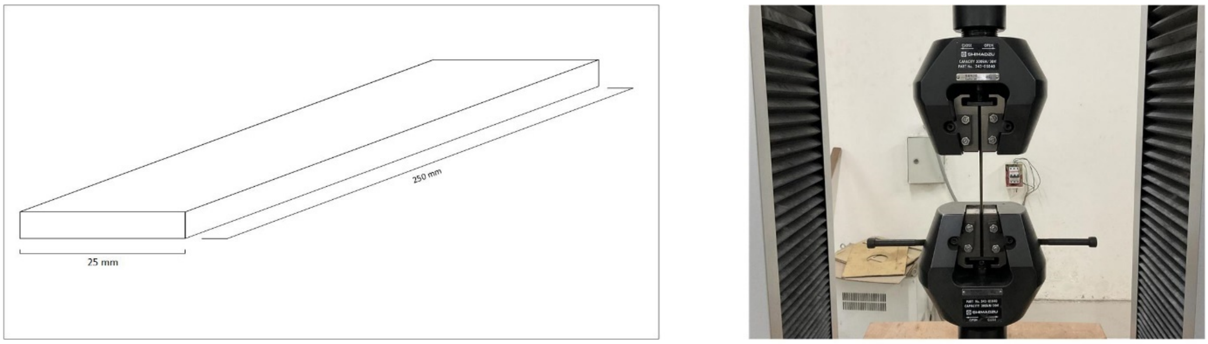
For the cutting process, at first, the composite was loaded onto the cutting bed of the laser cutting machine. Then the composite and the nozzle were positioned correctly over the material to ensure accurate cutting. The laser was beam was set in pulsed mode. The laser beam cut through the material, following the path that was marked beforehand. Choudhury *et al* used a single-pass and double-pass 500 W CO_2 laser beam to assess the cut quality of GFRP composite. They discovered that, in comparison to single-pass laser beams, double-pass laser beams generate cut surfaces of higher quality and cause fewer burns (Choudhury & Chuan, 2013). In order to get finer cut, the composites were cut using double – pass method in this experiment. The excess materials were removed after the desired pieces were cut.

3.5 Characterization

3.5.1 Mechanical characterization

3.5.1.1 Tensile test

In general, tensile testing is defined as "the application of a uniaxial tension force to quantify the performance of a test sample, up until it gives or breaks". Uniaxial tensile tests on rectangular specimen were conducted on a Shimadzu AGX – V2 series (Shimadzu corporation, Tokyo, Japan) with a standard head displacement rate of 2 mm/min. The goal is to fully comprehend the material's tensile characteristics, including yield strength, ductility, and tensile strain. The dimensions of the specimen are taken according to ASTM D3039/D3039M-17 (*Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials 1*, n.d.) having dimension of 250 × 25. A schematic of the test specimen and the experimental set – up is shown in Figure 13.



(a) **(b)**

Figure 14: (a) Schematic of tensile specimen & (b) Experimental set - up for tensile test

3.5.1.2 Flexural test

The flexural properties of a composite material relate to its capacity to withstand stress caused by bending. This is a fundamental mechanical property that is often evaluated through a bending test involving either three or four points of contact. The flexural properties are influenced by various factors such as the type, quantity, and alignment of the reinforcing fibres or particles, the characteristics of the matrix material, and the production process used to make the composite (Sayeed et al., 2023; Yang et al., 2019).

The main flexural properties of a composite include flexural strength, flexural modulus, and flexural specific modulus. Flexural strength represents the maximum amount of stress that a composite can tolerate before breaking when bent, while flexural modulus is an indicator of a composite's resistance to bending. Meanwhile, flexural toughness gauges how much a material can be stretched or bent under a given load before it breaks, relative to its weight. These properties are crucial in many applications where composites experience bending loads, such as in construction, automotive, and aerospace industries.

In this experiment, a three-point bending test was performed on the Shimadzu AGX – V2 series (Shimadzu corporation, Tokyo, Japan) in order to get the flexural properties of the composites where a bar of the composite is rested on two supports and it's loaded by means of a loading nose midway between the supports as shown in Figure 12.

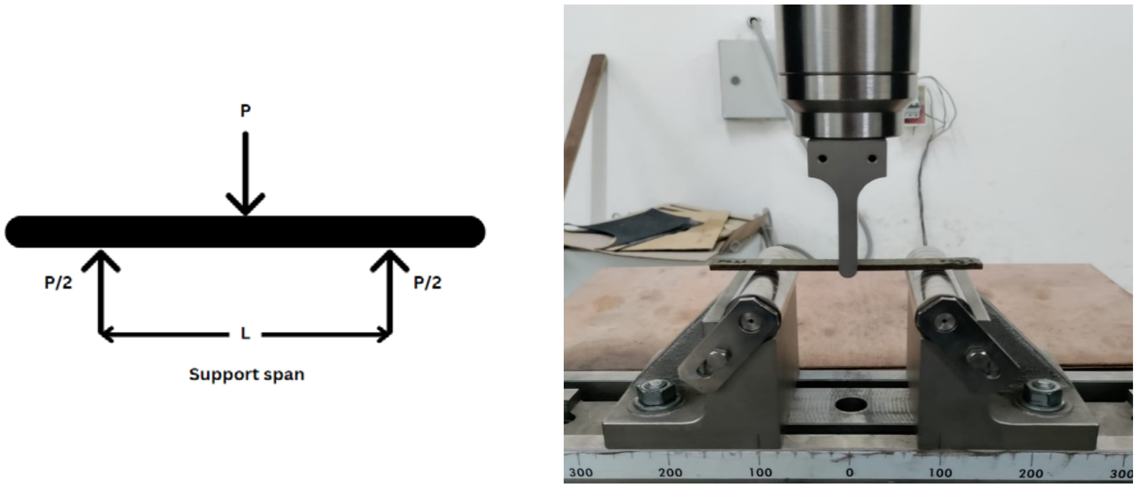


Figure 15: Three - point bend test set - up

The sample size for the flexural test was chosen according to the ASTM standard D6856 (*Standard Test Method for Flexural Properties of Polymer Matrix Composite Materials 1*, n.d.) where the span-to-thickness ratio is 25:1, the standard specimen thickness is 4 mm on average, and the standard width is 13 mm on average with the specimen length of 100 mm.

During the flexural test or three-point bending test, the sample was placed horizontally on two supports with a specified distance (100 mm) between them as shown in the above diagram. A load was applied at the midpoint of the sample at the cross-head speed of 5 mm/min, through a third support that exerts a downward force. The force is applied at a constant rate until the sample fractures. During the test, the load and deflection of the sample are continuously recorded. After the test, the recorded data is analysed to calculate the flexural properties of the material.

Calculations:

The flexural stresses of the composites were calculated using the following equation:

$$\sigma = \frac{3PL}{bh^2}$$

where,

σ = stress at the outer surface at mid-span, MPa;

P = applied force, N;

L = support span, mm;

b = width of beam, mm and

h = thickness of beam, mm.

The flexural modulus is calculated using the following equation,

$$E_f = \frac{\Delta\sigma}{\Delta\varepsilon}$$

where,

E_f = flexural modulus of elasticity, MPa;

$\Delta\sigma$ = difference in flexural stress between the two selected strain points, MPa;

$\Delta\varepsilon$ = difference between the two selected strain points (nominally 0.002).

The specific modulus is calculated by the following equation,

$$SM = \frac{E_f}{\rho}$$

where,

SM = Specific modulus of elasticity;

E_f = flexural modulus of elasticity, MPa;

ρ = density of the composite, kg/m^3.

3.5.1.3 Impact test

The capacity of a composite to endure the impact or shock loading is evaluated by an important factor called the impact energy of composites. In this experiment, modified Charpy test was performed in order to get the impact energy of the composites.

