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Processing and properties of jute (*Corchorus olitorius* L.) fibres and their sustainable composite materials: a review

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Materials that are non-toxic and environmentally friendly are in demand due to the lack of resources and increasing contamination of the environment. In this case, fibre-reinforced composites (PFRCs) have become alternatives to synthetic fibre-reinforced composites (SFRCs) in many applications. Among the PFRCs, jute fibre-reinforced composites (JFRCs) have attracted attention from researchers and have been industrialized in many fields. This review focuses on the research prospects and challenges in jute plant cultivation, harvesting techniques, structure and properties of jute fibres, the effect of surface modification, fabrication methods, and properties of JFRCs. Furthermore, the structure–property relationships, finite element analysis (FEA), machining characteristics and life-cycle assessment (LCA) of JFRCs are discussed in detail. Also, a summary of the innovations in the field of JFRCs is provided. Finally, considering the promising future of this bio-material, several open questions and ideas for its transformation are discussed. According to the findings from the literature, it can be concluded that jute fibres are potential alternative reinforcing constituents to synthetic fibres.

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1. Introduction

The use of composite materials has increased drastically in the last few decades.^{1,2} These materials are heterogeneous in nature and formed by the combination of reinforcing constituents such as fillers, particulates, and fibres with matrices.^{3,4} Furthermore, they have the ability to replace traditional materials such as metals and alloys by providing high strength and stiffness.⁵ Presently, the focus on protecting the environment has sparked interest in the research and fabrication of sustainable and bio-degradable materials.^{6–19} Accordingly, thin films, plastics and adhesives have been replaced by lightweight, convenient and attractive materials in diverse applications.^{20,21} Specifically, it is necessary to reduce the use of debris-creating pollution to maintain a safe and green environment.^{22–24} Extensive studies have proven the potential of natural fibres to replace synthetic fibres as reinforcement materials in composites,^{25–30} where several experimental trials have performed to replace synthetic fibres with natural fibres. Synthetic fibres, together with plant fibres such as flax, jute, sisal, hemp, ramie, and kenaf have been reported to show a good reinforcing effect.^{31–40}

Natural fibre-reinforced composites (NFRCs) have become popular due to their degrading capability.^{41–43} Recent

experimental studies demonstrated the fabrication of composite materials that are entirely biodegradable employing natural reinforcing fibres,^{44,45} where NFRCs have a positive effect on the environment and possess attractive properties.^{26,46–49} Natural fibres of different origins are readily available, allowing researchers to manufacture composites for housing and other applications.^{19,50,51} These fibres are lightweight and possess high stiffness and moderate mechanical and thermal properties, and thus can also be used as reinforcements in composites.^{52–65} Among the natural fibres, cellulose-based plant fibres have emerged as significant materials in recent years.^{66–70} The mechanical properties of plant fibres are dependent on the season of harvesting, area, local climate, rain, soil conditions, part of the plant harvested and plant maturity.^{22,23,71,72} Many research articles have explained the importance of several plant fibres and their composites.^{7,11,13,14,62,71,73–78} In this review, we focus on the processing, properties and applications of jute fibres and their composites. The structure of the review is presented in Scheme 1.

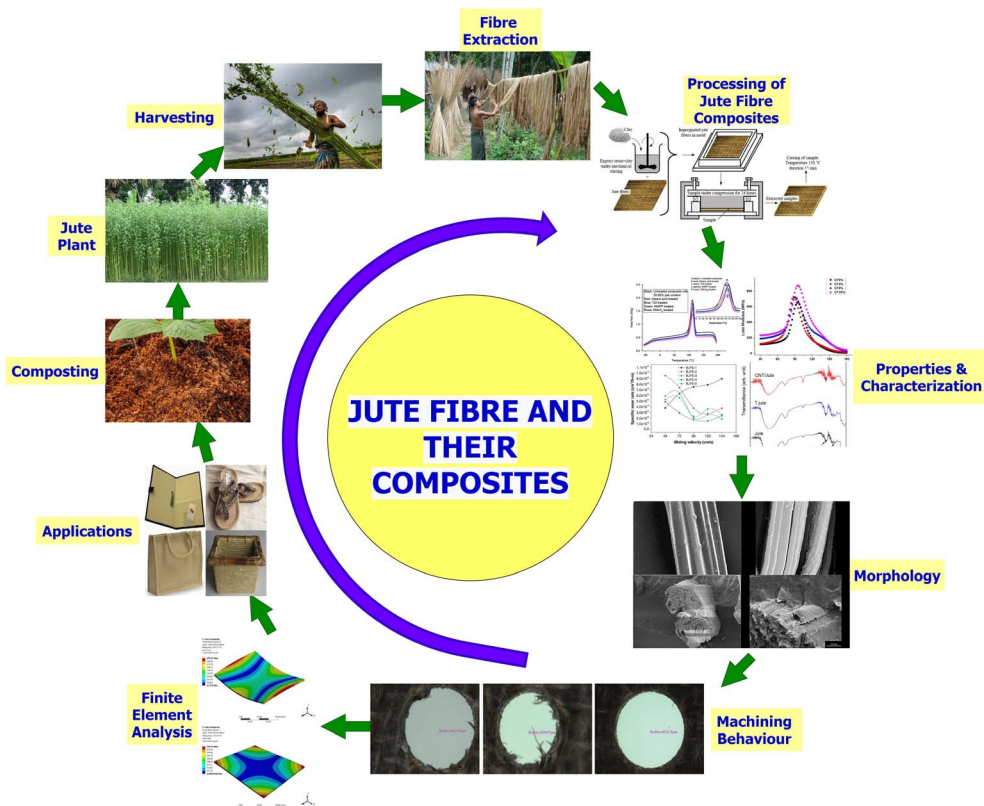
2. Background

2.1 Plant fibres

Plant fibres are beneficial raw materials for several industrial applications.^{78–80} They have numerous key advantages, including light-weight, high resistance, bio-degradability, CO₂ neutralization, renewability, recyclability, moderate mechanical and physical strength, non-toxicity, strong degree of curvature

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Scheme 1 Structure of this review.

without fracture, good insulation properties, durability, low wear during machining, good aspect ratio, acceptable modulus, increased vibration damping and easy surface modification.^{7,9,13,48,50,64,81–123} The fibres derived from the outer stem of plants possess very strong mechanical properties.^{124–127} However, the swelling of the fibres leads to micro-cracking, and ultimately a decrease in the mechanical strength of the composites.^{109,126,128} Many possible benefits of the use of fibres may include the global availability of diverse plant fibres, creation of employment opportunities in agriculture, improvement of the non-edible farm economy and low energy utilization.^{129–131} A description of plant fibres is presented in Table 1.

Among the plant fibres, jute fibres appear to have great potential as a reinforcement material due to their high strength and rigidity.^{146–152} Furthermore, jute fibres can be used effectively in the production of composites given that they possess attractive physical and mechanical properties.^{153–161} Their characteristics significantly depend on the geographical region, surrounding climate and processing methods.^{156,162} However, they also undoubtedly have bottlenecks as a reinforcement in polymer matrices.^{25,52,56,163,164} In this case, jute fibres can be surface treated to enhance their properties, where chemical modification has been proven to be a favorable method for surface treatment.^{14,165–173} Several experiments have been conducted for the analysis of characteristics of jute produced in different ways.^{174–182}

2.2 Plant fibre-reinforced composites

Recently, the use of PFRCs instead of SFRCs has been increasing due to their economic and environmental advantages in numerous practical applications.^{183–185} Recent studies have indicated the substantial progress in the application of renewable materials with enhanced support for global sustainability.^{11,25,82,90,186–188} Plant-based agro-waste and particulates originate from different parts of plants, which can be used as reinforcements in the development of bio-based materials.¹⁸⁹ PFRCs have been applied in several fields and widely accepted by scientists because of their superior performances.¹⁹⁰ Plant fibres have high/moderate thermal resistance and thermal stability when recycled, but they have issues when combined with pure bio-degradable polymers due to their low resistance and transition temperature.^{153,191–193} Fibres from bamboo, banana, abaca, roselle, sugar-cane bagasse, kusha grass, cantala, hemp, sisal, coconut, flax, kenaf, milkweed, curaua, coir, hay, cocos, Cordenka and jute can reinforce polymer matrices.^{52,64,110,112,115,194–216} Bird feathers, soybean seeds and oils, wheat straw, starch-based polymers and algae extracts are also used as composite reinforcements.^{26,217–229}

Short-fibre composites have gained significant attention in the last two decades due to their processing advantages, anisotropic properties and rapid dispersion.^{25,58,156,230,231} Consequently, various PFRCs have been studied and their properties investigated.^{57,138,232–247} Many studies have extended their experimental findings related to different forms of plant fibres.^{100,248–251}

Table 1 Brief description of plant fibres

Plant	Species	Annual production (lakh ton)	Major producer	Advantages	Disadvantages	Ref.
Abaca	<i>Musa textilis</i>	0.70	Philippines, Ecuador and Costa Rica	Strong, hard and high tensile strength	Low production	11, 132 and 133
Alfa	<i>Stipa tenacissima</i>	—	Southern Spain, North Africa	Good modulus	Low strength and elongation	132 and 134
Bamboo	<i>Bambuseae</i>	300	Asia	High tensile strength	Low tensile modulus	11, 132, 133 and 135
Banana	<i>Musa acuminata</i>	—	India, Australia and South Africa	Good tensile strength	High moisture absorption	132
Coir	<i>Cocosnucifera</i>	1.00	India and Sri Lanka	Low density, high elongation and low thermal conductivity	Low strength and modulus	11, 132, 133 and 136
Cotton	<i>Gossypium sp.</i>	250.00	India, Pakistan, China and USA	High production	Low mechanical strength	11, 132, 133 and 137
Curaua	—	—	Brazil	Good elongation at break and tensile strength, and low density	High moisture absorption	25 and ¹³²
Date palm	<i>Phoenix dactylifera</i>	—	India, Pakistan, United state, Middle East, Northern Africa and Canary Islands	Good elongation	Low modulus and strength	132
Flax	<i>Linum usitatissimum</i>	8.30	France, China, Belgium, Ukraine, Canada, United state and India	High specific mechanical properties	Diverse properties due to environmental effects	11, 132, 133, 138 and 139
Hemp	<i>Cannabis sativa</i>	2.14	China and Central Europe	High growth rate, doesn't need of herbicides, pesticides and fertilizers	low elongation at break	11, 132, 133, 139 and 140
Henequen	<i>Agave fourcroydes</i>	0.30	Mexico, Asia and South America	Good toughness and resiliency	Low modulus and elongation at break	11, 132 and 133
Jute	<i>Corchorus capsularis</i>	23.00	India, Bangladesh, Nepal, China, Indonesia, Thailand and Brazil	Fine texture, heat and fire resistance properties	Low elongation at break due to high lignin content, high moisture absorption and low tensile strength	11, 46, 132, 133, 141 and 142
Kenaf	<i>Hibiscus cannabinus</i>	9.70	India, China and Thailand	High growth rate (3 m in months)	Brittle and difficult to process	11, 23, 132, 133 and 139
Nettle	<i>Urtica dioica</i>	—	Central Europe and UK	Good tensile strength and modulus	Slow growth rate	143
Oil palm	<i>Elaeis guineensis</i>	—	West Africa and Congo Basin	High diameter	Low tensile strength	11, 132 and 133
Pineapple fibre (PALF)	<i>Ananus comosus</i>	—	Brazil and Philippines	Mechanical properties comparable to jute fibre	Low microfibrillar angle	132
Ramie	<i>Boehmeria nivea</i>	1.00	China and Eastern Asia	Strong natural fibre and high specific tensile strength and modulus	Stiff, brittle, and low elongation	11, 132, 133, 138 and 139
Roselle	<i>Hibiscus sabdariffa</i>	—	China, Sudan, Thailand, West Africa and India	Good elongation	Slow growth rate	132
Sisal	<i>Agave sisalana</i>	3.78	China, Brazil, Kenya and Tanzania	Good strength and durability	Low tensile strength, modulus and toughness	11, 132, 136 and 144
Sugarcane bagasse	—	750	India	Low growth time and high production	Low strength and modulus	11, 136 and 145

A few research groups have devoted their efforts to determining how different factors influence the characteristics of PFRCs.^{252–256} The curiosity of materials investigators has led to growing

interest in the review of PFRCs.^{13,26,71,74,75,77,78,90,140,186,257–259} Predominantly fibres from bast and leaf, and their reinforcements studied and compared to other fibres.^{23,54,100,138,260–277}

3. Jute plants

3.1 History

Jute is among the most essential natural fibres, which is grown extensively in China, Thailand, UK, and Vietnam besides India and Bangladesh.^{278,279} Jute was discovered in Dunhuang, Gansu Province, China around BC-221AD. It is believed that jute was produced during the Western Han Dynasty.^{280–282} Historical records have shown the use of jute fibre products primarily in India during the Mughal Empire (1542–1605). Indian jute was merchandized by the British East India Company. During the 1800s, the jute fibre-processing industry was established in Dundee.^{283,284}

3.2 Cultivation and harvesting

Jute, a crop cultivated in the rainy season, grows faster in humid and warm climates. This plant, which grows in alluvial, loam, sandy soils, and in places with a temperature above normal and more than 1000 mm of annual rainfall, can also survive in

extreme water flooding. After planting, jute takes about 90–120 days to mature, and harvesting occurs amid blooms and blossoms, leading to the development of seeds. Once the plants are around 6 feet in height, they are harvested and graded according to colour, intensity and length.²⁸⁵ Fig. 1(a and b) show jute plants. An ideal temperature range for good yields is 22 °C to 38 °C with 75% humidity. Throughout the season, jute plants are axed and preserved in water/dew retting for the separation process, where the fibres are separated from the outer stem of the plant. The stalks are tied into bundles and soaked in water for a few days. This process softens the outer cell wall and breaks the bonding between the outer and the inner fibre stick between hard pectin and it allows the separation of the fibres.²⁸⁵ Jute plant harvesting is described in Fig. 1(c–f).

3.3 Fibre extraction

Jute fibres are abundant, and hence considered to be a prospective source of reinforcements for composites.²⁸⁶ They are extracted from the stem of the jute plant through a combination of processes involving various steps such as cutting, retting, shredding, and drying.²⁵⁰ In most cases, jute fibres are extracted through the retting process. The stems are tied into small bundles and immersed in water for around 28 days, and during this time the outer bark is fully decomposed and fibres are exposed.¹⁹⁷ The different types of retting include mechanical retting, water retting, chemical retting, microbial process and dew retting. The choice of retting process is based on the quality of water and cost. The water retting process is the traditional and common method for separating bast/stem fibres, where water is sprayed after the fibre is loosened, and then it is squeezed for dehydration. Subsequently, the separated fibres are washed with clean tap water and dried under sunlight. The obtained fibres are around 3–6 feet long and white or brown in colour, which are tied into bales for delivery to end-users. Upon drying, the bales are sent to jute mills to be processed into jute yarn and hessian.² The extraction of jute fibres by the retting method is shown in Fig. 1(g–j).²⁸⁵ Table 2 presents an overview of the fibre extraction methods, comparing various retting processes and mechanical extraction.

3.4 Jute fibres

Jute is a lignocellulosic fibre, having a low density, high strength and abundant availability. Consequently, researchers have shown interest in applying this fibre as a reinforcement in composites.^{290–292} Jute is the most flexible fibre with immense potential for the manufacture of composites due to its safety, strong, non-abrasive nature, viscoelasticity, biodegradability, combustibility, compostability, and good acoustic and thermal insulation.^{293–295}

3.5 Fibre structure and composition

3.5.1 Structure. Plant fibres possess a complex microstructure consisting of hundreds of micro-cells with a typical length of 2–5 mm and a nominal diameter of 0.2 mm, arranged with lignin and natural adhesive.^{296,297} Basically, plant fibres are multi-cellular in nature, possessing a variety of continuous cells



Fig. 1 (a and b) Dry and rainy season jute plants, respectively. (c–f) Harvesting of jute plants and (g–j) extraction of fibres from jute plants (<https://www.jute.org>).

Table 2 Overview of fibre extraction process^{126,192,287–289}

Extraction Process	Retting		
	Dew retting process	Water retting process	Mechanical method
Procedure	Stems are spread on grassy fields for reaction with bacteria, sunlight, and mist under normal air flow	Plant stems are submerged in water (rivers, ponds, or tanks) and checked periodically (microbial retting)	Fibres are separated using a hammer or/and decorticator
Days	14–21 days	7–14 days	Depending on production of fibres
Benefits	This process is applicable in heavy dew and water-scarce areas	The fibres produced are uniform and of higher quality	Produces large quantities of fibres in the minimum time
Disadvantages	The separated fibres are very dark in colour with low strength For this process, the cultivating lands are occupied and the fibres are contaminated due to fungi	Comparatively high processing cost, environmental concerns and low strength, but better than fibres produced through dew retting process	High cost and acceptable quality fibres

of varying sizes, shapes, and fibre alignments. The central regions of many plant fibres may have a cavity, which is called a lacuna, surrounded by several lumens in their cellular structure. The orientation angle of jute fibre is lower than that of other plant fibres, endowing it with high strength but making it brittle in nature.²⁹⁸ The structural characteristics and high water absorption nature of plant fibres lead to inadequate bonding between fibres and matrices, resulting in fibre pullout during use.^{299–301} Microfibrils of jute fibre are bound with other plant fibres of identical structure.¹⁰⁵ Jute is multi-celled in structure, as shown in Fig. 2(a),²⁶ where its fibre cell wall consists of three layers with primary and secondary walls. Cellulose has the highest concentration, while lignin is the major portion of the cellulose-free middle lamella. Usually, the secondary layer is the thickest and governs the strength and characteristics of the fibres. The long cellulose chains are intertwined in bunches called microfibrils.²⁶ Fig. 2(b)⁶¹ shows the changes in the chemical structure of jute fibre upon alkaline treatment and the reaction occurring during its processing is presented in Fig. 2(c). Due to the presence of hemicelluloses, lignin, pectin and wax, the surface of the alkaline-treated fibres becomes more polar and rougher.

3.5.2 Composition. Jute fibres contain large sub-categories of substances including cellulose, hemicelluloses, lignin, pectin, polyuronide, acetyl matter, fat, ash, nitrogen (N₂) and some other components such as aqueous extracts and mineral substances.^{302–304} Jute fibres composed of lignocelluloses have been observed to possess strongly polarized hydroxyl groups.^{58,305,306} Details of the composition of the jute fibres are presented in Table 3. The bonding between the cellulose chains ensures their stability and solubility in water and chemical solutions.³⁰⁷ They exist as micro-fibrils in the plant cell-wall and their diameter varies in the range of 3–35 nm.³⁰⁸ The hydroxyl cellulose groups create fibrils by forming hydrogen bonds adjacent to and partially crystallizing other molecular cellulose chains.³⁰⁴ Hemicellulose is the second most important constituent, accounting for 15–30% of the cell-wall.³⁰⁹ Hemicelluloses are bound polysaccharides and closely connected with the cellulose microfibrils. The four major hemicellulose forms

based on their structure are xylans, mannans, xyloglucans and glucomannan.^{291,310} The bonding of hemicellulose is very strong with cellulose fibrils presumably by hydrogen bonds, which is semi-soluble in water. Lignin is a highly complex amorphous, aromatic bio-polymer and has the least water absorption of the components of plant fibres. It is a naturally developed heterogeneous and irregular cross-linked polyphenol consisting of units of phenylpropane. These three elements are responsible for physical properties of the fibres.^{310–312} Another significant characteristic is that lignin is thermoplastic (*i.e.*, it begins to soften at 90 °C and begins to melt at 170 °C).²⁹¹

3.6 Properties of jute fibres

Among the plant fibres, jute stands out due its unique properties,³²³ which are strongly influenced by factors such as chemical composition, fibre structure, microfibrillar angle and cell-wall thickness.^{78,246} Variations are seen in these characteristics among plants, leading to significant variations in the properties of the fibres.^{26,324} The main factor determining the final properties of the fibres is their anatomical origin, given that fibres of different origins typically have different properties. However, factors such as age, growing trends, environmental conditions, and extraction techniques should be considered to affect the properties of the fibres.³²⁵ Jute is predominantly polar, owing to the availability of various polar groups on its backbone.³²⁶ Also, its tensile strength is in the range of 250–300 MPa, which is around seven-times lower than that of steel, but it has relatively high resistance.³²⁷

Research on the measurement of the tensile strength of jute fibres in dry and alkaline environments was carried out and it was found to be very high and increased when submerged in an alkali-solution ranging from 5–32%.³²⁸ Many sources have reported that the Young's modulus of jute fibre varies from 20–40 GPa, which is substantially lower than that of glass fibre of about 73 GPa. Discrepancies may occur due to circular cross-section assumptions, in addition to the inherent mechanical variability of natural resins.^{329–333} The decomposition of the constituents of jute begins with hemicellulose at 290 °C, then

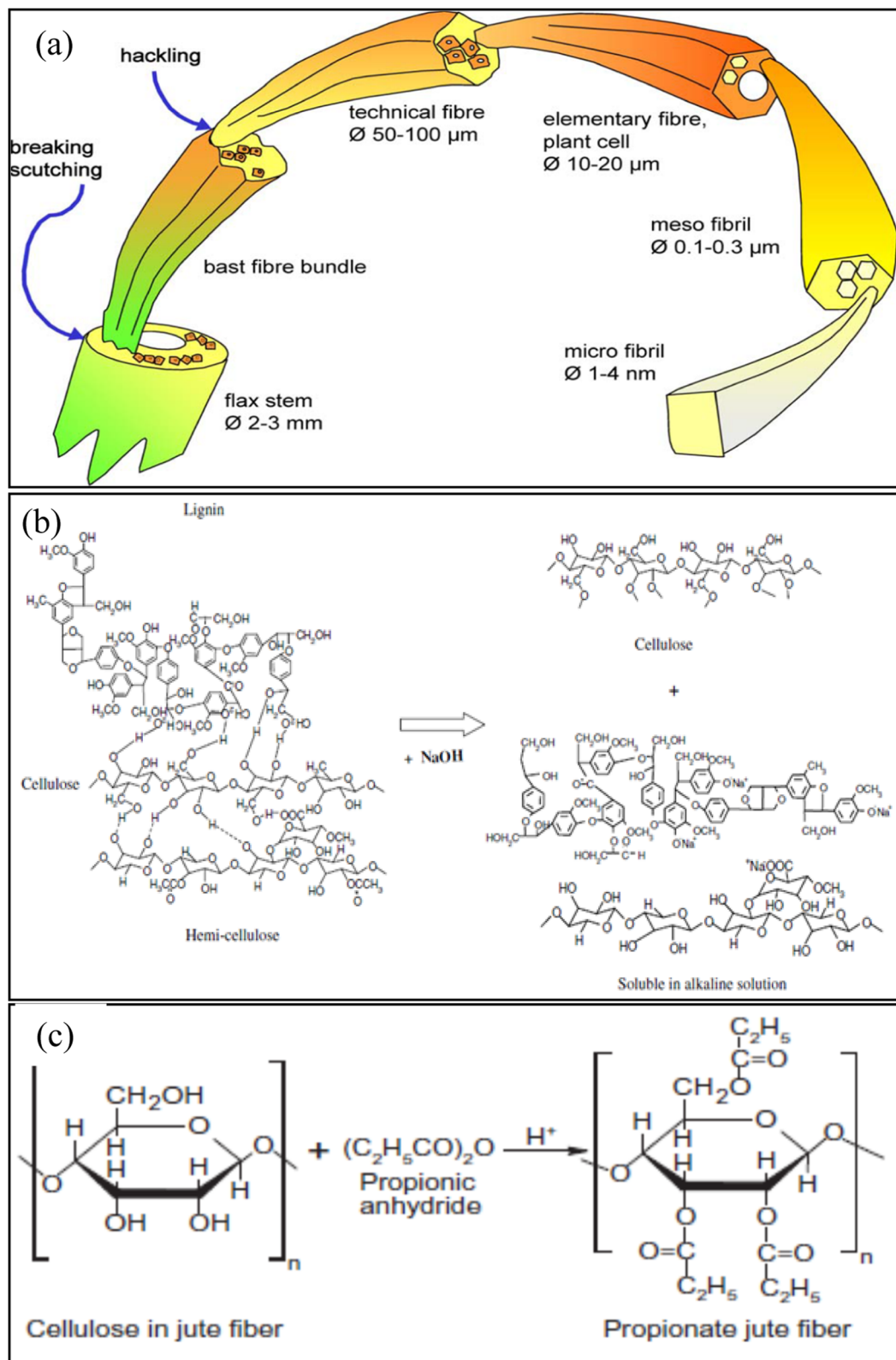


Fig. 2 (a) Structure of a jute fibre. Reproduced from ref. 26 with permission from Elsevier, 2006. (b) Change in its structure by alkaline treatment. Reproduced from ref. 61 with permission from Elsevier, 2008. (c) Reaction of jute fibre with propionic anhydride.

cellulose at 332 °C, and finally the lignin at 447 °C. Therefore, the processing of jute fibres was carried out in most experimental studies by compression or injection moulding process

below 200 °C.^{334,335} The physical and chemical characteristics of jute fibres are presented in Tables 4 and 5 lists the main suppliers of jute fibres.

Table 3 Strength and composition of jute fibres

Density (g cm ⁻³)	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation (%)	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ref.
1.4	400–800	10–30	1.8	50–57	—	8–10	153
—	256 ± 1.02	31 ± 1.34	0.78 ± 0.05	—	—	—	348
—	—	—	—	58–63	20–24	12–15	302
1.48	410–780	18–50	1.9	—	15–20	11–15	314 and 315
1.3	393–773	26.5	1.5–1.8	—	—	—	1
1.4	420–820	10–30	1.8	40–60	—	—	316
1.3–1.5	610–780	12–60	1.0–1.9	59–70	15–20	11–15	317
1.3–1.4	393–773	13–26.5	1.2–1.5	61–71.5	13.6–20.4	12–13	318
—	—	—	—	74.4	15	8.4	319
1.3–1.5	400–800	70	—	59–61	22.1	15.9	320
1.3	450–550	26–32	1.5–1.8	58–63	12	12–14	321
—	—	—	—	61–71.5	17.9–22.4	11.8–13	322
—	—	—	—	73.2	13.6	13.4	310

4. Fibre surface modification

Perfect reinforcement and good interfacial bonding must be ensured to obtain durable composites for industrial applications, enabling the maximum use of the fibre reinforcement capacity.^{230,394,395} The strength of plant-based fibres can be enhanced through surface modification by the appropriate methods.^{14,165,168,169,352,396–398} However, plant fibres have some disadvantages, including hydrophilic behavior, poor thermal stability, and in particular poor adhesion with a hydrophobic resin, which results in poor interfacial bonding and low mechanical strengths.^{235,399} In this case, surface modification^{173,278,303,400–406} has the ability to solve most of these drawbacks. The limitations of PFRCs can be strengthened through the use of chemical or physical methods, treatment of fibres, or use of coupling agents.^{37,38,407–409} Slight leaching of the fibres leads to the degradation of hemicellulose, lignin and

pectin, weakening their internal structure and causing the fibres to separate. A cleaner and rougher surface is beneficial for fibre/matrix bonding, which promotes mechanical inter-locking and bonding due to the exposed hydroxyl groups on the resin.³⁷¹ Also physical and chemical treatments^{38,56,154,163,164,305,375,400,410} have attracted attention from researchers for further investigation due to their many benefits. Consequently, numerous surface treatments, for example, alkaline, acetylene, silane, peroxide and permanganate, benzylation, electrical discharge, and cyano-ethylation treatments, have been developed by various researchers and are discussed in the literature.

4.1 Physical modification

Several physical modification methods can enhance the cleaning of the fibre surface, consequently improving the solid bonding at the interface.⁴¹¹ Many physical treatments such as plasma treatment,^{341,412–415} photo-oxidation by UV irradiation,³⁵

Table 4 Properties of jute fibres

Physical properties		Chemical properties	
Parameter	Value/result	Parameter	Result
Abrasion resistance	Relatively good	Dye ability	Good, easily dyed
Colour	Yellow, brown, golden	Effect of acid	Destroyed by hot concentrated acids. Dilute acids cannot harm the fibre
Cross section	Uneven, thick cell wall with lumen	Effect of alkali	Strong alkali destroys the fibre and reduces its strength
Diameter	18 μm	Effect of bleaching	Not affected by oxidizing and reducing bleaching agents
Dimensional stability	Good	Effect of insects	Resistance is good
Heat resistance	Good	Effect of mildew	Better than cotton and linen
Length (inch)	0.2–30	Electrical conductivity	Moderate
Moisture regain	13.75%	Organic solvent	Resistant to organic solvents
Resiliency	Not very good	Thermal conductivity	Poor
Specific gravity	1.48–1.5	—	—
Specific heat	0.324	—	—
Stretch and elasticity	Not good and 2% elongation at break	—	—
Tenacity (g per den)	3.5–4.5	—	—

Table 5 Major suppliers/vendors of jute fibres

Name of the firm	Country	Ref.
Alam Fibre Impex Ltd	Bangladesh	336–338
Amazon Region	—	296
Anil Ltd	Turkey	339
Babu Bazaar	Dhaka, Bangladesh	340
Bangladesh Jute Mills Corporation	Dhaka, Bangladesh	341
Bangladesh Jute Research Institute (BJRI)	Bangladesh	313 and 342–347
Carol Leigh's Hillcreek Fibre Studio	USA	58 and 61
Chandra Prakash & Co. Pvt. Ltd	Jaipur, India	317, 348 and 349
Composites Evolution	UK	350
Cia Textile Castanhal	Brazil	230
Dharmapuri District	Tamil Nadu, India	153, 314 and 315
Extra Weave Private Ltd	Kerala, India	351 and 352
Fatima-Alyaf Tala-e Jute Industries Ltd	Gazipur, Bangladesh	41
Gloster Jute Mill	Kolkata, India	353–358
HP Johannesson Trading	Sweden	297
Hunan Shangke Co. Ltd	China	359
Indarsen Shamlal Pvt. Ltd	India	83
Indian Jute Industries Research Association (IJIRA)	Kolkata, India	360–362
Janata Jute Mills Ltd	Bangladesh	363 and 364
Kamarhatty Company Limited	India	365
JB Plant Fibres Ltd	UK	366
Kancheepuram District	Tamil Nadu, India	367
Konark Jute Mills	Orissa, India	56
Krishna Jute Industries	India	368
Longtai Weaving Co., Ltd	Changshu, China	369 and 370
Local Market	Vijayawada, India	302
Local Market	Bangladesh	371–374
Local Market	India	375 and 376
Maranda Halli Market	Tamil Nadu, India	316
Meena Fibres	Puduchery, India	377 and 378
Materials Lab of UFSCar	Brazil	379
Muszaki Konfekcio Kft	Szeged, Hungary	182
Open Market	Rajapalayam, Tamil Nadu, India	322
Redbud Textile Tech. Inc.	China	380
Sakthi Fibres Pvt. Ltd	Chennai, India	381
Schilgen GmbH & Co.	Germany	292, 382 and 383
Sisalsul	Brazil	384
Sonajute	Casablanca, Morocco	385
Spinnerij Blancquaert NV	Lokeren, Belgium	59 and 105
Stuart C. Hurlbert & Co.	Framingham, MA	386 and 387
Textiles Divers Algerie	Algeria	388
Tesac Co.	Japan	81
Women Development Organization	Uttarakhand, India	389–392
Yano Seikei	—	393
ZKK, Sdn Bhd	Malaysia	321

ionizing radiation,⁴¹⁶ gamma radiation,⁴¹⁷ corona treatment,^{418–422} cold plasma treatment,⁴²³ ozone treatment,⁴¹² laser treatment,⁴²⁴ boiling under or without pressure,⁴¹¹ and high pressure plasma treatment⁴²⁵ have been used to enhance the compatibility of fibres. Table 6 presents the purpose and effect of various processes of physical treatment.