According to the International Standard ISO:1997 Standard (Yang et al., 2019), the unnotched samples were cut by CO_2 laser cutting and the test were carried out on an impact testing machine: model CEAST 9050, Instron Corp., Norwood, MA, USA. The dimensions for each sample were taken $75 \times 10 \times 4$ mm^3 with a span-to-depth ratio set at 18.

(a)

(b)

Figure 16: (a) Pendulum impact testing machine & (b) Fixture to set impact specimen.

During the test, the sample is placed in the testing machine and is positioned horizontally and supported at both ends. The pendulum of the machine is then released from a height at 5.35 m/sec and swings down to strike the sample. The sample gets damaged or distorted due to the impact, which causes the pendulum to swing upward on the opposite side of the sample. The variation in the height of the pendulum prior and after the impact is measured to determine the energy that the sample has absorbed.

Calculations:

The impact energy of the composites is calculated using the following equation,

$$E = (\text{Total division number} \times 1 \text{ division value}) \div \text{Area of the specimen}$$

E = Impact energy;

Total division number = distance travelled by the hammer;

1 division value = **2 J.**

3.6 Water absorption test

The hydrophilic nature of the natural fibres can have significant effect on the performance of a composite that is reinforced with them (Faruk et al., 2012). The amount of water absorbed under particular circumstances can be calculated using the water absorption. The type of plastic, the additives used, the temperature, and the duration of exposure are factors that affect water absorption (Senthiil & Sirsshti, n.d.). The test sample dimension is taken in accordance to ASTM D5229/D5229M-20 (*Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials 1*, n.d.) with a dimension of 50×50 mm^2. The samples were immersed in a body of water at ambient temperature and the mass of each sample were taken for 12 days.

The following equation was used to calculate the water absorption in percentage:

$$\text{Water absorption (\%)} = \frac{m_2 - m_1}{m_1} \times 100$$

3.7 Scanning electron microscopy (SEM)

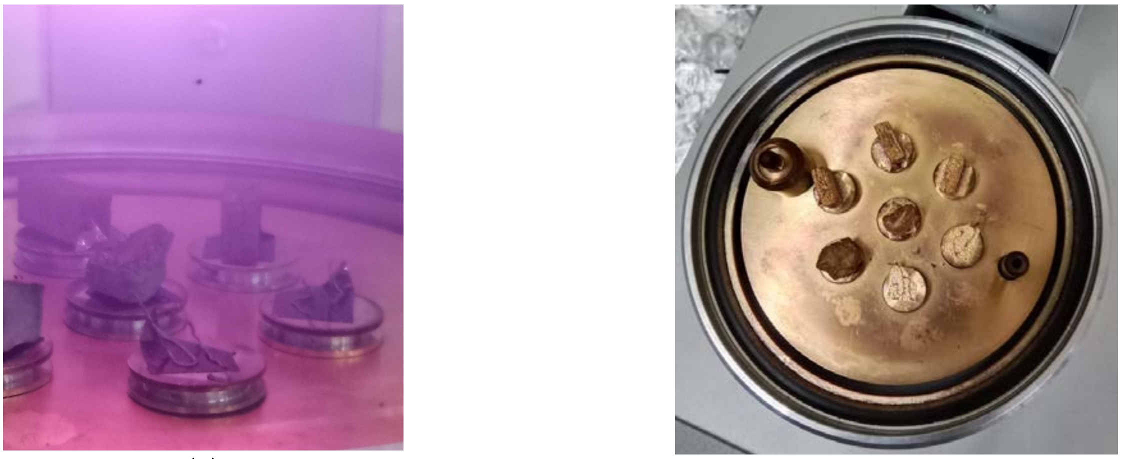
The microstructure of composite laminates was examined using SEM imaging. A sample cut from the laminates was mounted in acrylic resin to reveal the cross-section through the thickness. The SEM (Scanning Electron Microscopy) procedure for the composites involved the following processes:

At the outset of the SEM imaging procedure, the specimens selected for analysis underwent meticulous preparation. In particular, a total of five samples were chosen, including one jute yarn and one pineapple yarn to facilitate an assessment of the fabrics' fibre distribution and architecture, as well as a pineapple fibre reinforced epoxy polymer composite (PFRP), a jute fibre reinforced epoxy polymer composite (JFRP), and a pineapple-jute fibre reinforced hybrid composite.

The yarns were derived from the fabrics employed in the experiment, while the composite samples were obtained via CO_2 laser cutting from the fractured portions of the tensile testing specimens, with a dimension of 10 mm as shown in Figure 16

Figure 17: SEM samples before gold sputtering

Subsequently, these samples were mounted onto a sample holder. To prevent charging during SEM imaging, a conductive material was coated onto the soft matrix material of the composites; in this case, gold sputtering was employed for the purpose of adding a thin layer of gold to the material surface. The process of sputtering and the finished samples are shown in Figure 17.



(a) **(b)**

Figure 18: (a) Sputtering of SEM samples & (b) Sputtered samples

The samples were then placed into the SEM chamber, where an electron beam is focused onto the surface of the composite. This beam causes secondary electrons to be emitted from the surface, which are then detected by the SEM detector. The detector creates an image based on the pattern of electrons emitted from the surface, which provides information about the microstructure of the composites.

Various magnifications were employed for the scanning of the yarns and composite materials, ranging from 50 µm to 500 µm. Specifically, for the yarns, the magnifications utilized were 50 µm, 100 µm, and 200 µm, whereas for the composites, the magnifications ranged from 50µm to 500µm, including 50µm, 100µm, 200µm, and 500µm. In order to optimize the quality of the results, images were captured at diverse positions and magnifications.

Chapter 4

Results and discussion

The hybrid natural fibre reinforced epoxy – based composites were fabricated in various hybrid ratios of PALF/jute as shown in Table 1. The fibre, resin and void volume fraction was found using Equation (1).

$$V_f = \left(\frac{W_{f,jute}}{\rho_f, jute} + \frac{W_{f,pineapple}}{\rho_f, pineapple}\right).\rho_c$$

$$V_f = \left(1 - W_f\right).\frac{\rho_c}{\rho_r} \qquad (1)$$

$$V_f = 1 - (V_f + V_r)$$

4.1 Tensile properties of the composite

The typical tensile properties of both the pure jute – cotton, hybrid and pure PALF - cotton composite along the longitudinal direction is shown in Figure 19a – d shows. With an increase in the volume fraction of jute, the tensile modulus and strength was observed to be 1.45 GPa and 35.16 MPa respectively for JFRP to 1.37 GPa and 31.91 MPa respectively for PFRP, therefore showing, as expected, the strengthening and stiffening effect of incorporating jute into the hybrid. PFRP, however, was able to show greater ductiliy and no defenitive yield point.

The highest breaking stress was for jute (35.16 MPa) and the lowest was for 4P5J – 1 (27.73 MPa) , showing a 26.67% and only a 5.84% decrease in tensile strength and modulus respectively. It was observed from Figure c – d that, even though the fibre volume fraction were same for the hybrids, the strength and modulus varies with stacking sequence, with the highest being for 4P5J – 2 (32.16 MPa). This may be due to poor interfacial adhesion between the jute and PALF plies, which is more apparent in 4P5J – 1, resulting in its poor breaking stress. With stacking more plies of the same type together, there is a general increase in tensile strength. Concentrating jute fibre plies on the outer edge or on the middle did not show any significant change in the tensile strength. However, the tensile modulus increased significantly (by 19.2%) when the jute fibre plies were concentrated on the outer edges (from 1.103 GPa (4P5J – 4) to 1.315 GPa (4P5J – 2)). Pure PALF (PFRP) also did not show much elongation (12.44%) when compared to pure JFRP (14.43%) but the hybrid composite was able to show the highest elongation for 4P5J – 4 (15.94%).

As the density of PALF is higher than jute, the hybrid composites can be marginally improved in specific strength and moduli when jute was incorporated in pure PALF composite, 4P5J – 2 showing the highest specific strength (29.94 MPa $(g.cm^{-3})^{-1}$) and 4P5J – 1 showing the highest specific moduli (1.24 GPa $(g.cm^{-3})^{-1}$).