4.1.1 Plasma treatment. Gases such as oxygen (O₂),⁴¹³ helium (He),⁴¹⁴ helium and nitrogen (N₂),⁴¹⁵ and air⁴²⁶ are employed for the modification of the fibre surface. The impact of plasma on the surface morphology of jute fibres and the structure–property relationships in JFRCs were studied. Jute fibres have been tested at atmospheric pressure for 60 s with dielectric plasma treatment.⁴¹² Other gases as well create an undesirable localized corona discharge rather than standard

treatment. Three gas mixtures were employed in the treatment of jute fibres, as follows: (i) He at the flow rate of 14 L min⁻¹; (ii) He and acetylene at 14 L min⁻¹ and 0.7 L min⁻¹; and (iii) He and N₂ at the same flow rate. The fibres were tied to a roller, which moved the plasma source. A modification was performed for the roller of 5, 25, 50 and 100 revolutions. The fibres were treated at a speed of 0.424 s per rev using the plasma source. The equipment was operated at 970 W and the power supply frequency was 90 kHz.³⁴¹

4.1.2 Ozone treatment. Ozone or oxy-fluorine gas was used in this process for the modification of the jute fibre surface. This treatment was done by placing the fibre in an air-tight jar completely filled with ozone for 1 h. An inter-connected jar with an ozone generator produced ozone gas continuously.

Table 6 Purpose and effect of physical treatment processes

Treatment type	Purpose	Effect	Ref.
Plasma treatment	To achieve an ablation or etching effect, resulting in a better interface with the matrices through mechanical interlocking	A semi-industrial atmospheric pressure glow discharge plasma process in order to treat the fibre surface	341, 412–415 and 426
Ozone treatment	To maintain mechanical strength	Surface modified by placing the fibres in an ozone gas-filled closed container	412
Laser treatment	To increase the structural stability of fibres	Improving the surface roughness by evaporating the materials from fibre surface using a laser beam	424
Corona treatment	To improve the acidity of the fibre surface	Activating the cellulose in the fibre to improve the efficiency of the grafting process	419–422
Hornification treatment	To increase the dimensional stability, which is known as hornification	Surface modified by passing heat waves on the surface	319
UV radiation/ionising radiation	To improve the adhesion, wettability, biodegradability and tribological properties	—	35 and 416

Subsequently, the fibres were exposed to the gas at 20 °C in an open atmosphere by maintaining the flow rate of 50 L h⁻¹ for 9 h.⁴¹²

4.1.3 Laser treatment. Laser irradiation with a commercial CO₂ pulse infra-red laser was performed on the surface of the fibre, producing laser beams. The sample was held in place of a concave–convex lens and a joule meter with the help of a sample holder. The parameters used in the determination of the laser marking intensity included speed, duty cycle, and frequency markings. The speed of the labeling was fixed at 200 bits per ms, service cycle at 50% and 5 kHz frequency. A convex–concave lens was employed to get a clear focus on the laser beam, which allowed the light to be altered in the irradiation area with a size in the range of 0.24–1.20 cm². The beams were connected with the fibres through material evaporation, thermal decomposition and surface roughness modification.⁴²⁴

4.1.4 Corona treatment. This treatment has been reported to increase the bonding resistance of composites. Bataille *et al.*⁴²² performed corona treatment as a type of pretreatment process to create active sites on the cellulose surface in the fibre and increase the effectiveness of its bonding. Research has demonstrated that the contact angle of a urea-formaldehyde droplet on a surface decrease by increasing the concentration of corona treatment.⁴¹⁹ In this case, the improved wettability contributed to enhancing the bond strength. Corona treatment increases surface energy, which is mainly due to the increase in the content of carboxy and hydroxy groups. Belgacem *et al.*⁴²⁰ demonstrated the use of high-alpha cellulose, hard-wood fibre powder as a reinforcement material in polypropylene (PP), substantially improving the yield stress and elastic modulus of the composite. The extent of the transition depends on the treatment conditions, duration and intensity of corona. Dong *et al.*⁴²¹ observed a decline in the property of polyethylene composites loaded with cellulose whether one or both compounds were corona treated.

4.1.5 Hornification treatment. Hornification is the process of stiffening the polymeric structure in the cells of fibres to improve their dimensional stability. It was reported that the fibres were kept in water at 22 °C for 3 h. Drying was performed at 80 °C in a furnace with a built-in electronic temperature monitor to measure the mass loss during the drying cycle. The furnace was programmed to heat to 80 °C at a rate of 1 °C min⁻¹ and hold this temperature for 16 h. Then, it was cooled to 22 °C to prevent possible thermal shock to the fibres.³¹⁹

4.2 Chemical modification

Plant fibres have hydrophobic surfaces and anisotropic internal structures, and therefore it is difficult for them to act as reinforcing materials in polymer matrices. Accordingly, chemical treatments have been developed for the modification of the surface of fibres.^{63,368,427–433} Several researchers reported the chemical treatment of jute fibres to enhance their hydrophilicity and properties of JFRCs.^{154,155,202,361,434–441} A few authors extensively investigated the effects of different forms of chemical treatment on plant fibres, in particular jute fibres.^{263,279,442} To date, various chemical treatments have been reported, using chemicals such as NaOH,^{38,58,61,63,230,302,361,371,382,386,387,441,443–451} silane,^{231,386,452–457} alkali and silane,^{458,459} potassium permanganate (KMnO₄),^{356–358,460} maleic anhydride polypropylene (MAPP),^{58,305,356–358,375,461–466} stearic acid,^{356–358,467} bleaching,⁴⁶⁸ toluene diisocyanate (TDI),^{355–358} sodium silicate,⁴¹⁷ UV radiation monomer grafting,^{383,469,470} maleic anhydride-grafted polypropylene (MAGPP),^{305,462,471,472} detergent washing, dewaxing, acetic acid treatment,⁴⁴³ graft co-polymerization,^{291,473–477} deposition of nano-SiO₂ (ref. 359 and 478) and other chemical treatments.^{102,342,365,378,409,412,434,479–485} Table 7 shows the different chemicals used and the effect of various chemical surface treatment methods.

4.2.1 Alkaline treatment. This is a basic chemical surface modification process for plant fibres when reinforced with

Table 7 Chemicals used and effect of chemical surface treatment processes

Treatment type	Chemical agents	Effects
Alkaline treatment	Sodium hydroxide	Reduces fibre diameter
Mercerization	Sodium hydroxide	Mercerization of fibre-length with pre-evaluated tension or free shrinkage mode
Silane treatment	Silane solution	Strengthening of the interfacial relationship between fibres and matrix
Benzoylation treatment	Benzoyl chloride	Makes fibres hydrophobic
Bleaching/peroxide treatment	Hydrogen peroxide	Improves the adhesion of fibres with the matrix
Graft co-polymerization	Methyl methacrylate, acrylonitrile, acrylamide, ethyl acrylate, and styrene	Reduces absorption of moisture by fibres
Sodium chlorite treatment	Sodium chlorite	Removes moisture from fibres
Acrylation and acrylonitrile grafting	Acrylic acid	Bonding capacity and stress transfer of the interface increase
Esterification	Acetic anhydride, dodecanoic acid, hexanoic acid, oleoyl chloride, octadecanoic acid, and docosanoic acid	Improves fibre/matrix bonding
Permanganate treatment	Potassium permanganate	Improves wettability and thermal stability of fibres
Enzymatic treatment	Pectinase, laccase, cellulase and xylanase	Enhances the linking/meshing of fibers with the matrix
Stearic acid treatment	Stearic acid	Eliminates the extra-particles from the fibre surface

polymer matrix.^{63,157} The addition of NaOH to plant fibres facilitates the ionization of the hydroxyl group.^{157,400} In one study, NaOH treatment reduced the fibre diameter by 13.7% and weight by 21%.³⁰² According to Arao *et al.*,⁸¹ alkali-treated JFRCs showed improved properties. NaOH treatment resulted in the removal of excess non-cellulosic materials, and thus weakened the fibre, reducing the strength of the composite.⁴⁸⁶ The influence of NaOH treatment on the properties of laminated JFRCs was investigated by Karabulut *et al.*¹²² The fibres were submerged in NaOH solution for 15 days at 25 °C and the results were analyzed using the Weibull distribution. It was observed that this treatment process had a significant influence on the mechanical properties of the fibres.

In a study, the fibres were soaked in distilled water containing 0.5 wt% alkali solution and held for 2 h at 25 °C, then washed in water, allowed to dry at 27 °C, and then they were placed in a heater for 4 h at 80 °C.⁵⁸ Also, fibres were immersed in 10% NaOH solution for 3 h at room temperature and then heated at 100 °C, where 1 mL of 10% alkali solution was added per 0.15 g of fibre. Then, they were rinsed with water after treatment and left to dry for 48 h.²³⁰ Subsequently, they were washed and dried at 60 °C for one day, pre-treated at 55 °C for 1.5 h with 0.1% H₂SO₄ solution, and then washed again. The washed fibres were subjected to the following processing: immersed in a solution of (i) 2% NaOH, 0.2% Na₂SiO₃ and Na₂P₃O₁₀, as well as 0.15% Na₂SO₃ at 100 °C for one hour, (ii) 5% NaOH at 20 ± 1 °C for 4 h, and (iii) 20 ± 1 °C for 4 h in 1.5% catalyst and 98.5% ethanol solution. Finally, the fibres were rinsed and dried at 60 °C in an oven for one day.^{371,487}

In one study, jute fibres were treated at 30 °C in 5% NaOH solution for 4 and 8 h and rinsed with pure water to eliminate the NaOH clinging to the fibre surface, and then neutralized with diluted hydrochloric/acetic acid. The fibres were dried for 48 h at 30 °C, followed by 6 h drying in an oven at 100 °C.⁴⁴¹

Then, they were immersed for 2 h in 5% NaOH solution, cleaned with water and soaked for 1 h in a 2% acetic acid solution,^{386,387} dewaxed at 50 °C for 2 days, dried at 80 °C for 4 h, and then heated for 30 min at 100 °C. The dewaxed fibres were modified with 1% NaOH for 4 h, the residual alkali removed, rinsed with water again, and dried.³⁸² Finally, the fibres were washed for 1 h with 4% NaOH solution, dried overnight and heated at 105 °C for 60 min after treatment.³⁶¹

4.2.2 Mercerization. Mercerization is applied to strengthen the interfacial relationship between the fibres and the matrix.^{488,489} Comprehensive structural modifications may be caused by fibre mercerization, which is dependent on the treatment conditions such as solution concentration, treatment time, fibre tension, and temperature.⁴⁹⁰⁻⁴⁹² The fibres were soaked at around 25 °C for 2 h in an NaOH solution.⁶¹ This mercerizing system enabled the systematic and continuous mercerization of the fibre length with pre-evaluated tension or mercerization in free shrinkage mode.⁴⁹³

4.2.3 Silane treatment. A silane agent was prepared and stirred for 5 min in methanol solution using a magnetic stirrer. Subsequently, the fibres were soaked in the prepared mixture for 60 min. The treated fibres were rinsed using water and dried for 12 h.³⁸⁶ In another study, the fibres were immersed in a 0.5 wt% silane solution for 1 min using an amino-silane binding solution and kept at 50 °C for 4 h.^{231,452} Also, the solvent was made by dissolving liquid oligomeric siloxane solution at 1 wt% and jute fabrics were alkalized, and later sonicated in an ultrasonic bath for 5 min. Then they were plunged in the oligomeric siloxane for 1 h and the treated jute fabrics were eventually dried for 24 h at 608 °C.³³⁹

4.2.4 MAPP/MAHgPP treatment. MAPP is the most effective coupling agent to enhance the interfacial adhesion between the fibres and matrix.^{99,105,494-508} Ultimately, the capability of MAPP to improve the strength of the composites relies on factors such

as the fibre structure, matrix miscibility, and composite processing conditions.^{162,305,504–509} The mechanical properties of JFRCs increased through the treatment of the fibres with up to 6 wt% MAPP.^{463–466} The mechanical strength increased to 50%, but the energy at the JFRC interface decreased due to the impact loading when the fibres were treated with 0.1% MAPP solution at 100 °C for 5 min.^{305,462} The fibres were washed in toluene solvent at 100 °C for 5 min with 1% MAPP solution, and then rinsed in toluene and dried at 60 °C.³⁷⁵ Park *et al.*⁵⁸ tested the interfacial properties of JFRCs and found that the modification of the fibres could enhance the performance of the composite. During the winding cycle, MAPP and acetone solutions were coated all the fibre layers, which were then left in an oven for 30 min at 60 °C to remove the acetone solution from the fibre surface.^{356–358}

4.2.5 Graft co-polymerization. Some researchers extensively studied the development of vinyl-based polymers and the effects of methyl methacrylate, acrylonitrile, acrylamide, ethyl acrylate, and styrene treatments on jute fibres.^{473–477} Samal *et al.*⁴⁷³ observed an improvement in the absorption of moisture by the fibres on co-polymerization with styrene and methacrylate. Also, acrylonitrile, styrene or vinyl acetate graft co-polymerization on jute enhanced the condensation and thus improved the moisture sensitivity of the fibres.⁴⁷⁴ Mohanty *et al.*⁴⁷⁵ reported that the co-polymerization of acrylonitrile on jute fibres enhanced the thermal stability of the resulting composites. X-rays were used in the study of the co-polymerization of vinyl monomers on jute graft.⁴⁷⁶ Tripathy *et al.*⁴⁷⁷ reported the co-polymerization of methyl methacrylate on chemically modified fibres using the redox method of KMnO_4 malonic acid. They successfully enhanced the inter-laminar strength by decreasing the amount of resin, filling higher fibre levels and decreasing the absorption of moisture in the composite with the use of these modifications. Then, the fibres were co-polymerized using different chemicals, resulting in a 10–30% increase in strength and modulus. Polymethylmethacrylate synthesis is also successful in this regard, albeit to a lesser degree.²⁹¹

4.2.6 Esterification. Anhydride reaction of fibres is known as esterification. This modification involves alkali-treatment accompanied by esterification.⁵¹⁰ Several chemicals, for example, acetic-anhydride, dodecanoic-acid, hexanoic-acid, oleoyl-chloride, octadecanoic-acid, and docosanoic-acid, have been employed for enhancing the fibre/matrix bonding during esterification.^{409,479,480} Jute fibres were soaked in 5% NaOH and mixed well at 27 °C for 1 h. The treated fibres were immersed in glacial acetic acid at 27 °C for 1 h. Then, the fibres were submerged again for 5 min in propionic anhydride containing concentrated H_2SO_4 . Finally, the fibres were rinsed with water and dried for 12 h at 80 °C.³⁴⁵

4.2.7 Oxidation. Extracted jute fibres were dried in an air-circulation chamber for 5 h at 105 °C and soaked in a liquor-based sodium solution in the ratio of 1 : 16. Oxidation was performed at 70 °C for 5 h, and then the oxidized fibres were then cooled and isolated. Subsequently, they were washed thoroughly with running tap water. Lastly, the washed oxidized

fibres were dried at atmospheric temperature, and again dried in an oven for 6 h at 105 °C.³⁴²

4.2.8 Enzymic treatment. Enzyme treatment can be used as an effective, low-cost and eco-friendly method for the processing of NFRCs.⁵¹¹ A solution was prepared by mixing 1% of Texazym DLG, 3% of Texazym BFE and 0.2 g Texawet DAF in distilled water and jute fibres were soaked in this enzymic mixture at 50 °C for 2 h. Then, the fibres were rinsed with clean water and dried for 48 h at 30 °C.⁴¹² In another experiment, jute fibres were treated through the use of laccase and multi-enzyme synergistic effect, resulting in an improvement in the mechanical properties of JFRCs due to improvement in the cross-coupling of lignin by the enzymes.³⁷⁰ Also, jute fibres were treated with enzyme solutions such as pectinase, laccase, cellulase and xylanase in different ratios. The enzymes were observed to destroy the structures of hemicelluloses, lignin and pectin, which resulted in a decrease in the diameter and increase in the aspect ratio of the fibres.⁵¹¹

4.2.9 Stearic acid treatment. During the winding cycle, a mixture of stearic acid in acetone solution was added to the layers of the matrix and jute fibres. Subsequently, the metal plate containing the treated fibres was held for 30 min at 60 °C to remove acetone from the surface. Then, it was kept in a mould for 9 min at 205 °C under the load of 0.2 MPa.^{356,357} Finally, acetone was added to eliminate the extra particles from the fibre surface during the winding of each layer.³⁵⁸

4.2.10 KMnO_4 treatment. During the winding of fibres, a mixture of KMnO_4 and acetone solution was prepared and added to the matrix and jute fibres. Then, the metal plate with the prepared matrix and fibres was held for 30 min in an air-circulating oven at 60 °C to remove acetone from its surface, and eventually moulded by compression moulding.^{356–358}

4.2.11 Bleaching. Bleaching, which is an essential process for the creation of glossy fabric through use of hydrogen peroxide (H_2O_2), produces pure white colour fibres with reduced tensile strength.⁴⁶⁸ Bleaching employing peracetic acid produces less tenacity loss and whiteness without impairing the feel of the fabric. Therefore, bleaching in two steps is performed using both H_2O_2 and peracetic-acid, resulting in better properties. A change in the colour of jute fibres was seen from light golden brown to a dark colour due to the treatment and bleaching with H_2O_2 developed light yellow based brown fibres, showing the partial removal of lignin, wax and fatty portions.⁴⁴⁸ The effect of this treatment curing time was observed at different temperatures, and the fibres were observed to be well separated and isolated when treated for 6 h due to the formation of irregularities on their surface.⁴⁴⁹ An increase in the crystallinity of the fibres was seen only after 6 h. An increase in the modulus of jute fibre by 12% was seen within 4 h, after which it increased by 68% and 79% when treated for 8 h.