Figure 20 shows the tensile fracture of the JFRP, 4P5J hybrids and PFRP. From visual inspection is was seen that fibre yarn pull – out was more apparent from the jute plies during tensile failure with PFRP showing almost brittle fracture. Figure 21 shows the SEM images of the fabricated hybrid composites. Compared to the smooth fracture of PFRP (shown in Figure 22(b)), suggesting that brittle fracture is dominant, the hybrid composite 4P5J – 1 shows more a more complex fracture pattern showing jute fibre pull – out, jute yarn pull – out and ductile fracture of jute yarn. These features are more defined in the fracture morphology of pure jute reinforced composite (shown in Figure 22(a)).

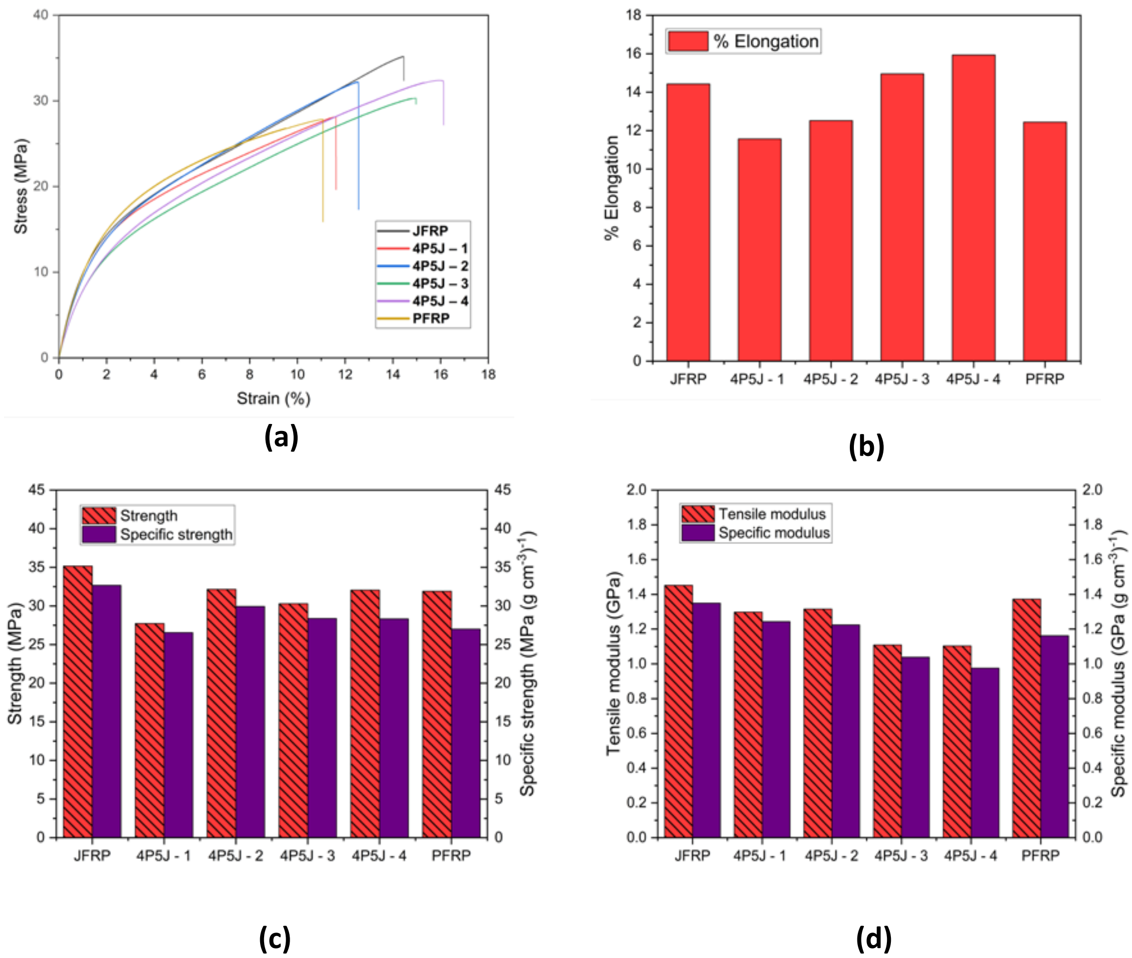


Figure 19: (a) Tensile stress - strain curve of fabricated composites, (b) % Elongation of fabricated composites, (c) Strength (left) and specific tensile strength (right) of fabrication composites & (d) Tensile modulus (left) and specific tensile modulus (right) of fabricated composites.

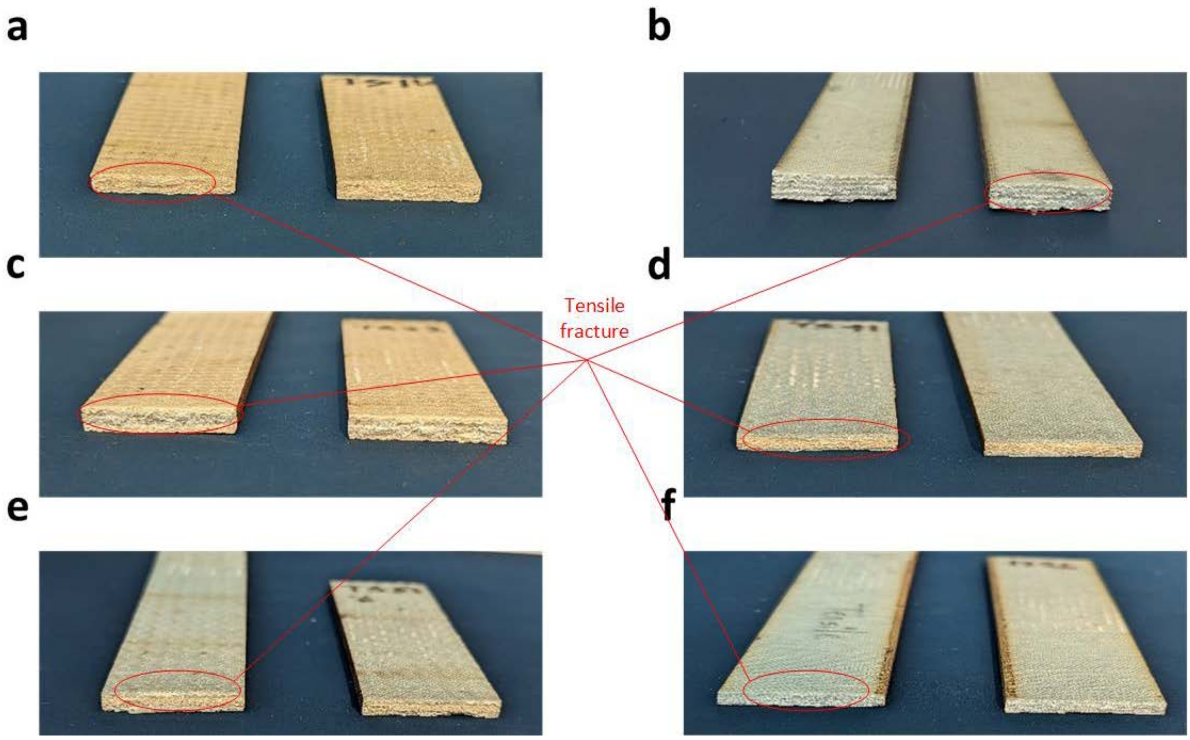


Figure 20: Tensile fracture of (a) J9 (b) J1P1J1P1J1P1J1P1J1 (c) J2P2J1J2P2 (d) P2J5P2 (e) P1J1P1J3P1J1P1 (f) P9