4.2.12 Toluene diisocyanate treatment. A solution of toluene diisocyanate in chloroform was prepared during winding and applied to the jute fibres and matrix. The prepared matrix and fibres were kept in a hot chamber at 100 °C for clearing the chloroform particles from the fibre surface and moulded by compression moulding process.^{356–358}

4.2.13 Nano-SiO₂ deposition. The deposition of nanoparticles on the surface of fibres is one of the most promising methods to enhance their interfacial bonding.^{512,513} Liu *et al.*⁴⁷⁸ used a sol-gel coating of nano-SiO₂ on jute fibres and found that nano-SiO₂ reacted with the jute fibres to create a stable interface with the hydroxyl groups. Then, the fibres were immersed in ethanol containing ammonia at a concentration of 0.15 to 0.75 mol L⁻¹. This solution was sonicated for one hour and stirred mechanically at 40 °C for 5 h. Lastly, the nano-SiO₂-coated jute fibres were washed in water to remove the excess material and then dried for 2 h at 70 °C.³⁵⁹

4.3 Advantages and disadvantages of chemical treatment process

4.3.1 Advantages. The objective of the surface treatment of plant fibres is to increase their bonding strength, and thus the stress transferability in the reinforced materials. The significance of surface-treated plant fibres was seen through the improvement of mechanical strength and dimensional stability of the resultant composites when compared with the untreated fibre-reinforced composites.⁷⁷ An improvement in the degree of crosslinking in the interface region and strong bonding can be achieved with the help of chemical treatments. Surface modification results in the breaking of bundles of composite fibres into micro-fibrils. This results in a decrease in the fibre diameter and roughness on the fibre surface, further resulting in better fibre-matrix interfacial adhesion with improved mechanical properties.⁴¹¹ Given that chemical treatment results in the removal of lignin and hemicellulose from the plant fibres, it affects their chemical composition, molecular orientation of the cellulose crystallites and degree of polymerization. Chemical surface modification can activate hydroxyl groups, which can create a strong bond with the resin.^{297,433,434} Effective modification is done through debonding of the hydrogen structure in the fibre, thereby enhancing the surface roughness of the fibre.⁴⁰⁰ The chemical treatment process removes impurities and hemicelluloses on the fibre surface and enhances the reactivity of the hydroxyl groups with the coupling agent. This treatment results in a finer fibre surface, increased crystallinity, reduction in defects, excellent bonding and hydrophobic nature.^{154,514} Chemical treatments are beneficial for the removal of large concentrations of wax, lignin, and oil that cover the outer layer of the fibre cell-wall, which decomposes cellulose and reveals crystallites. Also, this treatment induces fibrillation, which increases the effective contact area for interaction with the resin. Thus, the creation of an uneven surface texture and the improvement of the aspect ratio provide good interfacial bonding, which lead to a significant improvement in the properties of the composite.^{154,515,516}

4.3.2 Disadvantages. Chemical treatment decreases the spiral angle, and consequently the molecular orientation improves the elastic fibre package.^{435,446,517} The number of cellulose hydroxyl groups in the fibre-matrix interface is reduced by the chemical treatment process.⁵¹⁸ Acetylation involves the reaction of the cell wall hydroxyl groups of lignocellulosic materials with acetic or propionic anhydride at

elevated temperatures, which increases the hydrophobicity of the plant fibres. The hydroxyl groups of lignin and hemicelluloses react with the reagents, whereas the hydrogen bonding on the closely packed hydroxyl groups of crystalline cellulose prevent the diffusion of the reagent, and thus results in very low rates of reaction.⁵¹⁸

5. JFRCS

JFRCS consist of jute fibres as a reinforcement with a matrix at different interfaces. The fibres and matrix maintain their physical structure and chemical compositions during this process.⁵¹⁹⁻⁵²⁴ The reinforcement carries the load, while the matrix material holds the fibres in an exact position and direction, acts as a medium of load transfer to the fibres and protects them from environmental damage due to temperature and humidity.²⁹¹ JFRCS have high impact resistance and medium tensile and flexural strengths compared to other PFRCs.⁵²⁵ Studies have been reported on JFRCS, especially those containing polymer matrices.^{331,384,518,526-528} Not only the polymer matrix but also the reinforcement should be reusable for entirely bio-based composites. Therefore, the use of jute fibres together with polymer matrices in the manufacturing of composites at low cost has attracted attention from researchers.⁴⁶⁴ Contemporary studies recommended that jute fibres are excellent alternative reinforcement materials in polymer composites to replace synthetic fibres, which are costly and non-renewable.^{183,450} Table 8 presents the published works on JFRCS.

5.1 Processing methods

5.1.1 Hand lay-up method. Hand lay-up is an ancient method, which is the easiest and most extensively used technology for the manufacture of composites.⁵⁴⁷ Employing this method, fibres were impregnated in a matrix and layered in a mould over each other and pressed for 1 h. The resin and the hardener were mixed in the ratio of 100:32, before lay-up. Then, the mixture was applied to the fibres and spread uniformly using a roller, and the extra resin was squeezed out.^{548,549} An insulating material was placed between the mould plates and the releasing agent was applied to the inner surface of the mould. The composite was taken from the mould, kept at 30 °C for 48 h and cured for 16 h at 60 °C in an air-circulating oven.³⁴ The pressure was maintained, and the thickness of the material was dependent on the wt% of the fibres.⁵⁵⁶ A releasing gel was sprayed on the mould to enable the non-sticky removal of the materials.⁵⁴⁸ After 24 h of curing the resin at room temperature, it was heated for 2 h in an oven at 100 °C.³⁹³

JFRCS containing untreated and NaOH-modified jute fibres were produced in the shape of circular rods. The fibres were kept at 100 °C for 4 h, dipped in resin and pulled by hand through a cylindrical tube. The fibres inside the tube were cured at 30 °C for one day followed by 4 h at 80 °C.⁴⁴¹ The composite together with releasing sheets was placed between mould plates at 120 °C for 1 h and a uniform load applied.^{412,546} The composites were produced by chopping the fibres into plies and

Table 8 Reported experimental works on JFRCs

Reinforcement	Matrix	Surface treatment	Fabrication method	Ref.
Tossa jute	Polymer latex	Styrene butadiene	Mechanical mixing	353
Jute + glass	Isophthalic polyester	—	Hand lay-up	153 and 317
Jute	PLLA	—	Hot pressing	41
Jute	Epoxy	—	Stacking method	313
Jute	Epoxy	—	Vacuum-assisted resin infiltration	343
Jute	Epoxy	H ₂ SO ₄ , H ₂ O ₂ , Na ₂ SiO ₃ , Na ₃ PO ₄	Hand lay-up	434
Jute	Epoxy	NaOH	Vacuum infusion	451
Jute	Epoxy	NaOH + silane	Vacuum-assisted resin infusion	432
Jute	Epoxy (MGS RIM 135)	—	Vacuum infusion	529
Jute	Epoxy	NaOH/3-aminopropyl-triethoxy-silane solution/3-phenyl-aminopropyl-trimethoxy-silane	—	382
Jute	Epoxy	—	Compression moulding	315 and 381
Jute	Epoxy	—	Hand layup	83, 311, 314, 391 and 530–532
Jute	Epoxy, polyester	NaOH	—	5
Jute + clay	Epoxy	NaOH	Mould method	385
Jute	PLA	—	Injection moulding, compounding and extrusion	81
Jute	PLA	—	Compression moulding	363 and 533
Jute	PLA	NaOH	Injection moulding	302
Jute	PLA	NaOH + silane	Melt mixing	349
Jute	Polyester	—	Pultrusion	336 and 338
Jute	PP	—	Compression moulding	389, 392 and 534
Jute	PP	—	Melt mixing	390
Jute	PP	—	Injection moulding	535
Jute + banana	Cashew nut shell liquid (CNSL)	—	Hand layup	536
Jute	Poly(3-hydroxybutyrate-co-3-hydroxyvalerate)	NaOH + acetic acid	Compression moulding	443
Jute	Portland pozzolana fly ash cement + carboxylated styrene butadiene	NaOH	Mixing	327
Jute	Polyester (crystic P9901)	—	Pultrusion	337
Jute	PP	Sodium periodate	Compounding and extrusion	342
Jute	Soluble starch	NaOH + dimethyl sulphoxide (DMSO)	Solution casting method	376
Jute	Poly butylene succinate (PBS)	NaOH + Na ₂ SiO ₃ + Na ₂ P ₃ O ₁₀ + Na ₂ SO ₃	Mixing method	371
Jute	Vinylester (HPR 8711)	NaOH	Hand layup	441
Jute	Elekeiroz S.A and Resana S.A.	NaOH	Mould method	230
Jute	PP	—	Hot pressing	537
Jute	PP	—	Screw mixing	538
Jute + hemp	PP	NaOH + MAPP	—	58
Jute	PP	MAPP	Injection moulding	59 and 99
Jute	PP	MAPP	Compounding and extrusion	348 and 539
Jute	PP	MAPP	Pultrusion	292
Jute	Epoxy	Infrared laser, ozone, enzyme and plasma	Hand layup and compression molding	412
Jute	Glucufuranoside based trifunctional EP monomer	Methyl hexahydrophthalic-acid (MHHPA)	Compression moulding	182
Jute	Epoxy	NaOH	Vacuum infusion	386 and 387
Jute	Polyester	NaOH	Compression moulding	540

Table 8 (Contd.)

Reinforcement	Matrix	Surface treatment	Fabrication method	Ref.
Bangla Tossa	PP	NaOH	Compression moulding	347 and 515
Jute	PP	Alkaline + silane	—	61
Jute	Methacrylated soybean oil	NaOH	Compression moulding	297
Jute	Poly lactide (GCS PLA 4320)	NaOH + benzoyl peroxide	Solvent casting	368
Jute	PP	KMnO ₄ , MAPP, TDI and ST	Commingling	356–358
Jute	PP	KMnO ₄ , MAPP, TDI and ST	Compression moulding	355
Jute	Polyester	NaOH	Hand layup	367 and 541
Jute + glass	Polyester	—	Resin transfer moulding	542
Jute	Polyester	Alkali, micro-emulsion silicon and fluorocarbon	Compression moulding	543
Jute	Polyester	—	Film stacking	341
Jute	Vinylester	—	Compression moulding	361 and 544
Tossa jute	Epoxy	Corona + UV treated	—	383
Jute	PP	MAHgPP	Hot pressing	545
Jute	PP	MAHgPP	Compounding and extrusion	105
Jute	HDPE	MAPE	Melt mixing	362
Jute	Carboxylated styrene butadiene	NaOH	—	353
Jute	LDPE	2-Hydroxyethyl methacrylate	Compression moulding	344
Jute	Polyethylene	NaOH, H ₂ SO ₄ + CH ₃ COOH (acetic acid)	Hot pressing	345
Jute + glass	Epoxy	—	Hand layup	316
Jute + banana + glass	Epoxy	—	Hand layup	377
Jute	Epoxy	Succinic Anhydride + phthalic Anhydride	Hand layup	378
Jute	Epoxy (diglycidyl ether of the bisphenol A)	—	Mould method	384
Jute	Epoxy resin Kinetix R240	—	Hand layup	24
Jute	Green epoxy resin CHS-Epoxy G520	Sulphuric acid (H ₂ SO ₄) and NaOH	Hand layup	546
Jute + glass + Kevlar	Epoxy	—	Hand layup	547
Jute + basalt	Epoxy resin SX8 EVO	NaCl	Vacuum assisted resin infusion	350
Jute	Epoxy (LY 556)	—	Hand layup	548 and 549
Jute + Al ₂ O ₃	Epoxy (LY 556)	NaOH + ethanol	Hand layup and compression moulding	550
Jute	LPDE	NaOH + oligomeric siloxane (Z-6173)	Compression moulding	339
Jute	Tetraethyl orthosilicate (TEOS) and ethanol	NaOH + nano SiO ₂	—	359
Jute + carbon	Polyvinyl butyral	—	Hot press technique	321
Jute	Recycled PP	—	Hot press technique	388
Jute + copper slag	Polyester	NaOH + calcium hydroxide	Compression moulding	322
Jute	Polyester	NaOH + PLA coating	Hand layup and compression moulding	551
Jute	Epoxy	NaOH	Compression moulding	487
Jute	HDPE	—	Hot pressing	364
Jute + Kenaf	HDPE	—	Microwave assisted compression moulding	552
Jute	Epoxy DTE 1100	NaOH + SiC	Hand layup and vacuum bagging	553
Jute	PP	MAPP	Compression moulding	379
Jute	Polyurethane	NaOH + silane	Extrusion	374
Jute	PP	Phosphoric acid (H ₃ PO ₄)	Extrusion	554
Jute	LDPE	NaOH	Hot pressing	346
Jute	Polyester	—	Hand lay-up	555

applying resin to them. The fibre laminates inside the mould were allowed to cure for 24 h at 30 °C. Then, the laminates were left out for 15 days to obtain the samples for testing.²⁹³ The load was applied to the top of the mould and transferred to the bottom portion of the composite laminate, and then it was allowed to settle for 48 h at 30 °C. Subsequently, the composites were taken out and placed in an airtight container for 48 h for further testing.³¹¹

5.1.2 Film stacking method. The development of JFRCs using the film-stacking technique is explained in Fig. 3(a). The fibres are stacked on each side of multiple layers of resin along their length at 90°. The lay-ups are subjected to quick press consolidation involving the following steps: (i) pre-compression, (ii) heating under vacuum, (iii) quick transfer to

a press for consolidation and cooling, and (iv) removal from the press. In a standard experiment, a lay-up of PLA films and jute fibres was placed in a metal frame, pre-pressed at 3.3 MPa for 15 s, and then transferred to the heating stage. The lay-up was moved immediately, the heating section was automatically sealed, and the pressure was reduced to 400 Pa within 1–2 min. The pre-compressed lay-ups were heated at 180–220 °C for 3–10 min. Following the completion of the heating, the lay-up was moved back to the press, where the assembly was stabilized by applying a load of 3.3 MPa.³⁶⁶

5.1.3 Compression moulding. The compression moulding technique is used for the fabrication of composites without any damage to the fibre during processing.^{53,366,410,557} In this process, the resin is dissolved in chloroform and stirred for 4 h for the

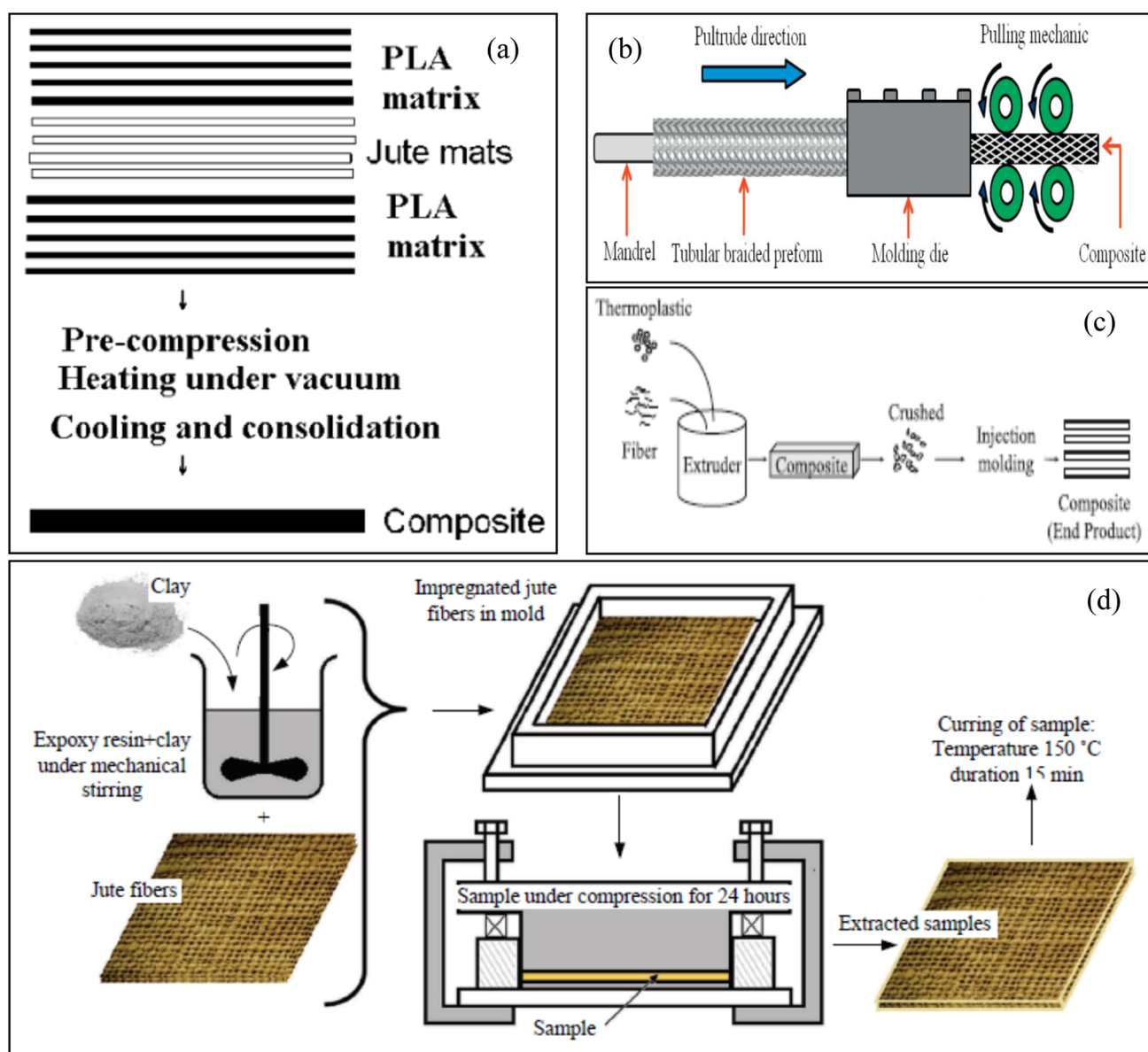


Fig. 3 Process for the fabrication of JFRCs: (a) film stacking process and (b) pultrusion process. Reproduced from ref. 129 with permission from Elsevier, 2013. (c) Injection molding. Reproduced from ref. 587 with permission from Elsevier, 2020. (d) Mould method. Reproduced from ref. 385 with permission from Springer, 2017.

preparation of a homogeneous mixture at room temperature. Then, the mixture is poured into a mould for the preparation of a film and dried at 60 °C to evaporate the excess chloroform. Finally, the dried films are kept in a press and a loading of 13.34 kN applied at 166 °C to obtain a uniform laminate.⁴⁴³ A hot compression moulding machine was used for the production of JFRCS from continuous fibres. The matrices were melted by heating and quickly wetted with jute fibres.³⁶³ The fibres were dried at 80 °C for 2 h under 50 mbar before composite preparation. Then, these laminates were pressed with a hydraulic pressure of 200 bar to get a uniform thickness.¹⁸²

Jute fibres were cleaned, dried and immersed for 1 h in NaOH solution, washed, and then dried again at 50 °C. Subsequently, 3% nano-clay was mixed with resin and stirred for 2 h, and then put in an oven for the elimination of voids such as air bubbles. The jute fibres were cut into dimensions of 300 × 300 mm², and the resin fibre ratio of 1 : 3 was considered. Two layers of jute fibres were employed for the preparation of the laminate together with 1.5 wt% resin, accelerator and catalyst.⁵⁴⁰ The mould was kept in a compression moulding machine at 200 °C and a load of 0.2 MPa was applied.³⁵⁵ The fibre was submerged in a benzene peroxide solution and allowed to dry for 2 h at 80 °C. Composites were prepared with the addition of three layers of low-density polyethylene sheets and two layers of jute. The sheets were processed by heating the polymeric granules at 115 °C and kept under pressure for 5 min. The laminates were placed at 135 °C and a hydraulic press was used for moulding them.³⁴⁴ The jute fibre was initially washed with water to clean the dust and other particles found in the fibres. The first layer of the specimen was placed in the die, then resin was spread over the fibre, and allowed to dry for 10–15 min. Prior to drying the resin, it was layered with a second layer, and then all the remaining layers were layered. Finally, the composites were kept in a moulding machine to achieve the perfect samples through application of a load in the range of 250–400 N.³¹⁵

5.1.4 Pultrusion. Pultrusion is an automatic and consistent process for producing composite components of high volume with uniform cross-sections, in which the wetted fibres are drawn through a die at a constant velocity. This method offers good efficiency and ease of processing, but there is a limited number of reports on the pultrusion of PFRCs, and in particular their resistance to moisture.^{337,558–561} There are many parameters to consider during this process, among which the drawing speed and temperature are the most important, which were taken as 180 mm min⁻¹ and 85 °C in a report.³³⁶ Previous studies^{337,560} demonstrated circular cross-section rods pultruded by reinforcing jute fibres. Fig. 3(b) shows a schematic of the pultrusion process for the fabrication of composites.¹²⁹

Jute fibres were impregnated in a resin tank filled with polyester resin. To obtain the desired form, a pulling system was used to move the resin-mixed jute fibres through a die. The curing was performed in the die, which was accurately designed to give the shape of the end product.^{562,563} The pultrusion technique was applied using an extruder fitted with a die assembly to cover fibres with a resin and MAPP mixture. Specifically, 3 wt% MAPP was applied to the pure resin. The extruder and die were heated at a maximum temperature of

200 °C. The coated fibres were washed with water and cut into 4 mm long granules. The granular particles were dried overnight at 110 °C. Under the same conditions, they were once again extruded with the same extruder to homogenize the fibre/matrix mixture.²⁹²

5.1.5 Hot pressing. The fabrication of composite by the hot pressing process is done in two stages. In a reported study, in the first stage, resin films with a thickness of 0.2–0.3 mm were fabricated by applying a load of 250 kN in a hot pressing machine at 250 °C. Then, the sheets were kept at 180 °C and 50 kN pressure for 10 min and cooled by running water. In the next stage, the jute fibres were dried for 24 h at 60 °C. A mould with a size of 15 × 15 cm² was used, filled by woven jute mat between the resin films and kept in a hot press.⁴¹ In another study, oven-dried jute fibres, polyethylene pellets, and nanoparticles were mixed, the mixture was moulded and kept in a press at 180 °C and a load of 7 MPa applied for 1 h. Then, the mould was cooled at 30 °C and the test specimens were prepared.³⁴⁵

5.1.6 Melt mixing method. JFRCS were processed using the melt mixing method in a setup with roller blades and a volumetric chamber. The fibres and resin were premixed at different wt% of fibre and kept in a pre-heated chamber at 150 °C. Mixing was achieved at a speed of 25 rpm for 10 min. Then, the chemically modified fibres were combined with resin at 160 °C and maintaining the same rotor speed. Subsequently, these pre-mixes were moulded using equipment at 150 °C and a uniform load applied.³⁶² Jute fibres were laid in a mould and Teflon coatings were applied on the top and bottom of the mould for the easy removal of laminates. Then, the assembly was hot-pressed by applying a load of 4 MPa at 165 °C. The polymer melted and impregnated the jute fibres at this temperature and compaction occurred. Subsequently, the pressure was increased to 6 MPa, and then the composite was left for 10 min. Then the composite was taken out from the mould and samples were prepared for testing.

5.1.7 Compounding and extrusion. Gunning *et al.*⁵⁶⁴ examined the processing conditions for the fabrication of composites through extrusion and found that the composite produced using lower temperature profiles exhibited good properties at higher temperatures due to the reduction in the thermal degradation of the fibres during the process. They manufactured composites with different fibre loadings using jute, hemp and lyocell fibres. While the average residual fibre length was short, the composites showed enhanced mechanical properties. Also, the extrusion temperature, screw speed, and configuration were found to have a dominant impact on the consistency of the composite regarding the operation employing a twin-screw extruder. Degradation occurred at 180 °C, particularly for the natural fibre, which was higher than the melting temperature of the resin. Thus, the temperature was set to 180 °C to minimize the degradation of the jute fibre during fabrication. The compound was actually extruded a temperature 10–20 °C higher than the set temperature. Furthermore, the screw velocity was operated at 150 rpm to reduce the thermal degradation by shear heating.⁸¹

Initially, jute fibres were mixed with PP granules and the mixtures were passed through an extruder unit at 135 °C. Using

a grinding machine, all the bits were broken into smaller granules with a size of 3 mm to 0.5 mm.³⁴² A twin-screw extruder was employed for the direct thermoplastic processing of long fibres.³²⁸ The fibres were washed and dried at 60 °C for 48 h before compounding, until a uniform weight was reached. Then, the fibres were integrated in the side feeder of the extruder, which fed them directly into the melting of polymers. Also, the content of the fibres in wt% was regulated by the speed of the screw, which was determined using the targeted material, fibre weight, length, and roving time.³⁴⁸ In another study, composite samples were compounded on a twin-screw extruder. Dog-bone shaped specimens were made from compound granules using an injection moulding machine.¹⁰⁵ Reinforcing jute fibres were dehydrated for one day at 50 °C and 4 h at 80 °C before extrusion to remove moisture. The constituent materials were fed into the extruder. The screw speed of the extruder was fixed at 145 rpm and the temperature range was 165–195 °C. The composites were pelletized to obtain granules, which were dried at 85 °C for 12 h and used for the fabrication of the samples.⁵³⁹

5.1.8 Injection moulding. The mass production of composites is usually performed by the injection moulding process,³⁰² which has the benefits of high processability and ability of the fibre to degrade during the process.⁸¹ In this process, the fibres are washed and chopped to 3 mm.⁵⁶⁵ Yang *et al.*⁵⁶⁶ manufactured JFRCs using an injection moulding process. They observed an increase in the modulus after the addition of the jute fibre; however, there was a decrease in the tensile strength. An extruder compounded the jute fibre, resin, and alkaline solution at a temperature in the range of 165–180 °C. Following immersion in a water bath, the fibres were cooled and cut into granules. For processing, the granules were kept at 100 °C for 4 h and injected into composites. The injection moulding process had three-zone temperature profiles ranging from 160–195 °C.⁵⁹ Fig. 3(c) shows the standard manufacturing cycle for JFRCs by injection moulding.⁵⁶⁵

5.1.9 Vacuum infusion. The method of vacuum infusion was designed for the manufacture of NFRCs.⁵⁶⁷ The fibres were arranged in layers and pre-compacted by applying a pressure of 1200 kPa in a hydraulic press. The fibres were layered on a mould and coated with silicone gel after compaction. Two vacuum tubes were connected, one with a pump, while the other was inserted in a resin bottle. The tubes were mounted on the laminate layers, and a sealant tape was used for sealing the entire system. Using a vacuum pump, 3 kPa vacuum was drawn. The laminate was kept under vacuum for 1 h to ensure the complete evaporation of moisture.^{386,387} Resin and hardener were mixed at a ratio of 24 : 100 and degassed in a desiccator. The product was drawn by the differential pressure produced by the vacuum through the laminate *via* the resin inlet tube, and the stacked fabric plies were completely wetted. After the laminates were fully wetted, the vacuum was reduced to 35 kPa by placing a dead weight on top of the laminate and it was left overnight for setting. Then, the panel was removed from the vacuum and heated at 50 °C for 3 h and 80 °C for another 3 h.

5.1.10 Solvent casting method. The solvent casting process is desirable for the manufacture of composites with improved

strength due to the improvement in wettability and better bonding of the fibre with the matrix. The resin is dissolved in chloroform and impregnated with jute fibres and the solvent is gradually evaporated. Subsequently, the pre-pregs are kept at 60 °C for one day to remove the chloroform completely. Finally, the pre-pregs are layered in the mould and pressed with a load of 5 MPa at 170 °C for 5 min.³⁶⁸

5.1.11 Mould method. Samples were prepared *via* the impregnation of jute fibres with resin and different clay materials in a mould. The mould was kept in a hydraulic press until it achieved the desired thickness. Then, the compressed system was maintained for 24 h and demoulding was done. The preparation of JFRCs by the mould method is shown in Fig. 3(d).³⁸⁵

5.1.12 JFRCs with adhesives. Composite laminates with jute fibres and Betamate 2096 adhesive were fabricated using the hand lay-up and hot press compression moulding technique using a steel mould and a heated plate hydraulic press. The composites were fabricated in a way to have a stacking sequence consisting of 5 layers of jute. The fibre loading was kept at around 30 wt% and the adhesive at 70 wt% of the final fabricated composite. The curing time was of 8 h at 70 °C and the adherends were cut from the composite plates.⁵⁶⁸

5.1.13 Characteristics of fabrication methods. The methods for the fabrication of PFRCs depend mainly on factors such as the characteristics of the matrices and reinforcements, the shape and size of the part produced, and the users. The characteristics of the fabrication technique varies from product to product. To obtain good adhesion, it is necessary to address issues such as material preforms, processing parameters, and post-processing techniques.⁵⁶⁹

5.2 Properties of JFRCs

PFRCs are highly dependent on major factors such as properties of the reinforcing fibre, matrix, and interfacial bonding between them.^{246,569,570} Composites are exposed to the environment during processing, which may affect their properties. The properties of the composites have been studied by several researchers, resulting in a substantial improvement in their properties.^{62,74–77,257,258,345} Heat treatment of fibres can reduce the water content in the fibres and improve the properties of the resulting composites by enhancing the compatibility between the fibres and polymer.⁵⁷¹ The mechanical properties and hardness of jute fibre-reinforced composites were evaluated and an enhancement in properties was observed with the addition of fibres. Furthermore, the composites exhibited good thermal stability and decomposition.⁵⁷² In another study, an enhancement in the properties of JFRCs was observed through the incorporation of graphene powder during the processing of the composites.⁴⁵¹