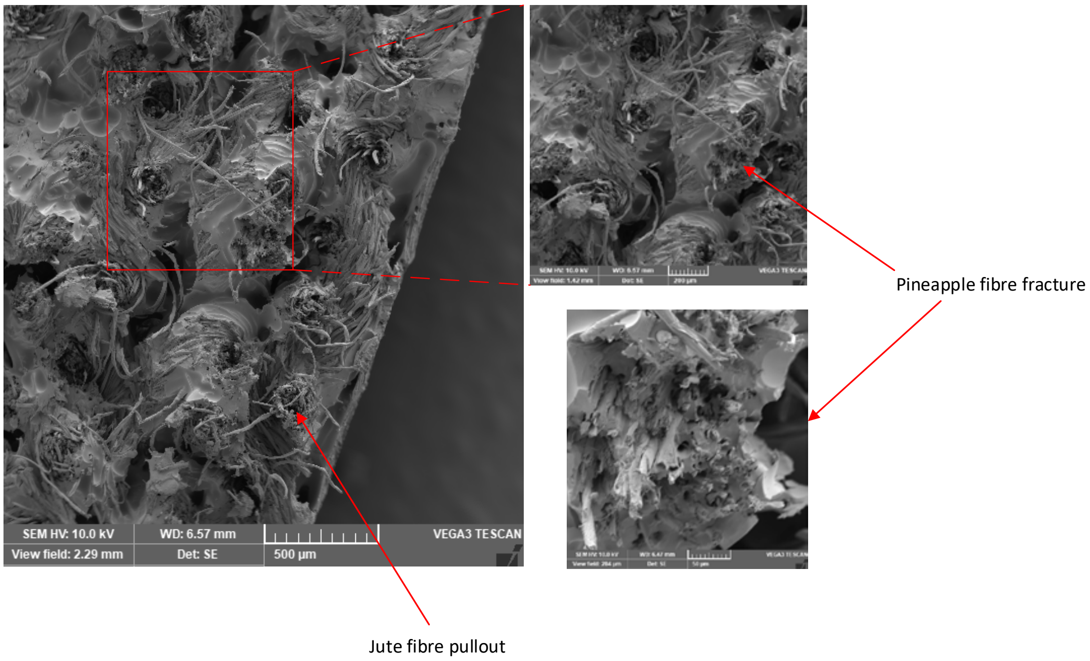


Figure 21: SEM image of tensile fracture of hybrid composite 4P5J - 1 at 500 µm (left panel), 200 µm (top right panel) & 50 µm (bottom right panel)

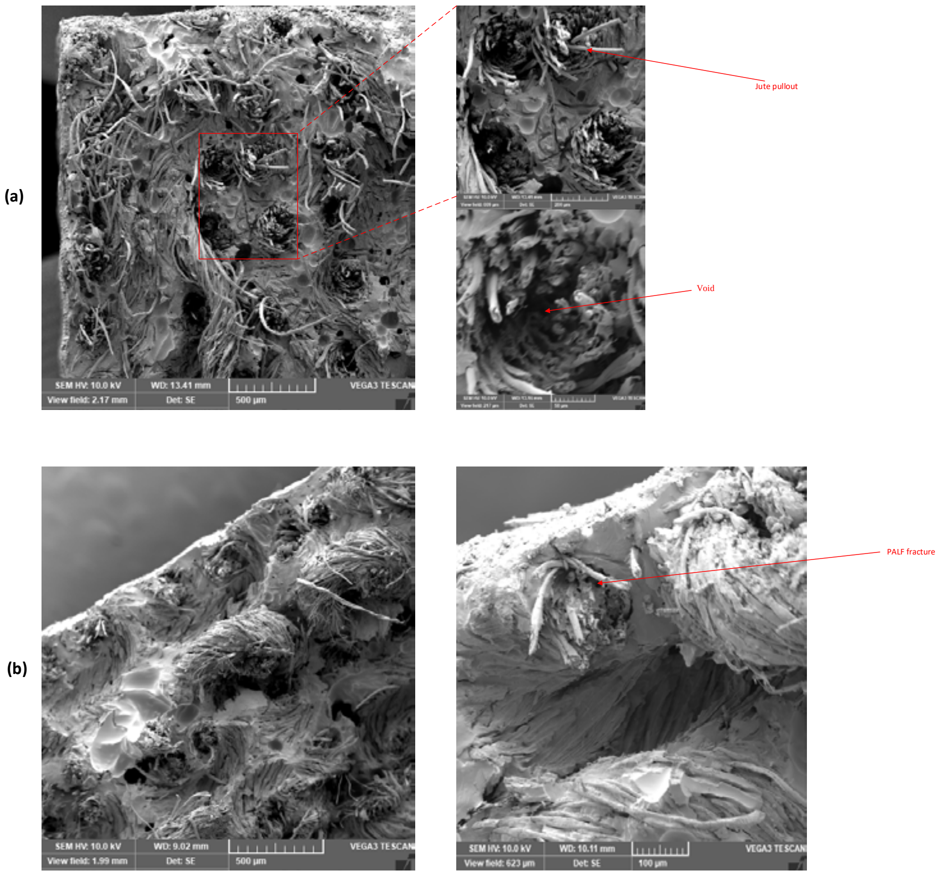
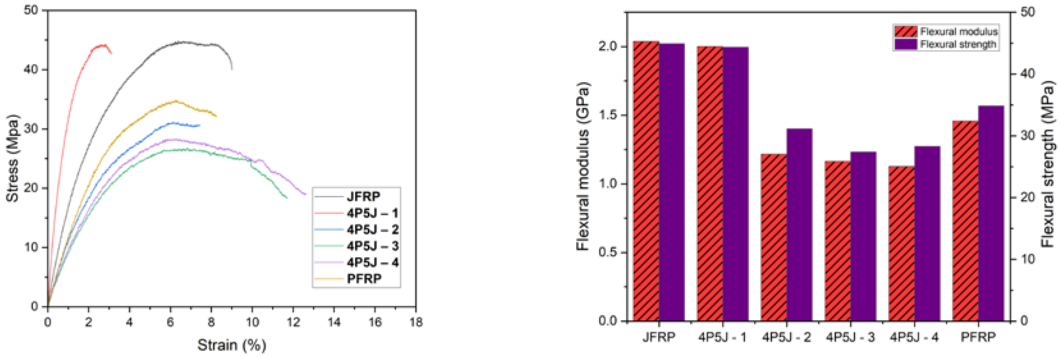


Figure 22: SEM image of (a) JFRP composites at 200 µm, 100 µm & 50 µm; (b) 200 µm & 100 µm

Overall, the hybridisation of jute and pineapple to make a composite resulted in a significant improvement in elongation with a slight decrease in tensile strength and moduli.

4.2 Flexural properties of the composite

In general, structural materials that possess high tensile strength also exhibit good performance when subjected to bending loads due to their favourable interaction with the fibre and matrix. The study investigated the flexural properties of the materials, which are presented in Figure 19 and 20. Unlike the tensile behaviour, the flexural stress-strain curve in Figure 19(a) demonstrated significant variations in flexural stress, with most composites exhibiting a continuous, one-step failure. Based on the figures, it is apparent that JFRP demonstrates the best flexural strength. While 4P5J-1, the first hybrid composite, exhibits the highest strength among the hybrid composites, even higher than PFRP. A decreasing trend in strength is observed from JFRP to 4P5J-3 and then an inverse trend from 4P5J-4 to PFRP in Figure 19(a).



(a) **(b)**

Figure 23: Typical flexural stress–strain curves of JFRP, 4P5J – 1, 4P5J – 2, 4P5J – 3, 4P5J – 4 and PFRP & (b) Modulus and strength of JFRP, 4P5J – 1, 4P5J – 2, 4P5J – 3, 4P5J – 4 and PFRP

Overall, the hybrid composites exhibited weaker flexural strength compared to the pure jute fibre reinforced composite. However, a similar trend was observed in terms of flexural modulus, except for the hybrid composites, which showed a decreasing trend. The first hybrid composite, 4P5J - 1, showed a significant improvement in flexural modulus, increasing by 78.57% from 1.12 GPa in 4P5J - 4 to 2.00 GPa with the only change being the variations in the stacking sequence. The flexural strength also increased by about 56.80%, from the lowest value of 28.29 MPa in 4P5J - 4 to 44.36 MPa in 4P5J-1. This may be due to the good interfacial adhesion between the jute and PALF reinforcement as well as due to the high elongation at break value of PALF, which helps the hybrid composite to withstand more load before failure. However, JFRP had the highest values in both flexural modulus and strength, with values of 2.03 GPa and 44.91 MPa, respectively.