5.3 Physical properties

5.3.1 Hardness. The hardness is measured using a Shore-D type hardness tester³⁵⁷ and a Rockwell hardness tester^{5,342} according to the ASTM D785 standard.^{5,342,357} In one report, this test was conducted using a 1/16" ball indenter and applying a load of 1000 N. The tester was placed vertically and pushed

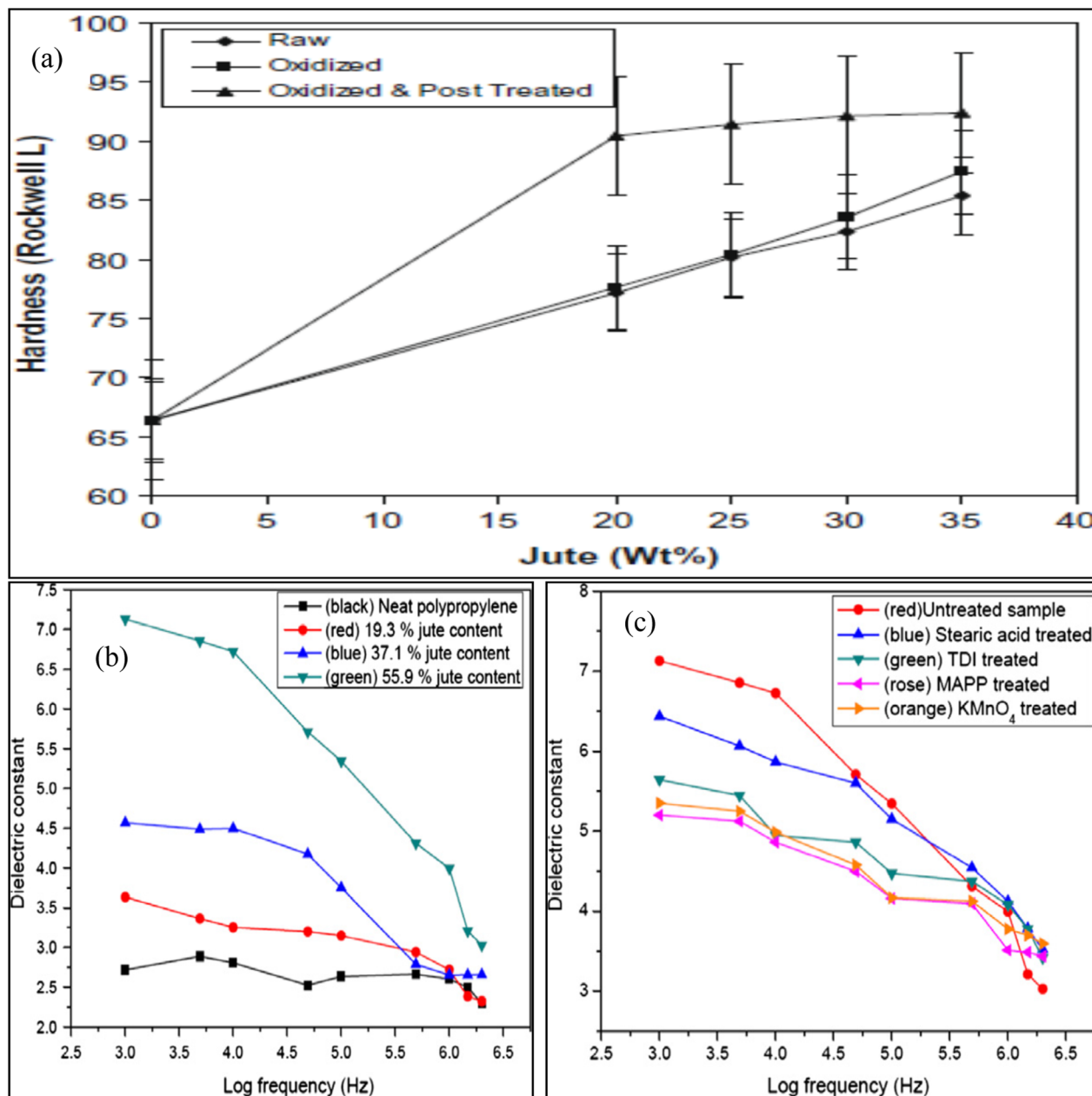


Fig. 4 Influence of the surface modifications of fibres on the (a) hardness and (b and c) dielectric constant values of JFRCS.

onto the specimen for alignment of the presser foot to the specimen.³⁷⁵ With oxidation of jute fibre and fibre packing, there was an increase in average hardness due to the flexibility and stiffness of the composites.⁵⁷³ Compared to the untreated and oxidized fibres, the treated JFRCS yielded much greater hardness (Fig. 4(a)). This was due to the uniform fibre dispersion in the resin with reduced voids and good bonding between them.³⁴² Also, the micro-hardness of the composites was measured using a Leitz micro-hardness tester. In this test, a diamond indenter was pushed into the specimen at 136° between opposite faces and subjected to load. The indentation mark left on the specimen was determined after the load was removed, and the arithmetic mean was calculated.⁵⁷⁴ The

durability of the Vickers number was determined using the equation:

$$H_v = 0.1889 \frac{P}{L^2} \text{ and } L = \frac{X + Y}{2} \quad (1)$$

where P is the applied load, L is the diagonal of the indenter and X and Y are horizontal and vertical lengths, respectively.

5.3.2 Chemical resistance test. The chemical absorption characteristics of composites have been evaluated to determine their resistance to chemical substances according to ASTM D543-87 requirements, where an increase in weight suggests that the material has low chemical resistance.⁵⁷⁵ The influence of chemicals such as benzene, toluene, CCl₄ and purified water; acids such as HCl, H₂SO₄ and acetic-acid and alkali solutions

such as NaOH, Na₂CO₃ and NH₄OH has been tested. For example, specimens were soaked in chemical solutions for a day, taken out and rinsed with water, and cured at ambient temperature. Then, the weight of the specimen was measured and the weight gain/loss percentage (W_{CR}) was calculated using the following equation:³⁴⁵

$$W_{CR} = \{(W_f - W_i)/W_i\} \times 100 \quad (2)$$

where W_i and W_f are the weight of the samples before and after immersion, respectively.

5.3.3 Dielectric properties. Dielectric experiments are conducted to measure the dielectric constant, loss factor, dissipation factor, resistivity and conductivity of a material. In composites, their dielectric properties depend on the polarization of their constituents and mainly influenced by interlaminar, dipolar, and atomic polarization.^{303,576–580} The polarization of the interfaces at very low frequencies has a greater effect on the dielectric properties. Interfacial polarization occurs when charge carriers are trapped at the intersection of heterogeneous systems, primarily by dipoles in the case of NFRCs. In an external field, these dipoles may get turned to some degree, and thus contribute to the polarization of the material. However, there is a decrease in the magnitude of the effect of interfacial polarization with an increase in frequency. In plant fibres, the availability of –OH groups contributes to their polarization.³⁰³ The dielectric constant is calculated using eqn (3), as follows:

$$\epsilon' = \frac{Ct}{A\epsilon_0} \quad (3)$$

where C is the parallel capacitance, t is the width, A is the cross-sectional area, and ϵ_0 is the free space permittivity (8.85×10^{-12} F m⁻¹). The resistivity and conductivity of the sample can be calculated using the following equations:

$$\text{Resistivity } \rho = \frac{RA}{t} \quad (4)$$

$$\text{Conductivity} = 1/\rho \quad (5)$$

where R is the parallel resistance and ρ is the resistivity.

In a study, the test samples were mounted in parallel plates between electrodes and the measurements were performed in the frequency range of 100 and 2×10^6 Hz. Higher temperatures were not investigated, given that resin softening occurred at 160 °C.³⁵⁵ The dielectric constants increased at all frequencies with an increase in the fibre wt%, as shown in Fig. 4(b). Both types of polarization contribute to the polarizability of non-polar molecules, hence displaying lower dielectric constants at all frequencies. Jute fibres are polar in nature, and therefore were incorporated in a non-polar resin, which resulted in an increase in the amount of polar groups in the material, resulting in dipole and orientational polarization. JFRCs are heterogeneous materials, and thus their interfacial polarization often effectively leads to a dielectric constant. Hence, the dielectric constant increased at all frequencies for the fibre materials. A full dipole orientation was possible at low frequency, leading to

an increase in the dielectric constant. However, at high frequency, the vibrations of the particles were strong, and thus there was no full orientation of the dipole. There was a decrease in the dielectric constant with an increase in frequency.³⁵⁵ The dielectric constant of the composites was determined using the following equation:

$$\epsilon = C/C_0 \quad (6)$$

where C is the capacitance and C_0 is the capacitance under vacuum. C_0 is determined from the sample dimensional information using the following equation:

$$C_0 = 8.8510 - 12At \quad (7)$$

where t is the thickness.

The influence of chemical modifications on the dielectric constant of JFRCs is presented in Fig. 4(c), which clearly shows that the raw fibre-reinforced sample has the maximum dielectric constant compared to the chemically treated fibre samples. This proved the reduction in the dielectric constant due to the chemical treatment of the fibres. The treatments minimized the hydrophilicity of the fibres, thereby reducing the absorption of moisture and number of polar groups in the composite. This resulted in a decrease in the polarization orientation, resulting in low dielectric constant values. The surface treatments improved the adhesion, resulting in a reduction in the amount of voids and defects and a reduction in the absorption of moisture, which caused a reduction in the dielectric constant. Treatment with stearic acid resulted in only slight topographical changes on the surface of the fibre, with a difference between the dielectric constants of the chemically treated and untreated fibre composites.³⁵⁵

5.3.4 Dimensional stability. The dimensional stability of composites undergoes changes in dimensional factors, namely, length, thickness, and width, when they are exposed to different environmental conditions such as humidity and dry and wet conditions. In this case, the stability of composites is important for determining their shelf life and end-user applications. To determine the dimensional stability, characterization tests such as density, thickness swelling, water absorption, weathering and cyclic tests are conducted.⁵⁸¹ Dimensional stability can also be analysed *via* FTIR, XRD, TGA, and DSC analysis.

5.4 Mechanical properties

Impregnation and good bonding must be ensured for the production of composites for industrial applications and the exploitation of fibres as reinforcement materials, given that the final mechanical properties depend on the bonding between the fibre and resin.^{156,582,583} Natural fillers have become significant over the past decade, given that they improve the strength of composites.^{525,581,584–587} The factors influencing the strengths of NFRCs are as follows: (i) type and strength of reinforcements, (ii) type of resin and mixing ratio, and (iii) method by which composites are blended, casted and cured.⁵⁸⁸ Among them, the fibre–matrix compatibility, which leads to a uniform distribution of fibres, is one of the prime factors affecting the strength

of composites.⁵⁸⁹ Many researchers have studied the mechanical properties of NFRCs^{98,100,331,435,517,590,591} and the results showed that the tensile and flexural strength of root portion fibre-based composites is higher than that of tip-portion fibre-based composites. Alternatively, the modulus of tip-portion fibre composites was observed to be higher than that of root-portion fibre composites.⁵⁹² Various researchers have conducted studies on the mechanical properties of jute fibres and JFRCs.^{103,156,202,230,278,331,383,593–606}

5.4.1 Tensile strength. Tensile testing involves the application of load along the axis of a specimen until it fractures, and the elongation of the specimen against the load is noted based on the increase in gauge length.²⁶⁰ The test specimens are prepared according to the ASTM D3039,^{5,83,315,329,336,343,379,387,393,529,540,574} ASTM D638,^{99,230,302,313,314,317,339,340,342,345,346,362,363,366,368,375,377,378,381,390,392,539,544,546,550,607–609} ASTM D882,^{41,376} ASTM D2256-02,⁴⁴³ ASTM D3822,³²⁷ ASTM C1557,³¹⁹ ISO 527,^{311,351,607} GB1447-83,³⁷¹ DIN EN ISO527-4 (ref. 365) and DIN 53455 (ref. 56) standards. Experiments were conducted using a universal testing machine

(UTM),^{5,41,81,83,311,314,315,317,327,340,342,345,346,351,362,377,379,540} including Instron models 4206,³⁵⁷ 1026,⁵⁶ 3385,³⁶⁸ 3382,⁵³⁹ 1122,⁶⁰⁷ 1342,⁶⁰⁸ 1195,⁵⁵⁰ 8821S,³⁸⁵ 3369,³¹³ 6025,³⁶⁶ and 4400R,³⁸⁷ Zwick Z020,¹⁸² Krystal-Elmec,³⁷⁸ Mecmesin,⁶⁰⁹ MTS 370,⁵⁴⁶ Shimadzu AG-X,³¹⁹ Shimadzu AUTOGRAPH AG^{339,529} and Zwick Roell.⁴⁴³ Samples were prepared with the dimensions of 300 × 25 × 3 mm,⁵⁴⁰ 140 × 10 × 2 mm,¹⁸² 255 × 25 × 2 mm,³⁸⁷ 110 × 50 × 10 mm,⁴¹ 165 × 13 × 3 mm,³⁶² 180 × 25 × 15 mm,^{363,366} 148 × 10 × 4 mm,³⁴² 115 × 6 × 3.1 mm,^{345,346} 200 × 20 × 4 mm,⁵⁴⁶ 150 × 15 × 3 mm,⁵⁵⁰ 165 × 25 × 3 mm,³³⁹ 250 × 25 × 3 mm,³⁷⁹ 165 × 19 × 3.2 mm (ref. 392) and 120 × 84 × 20 mm.⁸³ The test was performed with a cross-head speed in the range of 1–10 mm min⁻¹, gauge length of 5–50 mm and load-cell capacity of 5–100 kN.^{34,41,81,83,182,313,327,336,339,340,342,345,346,357,366,368,371,375,378,379,387,393,443,544,546,607–609} Many researchers have performed experiments under atmospheric conditions of 20–35 °C and 65% relative humidity. The test specimen geometry is indicated in Fig. 5(a).³¹⁵

The tensile strength and modulus can be evaluated using the following equations:

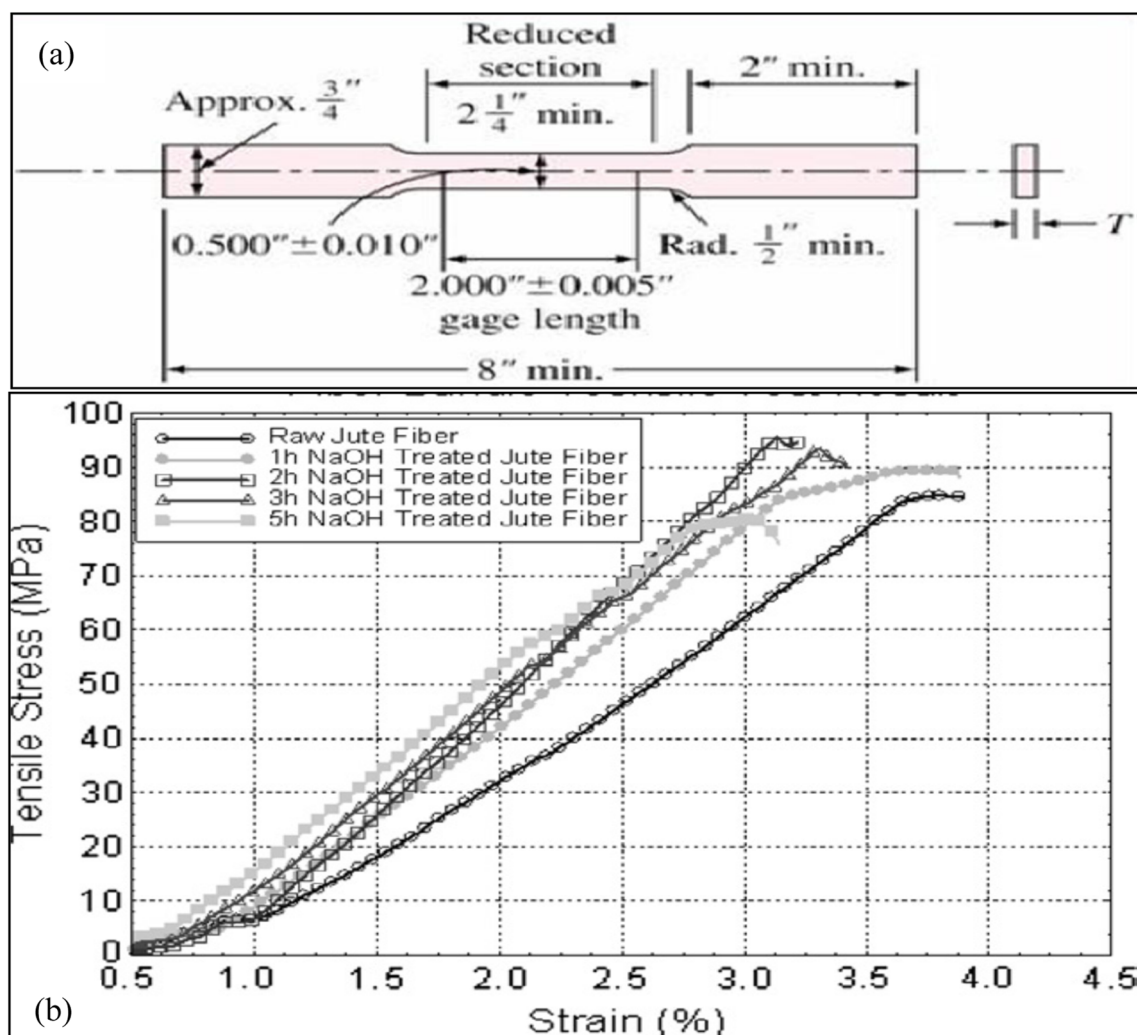


Fig. 5 Tensile testing of JFRCs: (a) scheme of the tension test specimen according to the ASTM standards. Reproduced from ref. 315 with permission from Springer, 2016. (b) Typical tensile stress vs. strain curves.