Although all of the composites exhibited flexural strength values that were higher or comparable to those of PFRP, the increase in flexural strength was not as significant as the increase in tensile strength. The poor flexural strength observed in the composites could be a result of the relatively weaker fibre strength and/or insufficient interfacial adhesion (Sayeed et al., 2023). By comparing the results of the modulus and strength in the hybrid composites, it can be said that the stacking sequence could be optimized to achieve better flexural performance. Based on this idea, a combination of jute and PALF, in a 1:1 stacking sequence could be employed to withstand both the highest normal stress on the outside and the highest shear stress on the inside, particularly under three-point bending.

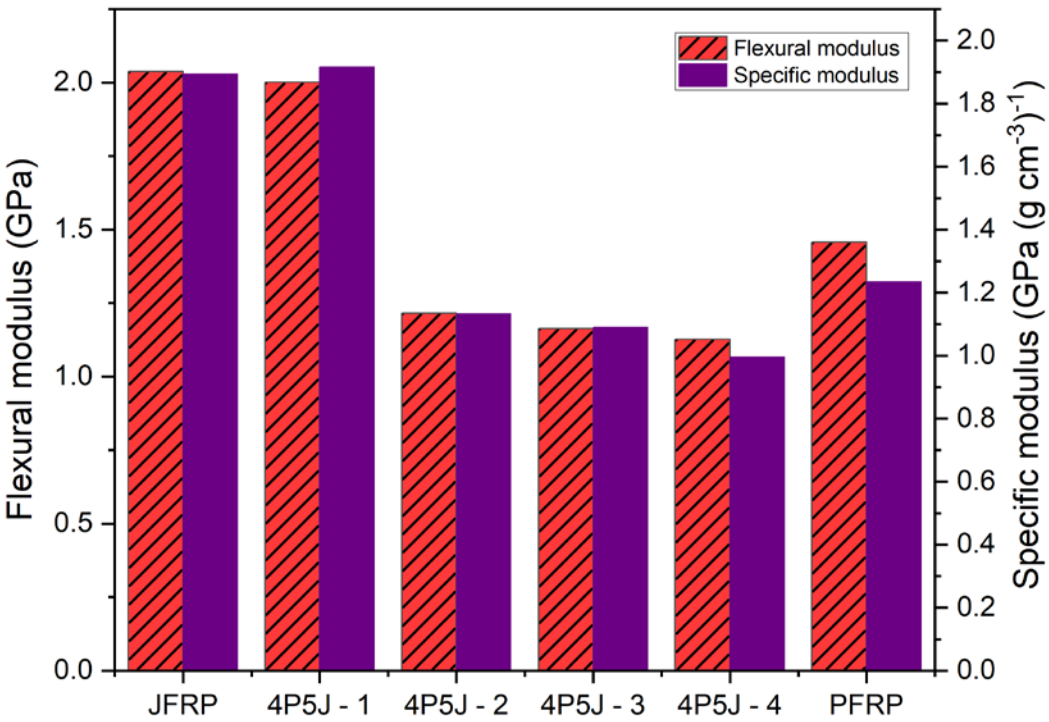


Figure 24: Modulus and specific modulus of JFRP, 4P5J – 1, 4P5J – 2, 4P5J – 3, 4P5J – 4 and PFRP having various

The specific modulus refers to a characteristic of a material that indicates the ratio of stiffness to weight. It is a measure of the material's ability to resist deformation under an applied load, relative to its weight. Specifically, it is calculated as the ratio of the material's modulus of elasticity (which is also known as Young's modulus) to its density.

The specific modulus of a material is a valuable parameter because it enables a comparison of the stiffness of materials that have varying densities. When a material has a high specific modulus value, it is more rigid for its weight, whereas a material with a low specific modulus value is less rigid for its weight.

From Figure 20, it can be observed that the specific modulus of the composites follows almost the same trend as flexural modulus except, the PFRP shows higher flexural modulus than specific modulus. The highest specific modulus can be seen in 4P5J – 1, 1.91 GPa which is higher than JFRP (1.89 GPa) and the 4P5J - 4 shows the lowest value being 0.99 GPa. Thus, it can be said that the first sample of hybrid composite 4P5J – 1 is the most rigid for its weight among the other composites. Here also, it can be observed that, the stacking sequence playing a significant role to achieve the best flexural value for the composites.

4.3 Impact properties of the composite

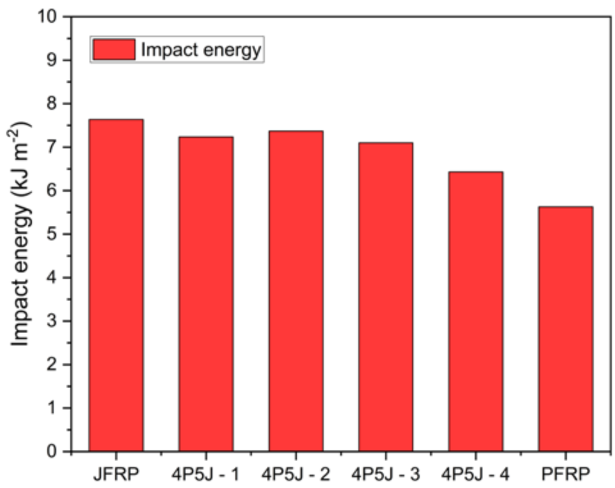


Figure 25: Impact energy of JFRP, 4P5J – 1, 4P5J – 2, 4P5J – 3, 4P5J – 4, PFRP.

The impact energy in Figure 25, shows the various impact strengths for the composites. The highest impact energy was found in JFRP. However, a decreasing trend can be noticed among the other composites except for 4P5J – 2 having closer impact energy (7.236 kJ/m^2) to jute (7.638 kJ/m^2) and pull out, whereas PFRP has the lowest value of 5.628 kJ/m^2. To examine this effect in the present hybrid-fibre composites, we utilized a modified Charpy impact experiment with unnotched test samples. Compared with the JFRP, which fractured in >7 kJ/m^2, hybrid-fibre composites and the PFRP displayed less plastic deformation in a prolonged fracture process of < 7 kJ/m^2. Similar to flexural deformation, the stacking sequence was also found to affect the impact performance of the 4P5J hybrid-fibre composites.

4.4 Water absorption properties of the composite

The composites were immersed in water for 12 days and the mass increase was recorded, shown in Figure 26. It was observed that all the composites showed a general increase in mass with each day. In about 6 days, the water absorption of hybrid composites and PFRP approached a plateau where the rate of absorption decreased but did not become constant. In the case of JFRP, we can see that the initial rate of water absorption is significantly high, reaching its plateau in about 4 days. It was observed that, for 4P5J – 1, the hybridization of pineapple increased the water absorption significantly (increase of 24.88%). For all the other composite, the hybridization was effective in reducing water absorption to about 11%, 15.2%, 33.2% for 4P5J – 2, 4P5J – 3, 4P5J – 4 respectively. Thus, we believe that hybridization with pineapple leaf fibres could alleviate the water absorption problem for jute fibre reinforced composites.

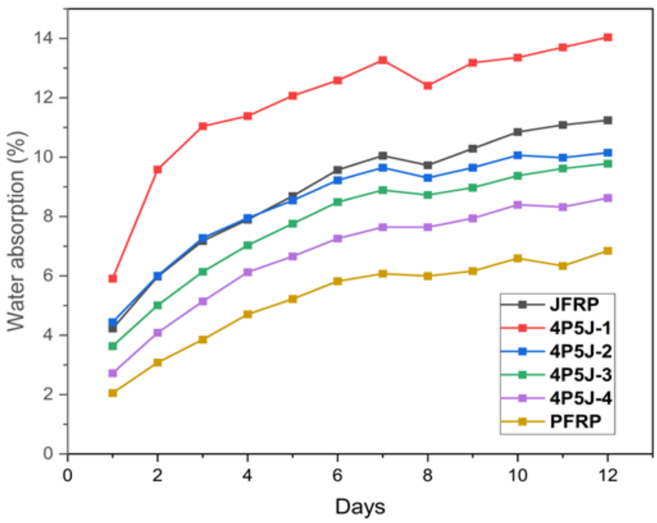


Figure 26: Water absorption (in %) w.r.t time (in days)

4.5 Analysis of SEM micrography

The microstructure of jute reinforced polymer composites, pineapple-jute reinforced hybrid composites, and pineapple and jute yarns were observed by taking images using a scanning electron microscope (SEM). These images are taken after tensile fracture and were then analysed to better understand the causes of failure and strength reduction.

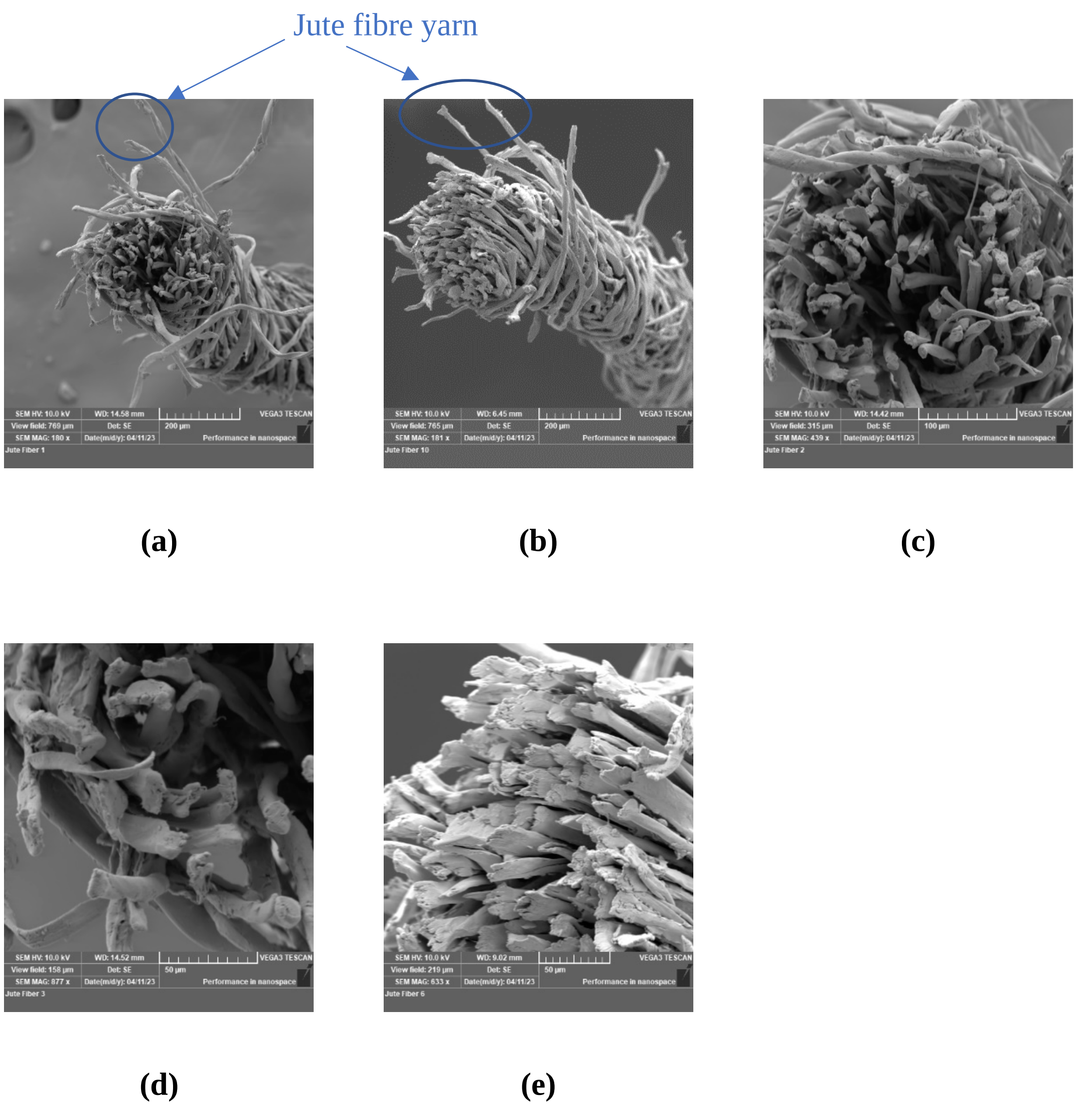


Figure 27: Scanning electron micrographs of jute fibre yarn in different magnifications and positions at (a) & (b) 200 µm magnification; (c) 100 µm magnification; (d) & (e) 50 µm magnification

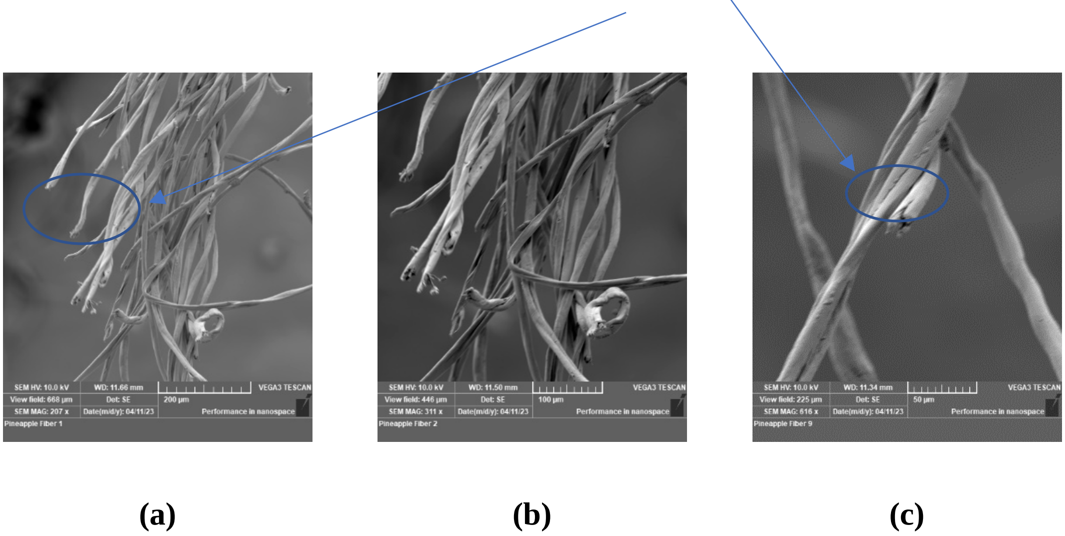
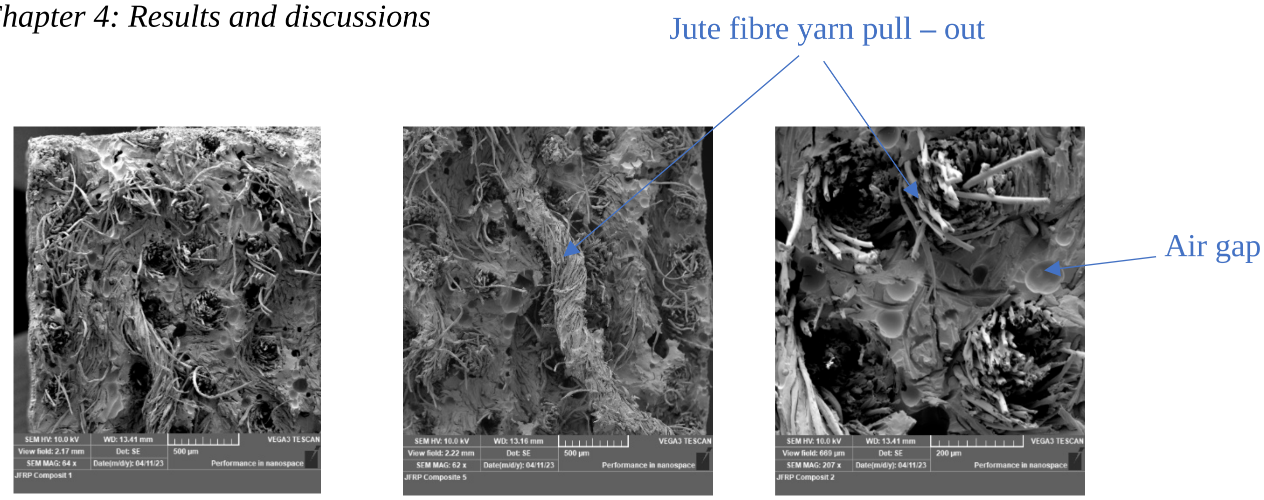