$$\text{Tensile strength } \sigma_t = Pl/bh \quad (8)$$

where P is load, b is the specimen width, and h is the specimen thickness.

$$\text{Young's modulus } (E) = \text{stress/strain} \quad (9)$$

Fig. 5(b) displays the typical uniaxial tensile test results of jute fibres. The strength and modulus of NaOH-treated fibres increased by 13% and 17% compared to the untreated fibres. The NaOH treatment induced the removal of non-cellulosic products, such as hemicellulose, lignin, and pectin from the inter-fibrillary regions⁶¹⁰ and resulted a higher tensile strength and modulus.⁶¹¹ An exposure period of 2 h with 5% NaOH showed acceptable results.⁴⁴³

5.4.2 Flexural/bending strength. Flexural strength is a combination of both compressive and tensile strength and directly varies with the shear strength. Specific processes, such as stress, stretching and shearing, occur simultaneously during flexural testing.⁶¹² The flexural test of JFRCS has been carried out using UTM,^{5,362,377,379,381} Hounsfield UTM,^{41,354,361,613} Zwick/Roell Z005,³⁵⁰ Zwick/Roell Z010,^{336,443,542} Zwick Z020,¹⁸² Instron 4303,^{361,441} Instron 3382,⁵³⁹ Instron 1195,⁵⁵⁰ Mecmesin,⁶⁰⁹ Shimadzu AGS-J,⁵⁴⁶ Shimadzu AUTOGRAPH AG-G Series,³³⁹ and LLOYD LR30K³⁴¹ machines. The sample dimensions for the flexural test were obtained according to the ASTM D790,^{5,41,99,153,313–315,317,336,339,341–343,350,357,361–363,368,377–379,381,390,392,441,443,539,542,546,608} ASTM 348–80,⁶¹⁴ ASTM D5943-96,⁵⁵⁰ ASTM CISO 14125:1998,^{311,351} GB 1449–1983,³⁷¹ DIN 53452,⁵⁶ DIN EN ISO14125 (ref. 365) and DIN EN63 (ref. 383) standards. The test was carried out using samples with dimensions of $79 \times 10 \times 1$ mm,⁴¹ $56 \times 20 \times 3.6$ mm,³⁶³ $100 \times 10 \times 2$ mm,¹⁸² $79 \times 10 \times 4.1$ mm,³⁵⁷ $80 \times 15 \times 1.5$ mm,^{362,383} $79 \times 10 \times 4.1$ mm,³⁴² $110 \times 20 \times 20$ mm,^{354,361,613} $160 \times 12.7 \times 4$ mm,⁵⁴⁶ $100 \times 15 \times 3$ mm,⁵⁵⁰ $13 \times 12.7 \times 5.5$ mm,³⁷⁹ $116 \times 24 \times 13$ mm,⁵³⁰ $76 \times 25 \times 3.2$ mm (ref. 392) and $110 \times 20 \times 20$ mm.⁶¹³ A span width in the range of 30–140 mm and cross-head speed in the range of 1–5 mm min⁻¹ have been considered by various researchers.^{41,182,329,341,350,357,361,362,368,371,441,443,542,608,609,613} The samples were screened at room temperature fitted with a 2 kN load-cell.³⁷⁹ The flexural strength can be calculated using the following formula:

$$\text{Flexural strength } \sigma_f = 3PL/2bh^2 \quad (10)$$

$$\text{Flexural strain} = 6sh/L^2 \quad (11)$$

where P is load, L is the length, b is the width, h is the thickness, and s is the deflection.

The details of the flexural strength of JFRCS are presented in Fig. 6. The results showed that the treated composites possessed improved strength and modulus. However, the brittleness in the composites was due to the addition of nano-clay, which was evident from the decrease in the strain value at high stress. No fibre fracture in both nano-phased and traditional composites was found. It was also noted that JFRCS exhibited high flexural strain, which suggests the presence of ductility in

these bio-composites.⁴⁴³ In the case of the treated composites, high flexural strength and modulus were obtained compared with the untreated composites. The nano-clay-infused samples also performed better in terms of flexural characteristics compared to traditional composites. Nevertheless, following the absorption of moisture, a reduction in the flexural intensity and modulus was observed. The highest decrease in flexural strength was observed in the untreated composites and the lowest found in the samples containing 4 wt% nano-clay. The flexural modulus values were 21% and 6–7%, respectively. This is an example of the moisture-arresting behaviour of nano-clay in the composites.⁴⁴³ The typical flexural stress vs. strain curves are shown in Fig. 6(a–c). With a shorter immersion duration, a dramatic increase in the average flexural load was observed. It was assumed that the cellulose content in the jute fibre was reduced and that the plasticization effect caused the jute fibres to become more versatile. The flexible nature of the material obtained was due to the absorbed water molecules, which were stored in the cavities and cracks, acting as a plasticizer.³³⁷ The flexural modulus can be calculated using the following equation:^{34,361}

$$\text{Flexural modulus} = L^3m/4bt^3 \quad (12)$$

where L is the support length, m is the slope of the load vs. deflection curve, b is the width and t is the thickness.

5.4.3 Compressive strength. Compression test specimens were prepared according to the ASTM D695 (ref. 337 and 540) standard. The size of the rectangular specimen was $13 \times 13 \times 3$ mm,⁵⁴⁰ while that of the circular specimen was 12.7 mm and 25.4 mm.³³⁷ Tests were conducted on a 400 kN capacity UTM,⁵⁴⁰ Instron 3367 (ref. 337) and hydraulic UTM.^{354,613} The tests were carried out at a cross-head speed of 5 mm min⁻¹ and loading rate of 13 kN min⁻¹. The cold crushing strength (CCS) was determined using the following relation:^{354,613}

$$\text{CCS} = F/A \quad (13)$$

where F is the fracture load and A is the face area of the cube.

Fig. 6(d–f) show the associated compressive stress vs. strain curves for immersion in fresh water, sea water, and acidic solution, respectively. It was observed that the maximum pressure increased with an increase in the soaking time after exposure to aqueous solution.³³⁷

5.4.4 Interlaminar/interfacial shear strength. Although several researchers have manufactured and studied different types of NFRCs due to their different properties, to date few experiments have been reported on the interfacial strength of JFRCS.^{7,155,158,437,446,590,614,615} Based on the ASTM D2344 (ref. 329, 339 and 550) and ASTM D3846 (ref. 387) standards, the ILSS test was conducted. The test was carried out using Instron 5569,³⁸⁷ Instron 3385,³⁶⁹ Instron 1195 (ref. 550) and Shimadzu AUTOGRAPH AG-G³³⁹ tensile testing machines with an attached load-cell. A narrow square cross-section beam of 45 mm was supplied at a rate of 1.3 mm min⁻¹ under 3-point bending, as shown in Fig. 7(a). When the loading cylinder produced a downward force, normal and transverse shear stress was exerted on the

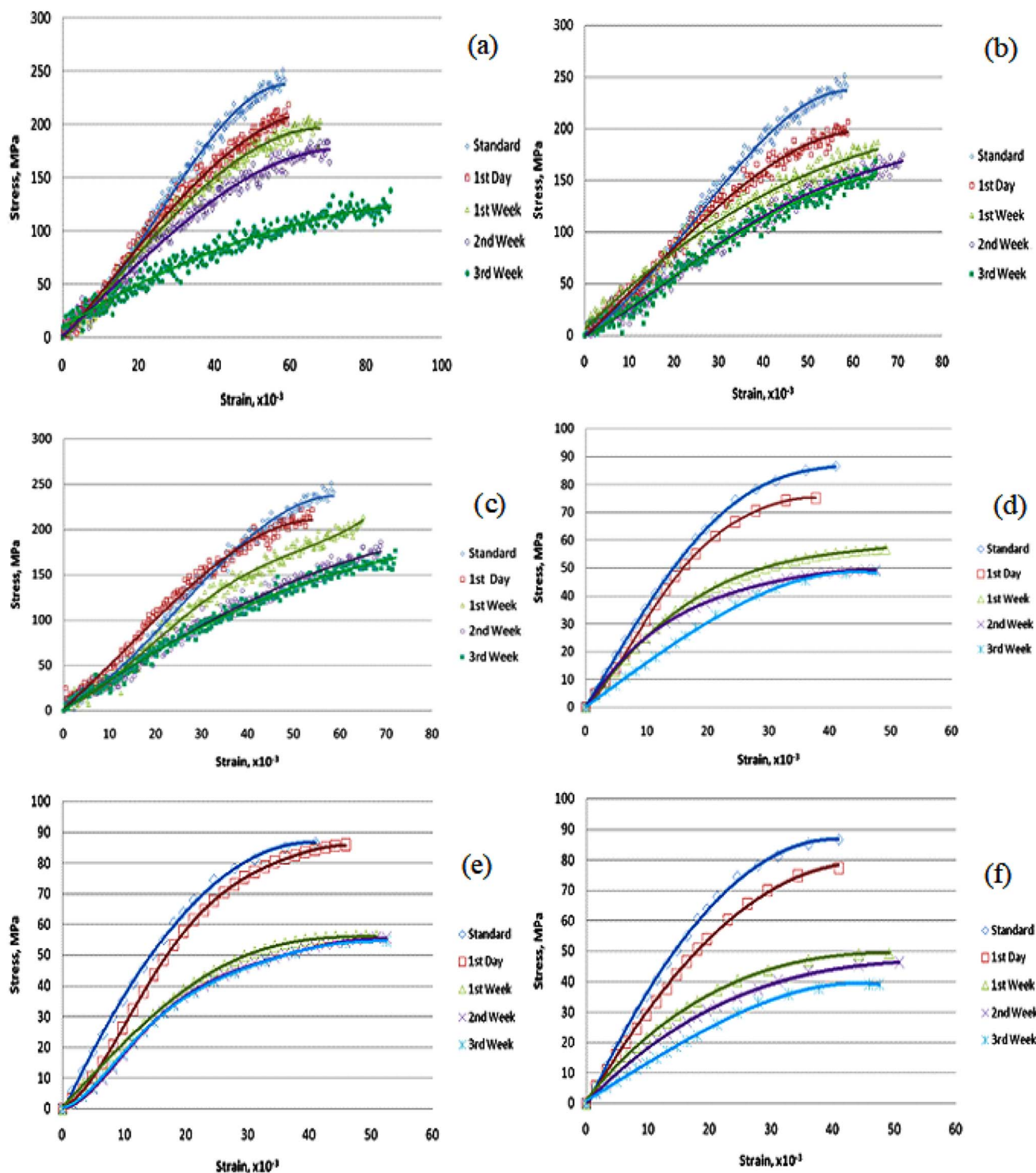


Fig. 6 Flexural testing of JFRCS: (a–c) typical flexural stress vs. strain curves and (d–f) compressive stress vs. strain curves after exposure to distilled water; sea water; and acidic solution, respectively.

specimen. The use of a short rod is believed to be sufficient for a reduction in stress due to bending, which results in an ILS fracture due to the cracking between the laminate on a horizontal plane.³²⁹

The shear properties of JFRCS were determined using the 3-point bending mode on a UTM at a velocity of 20 mm min^{-1} .

The measurements were performed on composite samples with a size of $30 \times 20 \text{ mm}$. The composite stress vs. strain curves were plotted and the breakage power, modulus, and elongation details were obtained.³⁶⁹ In another experiment, a UTM AG-G series was used with a cross-head speed of 1.3 mm min^{-1} . The test specimens had a size of $26.3 \times 6.4 \text{ mm}$.³³⁹ Using

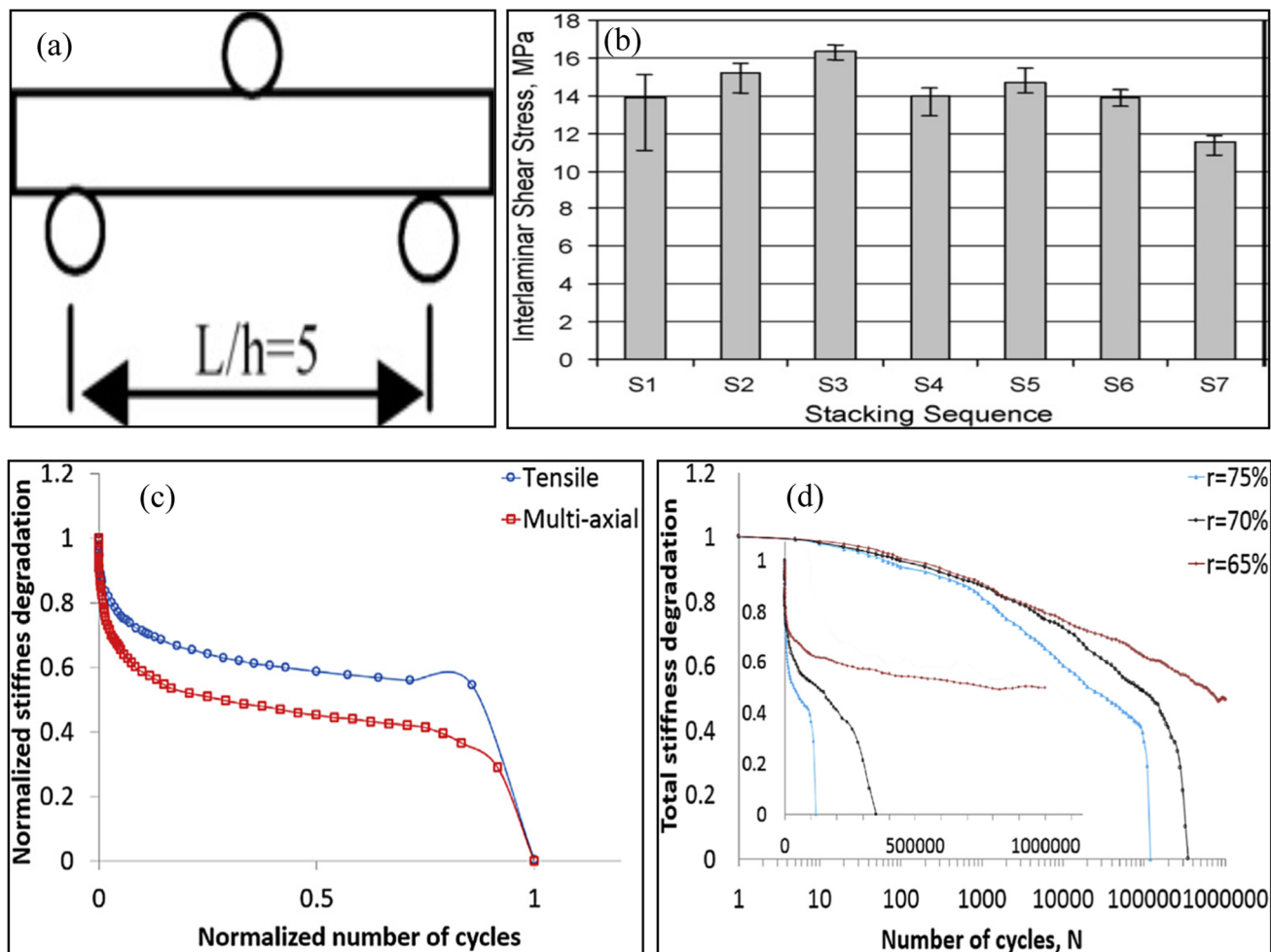


Fig. 7 (a) ILSS test configuration, (b) ILSS of JFRCs and (c and d) multi-axial fatigue test curves: normalized stiffness degradation and total stiffness degradation. Reproduced from ref. 293 with permission from Elsevier, 2016.