Figure 28: Scanning electron micrographs of PALF fibre yarn in different magnifications and positions at (a) & (b) 200 µm magnification; (c) 100 µm magnification; (d) & (e) 50 µm magnification

The different magnifications of the images help us to understand the construction of the materials clearer. Specifically, in Figure 26(a) with magnifications of 200 µm and 100 µm, the orientation of fibres within a jute yarn becomes readily apparent. Furthermore, in Figure 26(d), one is able to discern to some extent both the lumen and cell wall of jute fibres. In Figure 27(a – c), the fibres comprising the pineapple yarn are depicted, and in Figure 27(c), at a magnification of 50 µm, the cross-section of said fibres is revealed. Overall, the architecture and configuration of the fibres within both jute and pineapple yarns lend insight into the relative strength of the former in comparison to the latter.



(a) **(b)** **(c)**

(d)

Figure 29: Scanning electron micrographs of jute fibre reinforced composite in different magnifications and positions at (a) & (b) 500 µm magnification; (c) 200 µm magnification; (d) 50 µm magnification

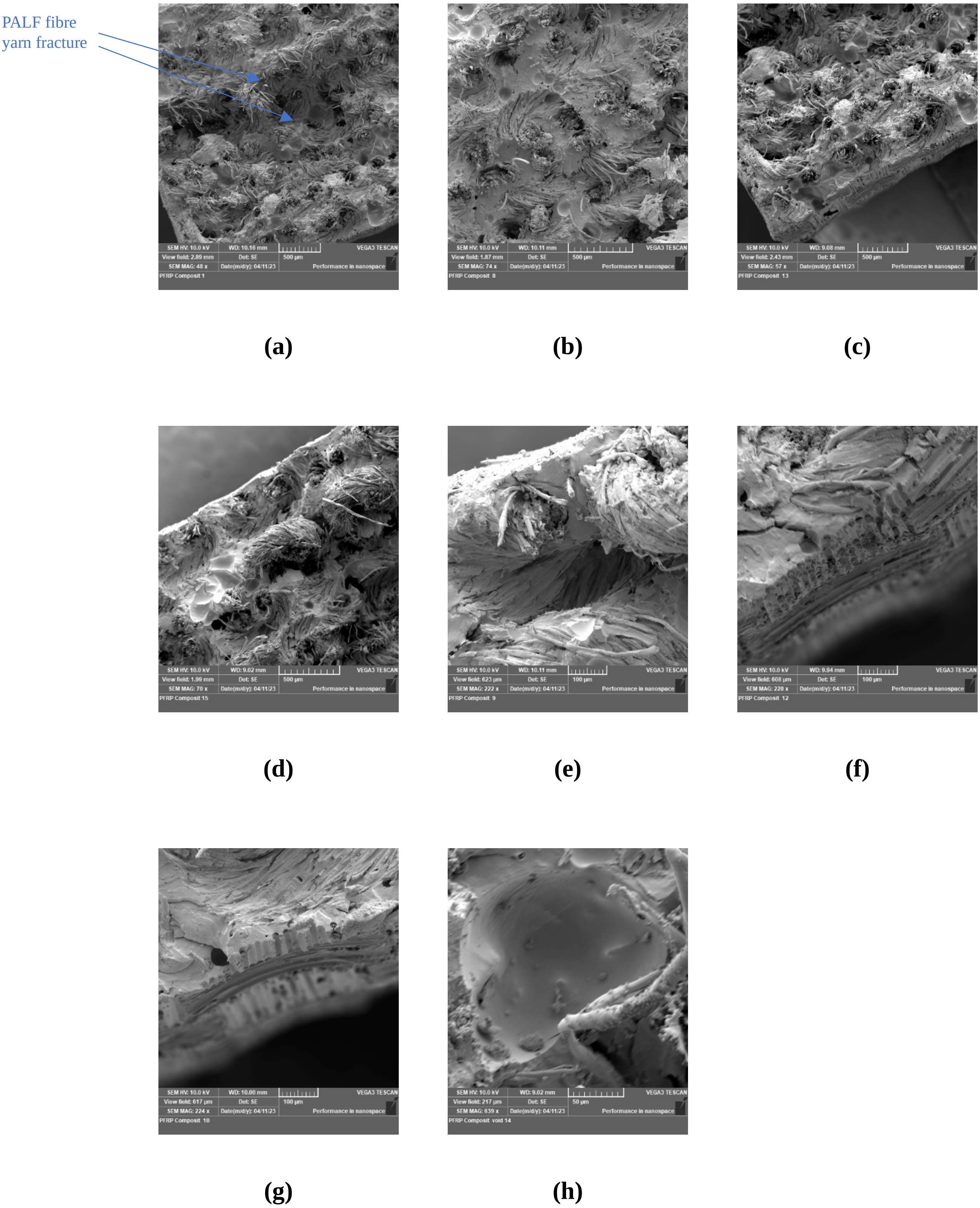
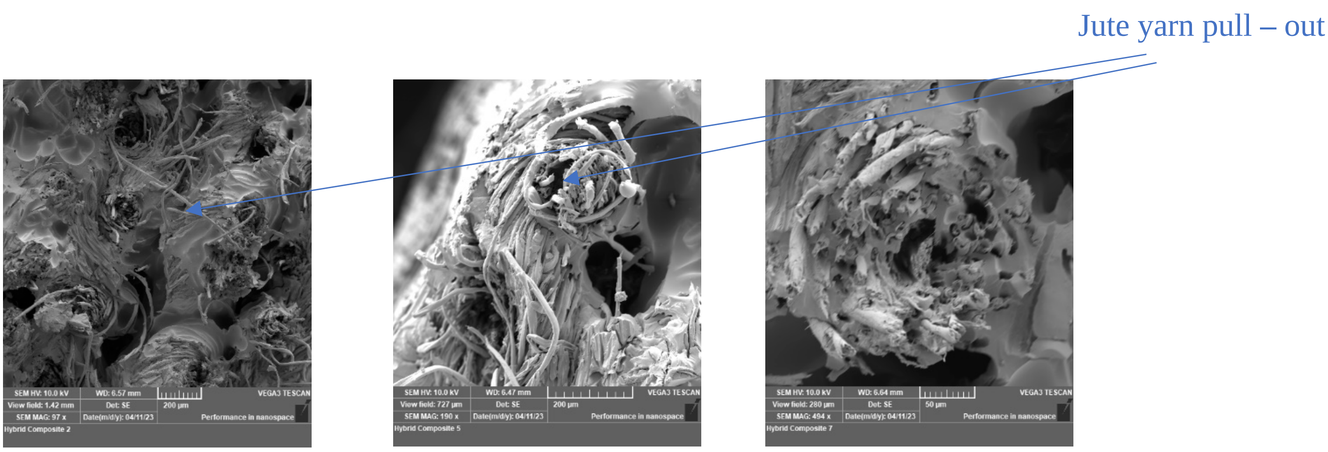


Figure 30: Scanning electron micrographs of jute fibre reinforced composite in different magnifications and positions at (a – d) 500 µm magnification; (e – h) 100 µm magnification; (h) 50 µm magnification

The SEM images provided reveal the evident fibre dispersion and distribution within the composite materials. Specifically, the jute-reinforced composites exhibit a stronger fibre orientation and distribution compared to the pineapple-reinforced composites. Air gaps depicted in the images (Figure 28(c) and Figure 29(h)) lead to reduced composite strength. In Figure 28 (c) and Figure 29(a), extensive fibre pull – out on the tension side of the fracture is visible, indicating poor fibre-matrix adhesion resulting in a considerable reduction in composite strength. Additionally, the figures indicate fibre agglomerations and fibre pull-out, which are the collective stacking of fibres in the matrix and non-uniform stress transfer, respectively, both contributing to reduced strength. Thus, the fibre-matrix adhesion, dispersion and orientation of fibres, fibre agglomeration, and presence of air voids are all influential factors leading to strength reduction in fibre-reinforced composites.



(a) **(b)** **(c)**

Figure 31: Scanning electron micrographs of hybrid composite in different magnifications and positions at (a) & (b) 200 µm magnification; (c) 50 µm magnification

The pineapple-jute fibre reinforced epoxy composite, as a hybrid composite, exhibits superior fibre orientation and distribution, leading to higher strength when compared to PFRPs. Nonetheless, its strength is still less than that of JFRP, as demonstrated by the results of the tensile analysis.

4.6 Comprehensive mechanical and water absorption properties of the composite

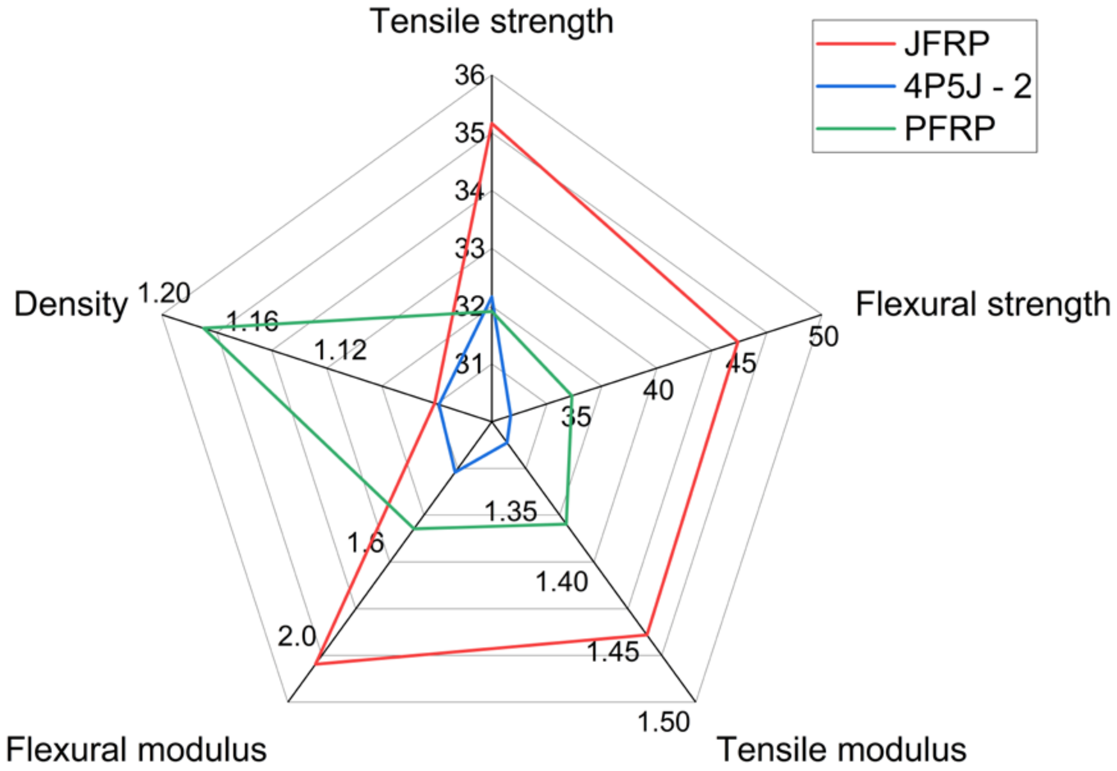


Figure 32: Comparative radar plots of key mechanical properties: Tensile strength (MPa); Flexural strength (MPa); Tensile modulus (GPa); Flexural modulus (GPa); Density (g cm^{-3})

Using the comparative plot shown in Figure 31 which shows the tensile and flexural strength, tensile and flexural modulus, and the density of JFRP, PFRP and 4P5J – 2, we can see that JFRP showed the highest mechanical properties in all the parameters except for density, where the density of PFRP is dominant. The hybrid composite showed a value between JFRP and PFRP in all parameters except for density which was slightly lower than JFRP. This particular set of mechanical properties could be ideally suited for automotive applications such as brake, accelerator and clutch pedal as well as mounting bracket (Sapuan & Abdalla, n.d.). Moreover, with its low density and cost (around $100 per composite), further improvement in the other parameters shown in Figure 31 can lead to its usage in other automotive applications such as dashboards and bonnets, and in aerospace applications where low strength is not an issue such as interiors of airplanes. In the future, in order to keep the green credentials, rather than hybridizing with synthetic composites, surface treatments can be done on both the reinforcements to ensure proper interfacial strength, which can be assumed as the biggest limitation in our experiment. Hybridization with stronger fabrics such as kenaf, ramie and flax can also be explored and applications in bioengineering such as bone tissue scaffolding can be a potential field if the matrix used was biodegradable.

Chapter 5

Conclusion

With the target to achieve greater sustainability, pineapple-jute reinforced epoxy composites were fabricated in six different stacking sequence in order to get a comprehensive evaluation of the mechanical properties and water absorption characteristics of such hybrid-laminate composites compared to jute-reinforced composites and pineapple-reinforced composite.

This study indicated subtle differences in the fiber structures of the composites with no strong evidence of chemical interaction between the fibers and composites. The natural – fibre reinforced hybrid composite in a 1:1 stacking sequence of jute and PALF showed similar flexural strength and modulus values to that of natural – fibre reinforced pure jute composite at 3 – point bend conditions.

Overall, all the composites showed better mechanical properties compared to the pure PFRP, but were not as strong as pure JFRP. But all composites showed improvement in tensile strengths which were higher than the flexural strengths. Among the hybrid composites, 4P5J – 1 showed the best results with good tensile and flexural strength that were as good as JFRP. In terms of impact strength, the composites can be ranked as PFRP > 4P5J -4 > 4P5J – 3 > 4P5J – 1 > 4P5J – 2 > JFRP. Among the composites, improved properties in the composites could be related to its higher fiber strength and better interfacial bonding as evidenced by the SEM images of the fractured surface. The results of the study show that the stacking sequence has a crucial impact on the mechanical properties of hybrid composites. The natural – fibre reinforced hybrid composite in a 1:1 stacking sequence of jute and PALF showed similar flexural strength and modulus values to that of natural – fibre reinforced pure jute composite at 3 – point bend conditions which shows significant improvements in the composite's characteristics compared to the other stacking sequences. Water absorption results showed that hybridization can decrease the water absorption, with the highest reduction being about 33.2% (8.6233% for hybrid 4P5J – 4). This finding highlights the critical importance of selecting the appropriate stacking sequence to achieve the optimal results in composite fabrication. The characterization of the hybrid confirmed that the hybrid composite made of PALF and jute reinforcement can be used to make brake, accelerator and clutch pedals in automobiles.

Thus, natural fibers can be chosen to produce composite materials based on the desired application requirements. In the future, further research can be conducted on the effect of aging (physical, thermal, and mechanical) on the mechanical properties of the fiber-reinforced composites.

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