a micrometer,^{315,317} the specimen was set in the UTM. The experiment was conducted at a cross-head speed of 1.4 mm min^{-1} . The ILSS was estimated as the ratio of maximum load to the shear area.³⁸⁷ The IFSS of the JFRCs was tested using a micro-droplet testing process. The fibres were kept in a steel frame and the resin micro-droplets were shaped on each fibre using a tip pin. The resin micro-droplets were placed in a hot chamber at $220 \text{ }^\circ\text{C}$ and shaped in a circular cross-section due to the decrease surface strength. A specially built micrometer was used for testing the micro-droplet specimen. The laminates used for the various stacking sequences are shown in Fig. 7(b). ILS failure did not occur in the middle of the laminate, whereas shear mode failure occurred along the interface. The IFSS was determined using the pull-out force F , which was estimated using the following equation:

$$\tau = \frac{F}{\pi D_f L} \quad (14)$$

where D_f is the fibre diameter and L is the length of the embedded fibre in the matrix.⁵⁸

5.4.5 Fatigue strength. Despite the fatigue behavior of SFRCs, NFRCs^{517,616–619} has received little attention from

researchers. Identification of the deterioration of the stiffness of a material was used to determine its damage from fatigue, which was defined by measuring the shift in load encountered under constant pressure, showing the stiffness deterioration value of the material during each loading period.⁶²⁰ Quaresimin *et al.*⁶²¹ analyzed the fatigue strength of composite materials and highlighted the major impact of shear stress on the fatigue life of their components. They also noted that the load on a sample under multi-axial charge arose from the rebuilding of the non-uniform stress distribution on the sample. Amijima *et al.*⁶²² interpreted data from multi-directional tension-tension tests as the ratio of pure application results for each loading form and combined the ratios to represent the multi-axial loading results.

A uniaxial tension-tension-type fatigue test was conducted in compliance with ASTM D3479M standards using a servo-hydraulic system powered by a 25 kN unit. The experiments were performed in the frequency range of 0.5 to 2 Hz with a constant load. The frequency was set below 2 Hz to avoid heat, which leads to a shortened fatigue life, and the constant stress ratio was kept at 0.1. Cycles for failure were developed using software, which predicted the fatigue strength.³⁴⁸ Katogi *et al.*¹⁹⁸

tested the fatigue of unidirectional JFRCs under tensile loading. They recorded a tensile strength of around 61 MPa and 55% endurance limit. Katogi *et al.*⁵⁹⁶ conducted experiments on the influence of fibre type, shape, fibre wt%, surface modification, and matrix type on the damping capacity of a material under tension–tension fatigue tests. In the case of JFRCs, treatment with silane on the jute surface resulted in the need for higher loads given that progressive damage to was observed. Shah *et al.*⁵⁹⁷ examined the impact of fibre size, shape and strain ratio on the fatigue life of JFRCs and compared the results with that of other NFRCs. The values showed that the JFRCs displayed the highest static tensile and fatigue strength. JFRCs also survived millions of cycles at a stress ratio of 45% under fatigue charge. The fatigue properties of JFRCs were evaluated by Gassan and Bledzki¹⁵⁴ by measuring the strength and modulus of the composites as well as their damping factor. They found that the reinforcement with chemically modified fibres resulted in a lower fatigue value and damage to the composite propagation. Fatigue studies on JFRCs were performed by reinforcing untreated and chemically modified jute fibres.²³¹ The defects in the JFRCs were identified to play a major role. Higher concentrations of defects were used to absorb high impact energy and enhanced the fatigue strength of the composite.

Fig. 7(c) demonstrates the typical JFRC load degradation during tensile and multi-axial fatigue test cycles. The curve of the degradation of tensile fatigue stiffness value was normalized through application of load during the cycle. The curve of the deterioration of multi-axial fatigue stiffness was built by integrating the normalized deterioration of stiffness resulting from tensile and torsional loads.²⁹³ Fig. 7(d) shows the weakening of the rigidity arising from multi-axial fatigue testing. The same findings were also observed for these curves in the stiffness deterioration of the fatigue tests, where the curves correspond to 75% and 65% strain rates. The results indicated an incremental failure compared to the tensile loading. Failure of the JFRCs under axial loading occurred at 75% after 90 000 cycles and at 70% straining point after 260 000 cycles. The resistance limit for the composites under these loads was found at a stress level of 65%, where the JFRC lost 50% of its resistance to rigidity.²⁹³

5.5 Impact strength

The impact strength of JFRCs can be assessed by a range of testing procedures, for example, weight loss single or repeat test, weighted pendulum test, projectile test, explosion test, constant strain test, Split-Hopkinson bar test, and instrumented pendulum test.^{623–625} The specimen placed between two conical tapered cups was repeatedly hit by a hammer-type pendulum attached to a revolving circular disc. When the release knob set at a fixed angle was touched, it released the pendulum, allowing it to fall. The hanging pendulum was taken up again after each impact and falling was continued until the specimen was broken.³⁶¹

5.5.1 Charpy impact test. The Charpy impact test is carried out to assess the energy storing ability of materials against sudden loads. This test is performed by taking a material off

a spinning pendulum in one blow.⁵ Notched specimens are prepared according to the ASTM D6110,^{41,315–317,342} ASTM D256 (ref. 384) and ASTM A370 (ref. 5) standards. The absorbed energy indicates the impact load-carrying capacity of the material. A hammer shatters the test component and the energy consumed indicates the resistance of the material to shock loads.⁵ The specimens are prepared with different sizes including 55 × 10 × 10 mm,⁵ 79 × 10 × 4.1 mm (ref. 342) and 120 × 12 × 10 mm.³⁸⁴ A schematic illustration of the Charpy impact test scheme is illustrated in Fig. 8(a).³¹⁵ The energy absorption with respect to time is shown in Fig. 8(b).⁵⁵⁶ Charpy measurements of composite materials consisting of jute fibres are shown in Fig. 8(c). The low strength was recorded because of the fibre content in the composite structure, which could initiate crack propagation, and eventually possible fracture. The high fibre content also increased the chances of fibre agglomeration, which could create a stress concentration region that needs minimal energy to cause a crack.²⁹²

5.5.2 Izod impact test. The Izod impact test is conducted according to the ASTM D256,^{99,357,362,366,368,381,550} ASTM D4812,⁵⁴⁰ JIS K7110 (ref. 81) and DIN 53433 (ref. 56) standards using an Izod impact tester, Impactometer 6545 and Zwick test machine. The specimens have a cross-section of 65 × 12 × 3 mm,⁵⁴⁰ 60 × 10 × 2 mm (ref. 357) and 63.5 × 12.7 × 3 mm.³⁶² Test samples having a notch of 45° and depth of 2.5 mm were prepared for experiments.³⁶² Also, a specimen was tested using a 15 J hammer pendulum at an impact speed of 3.8 m s⁻¹.³⁵⁷

5.5.3 Low-velocity impact test. The low-velocity impact test is carried out using a weight-balancing machine, CEAST 9350,⁵⁵⁶ Instron/Ceas 9340 (ref. 350) and Zwick/Roell HIT230F model machines with a constant impactor mass of 23 kg at a height of 110 mm and diameter of 19.8 mm.²⁹⁷ The experiments are carried out according to the ASTM D3763 and ISO 6603-2:2000 standards and the impactor stroke occurs in the middle of the samples through the use of a support with a diameter of 40–75 mm. The energy stored is chosen to allow the calculation of the damaged area but without encouraging perforation for most specimens.^{350,556} The height of the drop is taken to produce 25 J of impact energy to fracture specimens with a thickness of 1–3 mm.

5.6 Fracture behaviour

5.6.1 Torsion testing. The torsion test is conducted on a rheometer using a torsion specimen with a rectangular shape, and the thickness is defined by the number of layers in the composite laminate. The torsion modulus was obtained at a strain rate of 0.002 through small amplitude torsion tests performed in the frequency range of 0.1–40 Hz.³⁸⁵ The complex shear modulus (G) was obtained from the dynamic frequency analysis using the following equation:⁶²⁶

$$G = E' + E'' \quad (15)$$

where E' and E'' are the storage and loss modulus, respectively.

5.6.2 Creep behavior. Creep is an interesting end-use factor for any composite material, especially in the case of JFRCs given that their properties are dependent on time and temperature. As

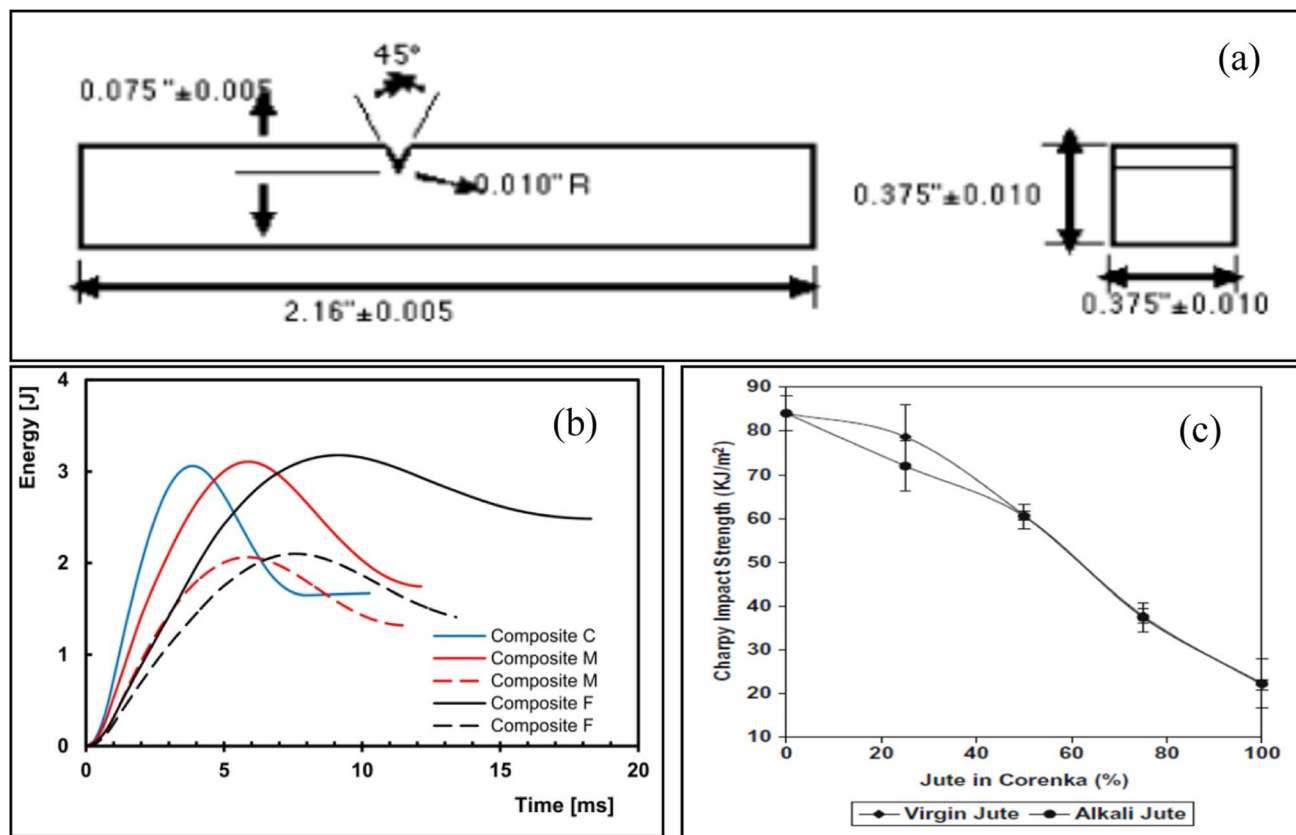


Fig. 8 Impact testing process of JFRCS: (a) schematic illustration of the Charpy impact test specimen according to ASTM standards. Reproduced from ref. 315 with permission from Springer, 2016. (b) Energy versus time during impact tests. Reproduced from ref. 556 with permission from Scielo, 2016. (c) Charpy impact strength. Reproduced from ref. 292 with permission from Elsevier, 2009.

a result of the viscoelasticity of the polymer matrix,⁶²⁷ temperature- and time-dependent degradation in strength and modulus can occur over time. Different methods of mathematical modeling were applied for the examination of the creep characteristics of composites.^{628–630} However, the details of the creep behaviors were barely considered due to the adhesive nature of the fibre/matrix.^{628,631,632} The experiments were carried out in the temperature range of 40–100 °C using a TA instruments Q800 DMA⁴¹² and PerkinElmer dynamic mechanical analyzer.⁶³³ The temperature range of the composites was chosen to be below their glass transition. The stress was applied for 30 min at the center of the sample after balancing at the optimal condition and the creep strain was measured. Then, the stress was measured, where the composites were identified as a linear viscoelastic region, confirming that the performance of the tests was linear.⁴¹² The experiments were performed using a 15 mm long specimen. The applied static stress for the creep was 4000 kPa, and then the material was allowed to recover below 4 kPa. Relatively low stress was applied during testing to confirm the linearity of the viscoelastic behavior.⁶³³ The experimental temperature was set at 50 °C. Details of the creep activity of JFRCS at different temperatures with and without fibre treatment are presented in Fig. 9. The curves in the figure indicate good agreement with the experimental results.⁴¹²

5.6.3 Fracture toughness. The measure of resistance against crack growth is called fracture toughness of

a material.⁶³⁴ The resistance of NFRCs to crack propagation or fracture toughness is based on various parameters including matrix toughness, additives, fibre characteristics, resin dispersion, fibre distribution, fibre orientation and duration after processing. Significant resilience of the fracture was observed to be influenced by the applied stress, fracture mode, matrix fractures, fibre debonding, fibre breakage, fibre pullout and bridging.^{635,636} Crack propagation depends on fibre debonding at the intersection of the fibre and matrix failure.^{94,637,638} In NFRCs, the stress applied is transmitted through their interface from the matrix to the fibre; therefore, the adhesion between the fibre/matrix plays a vital role in crack propagation.^{639–641}

5.7 Thermal properties

The thermal properties of NFRCs significantly limit their industrial applications due to the degradation of their fibre component at higher temperatures.⁴⁵ In NFRCs, thermal stability is their main property, which can be calculated using different thermal analyses. It is possible to apply thermal analysis to assess the moisture content and other volatile matter available in fibres that can induce the degradation of the fabricated composite materials. Most plant fibres appear to lose mass at about 160 °C.⁴⁹⁴ Among the important fibre constituents, lignin first starts to degrade at about 210 °C, whereas other constituents, mostly cellulose, are oxidized and deteriorated at

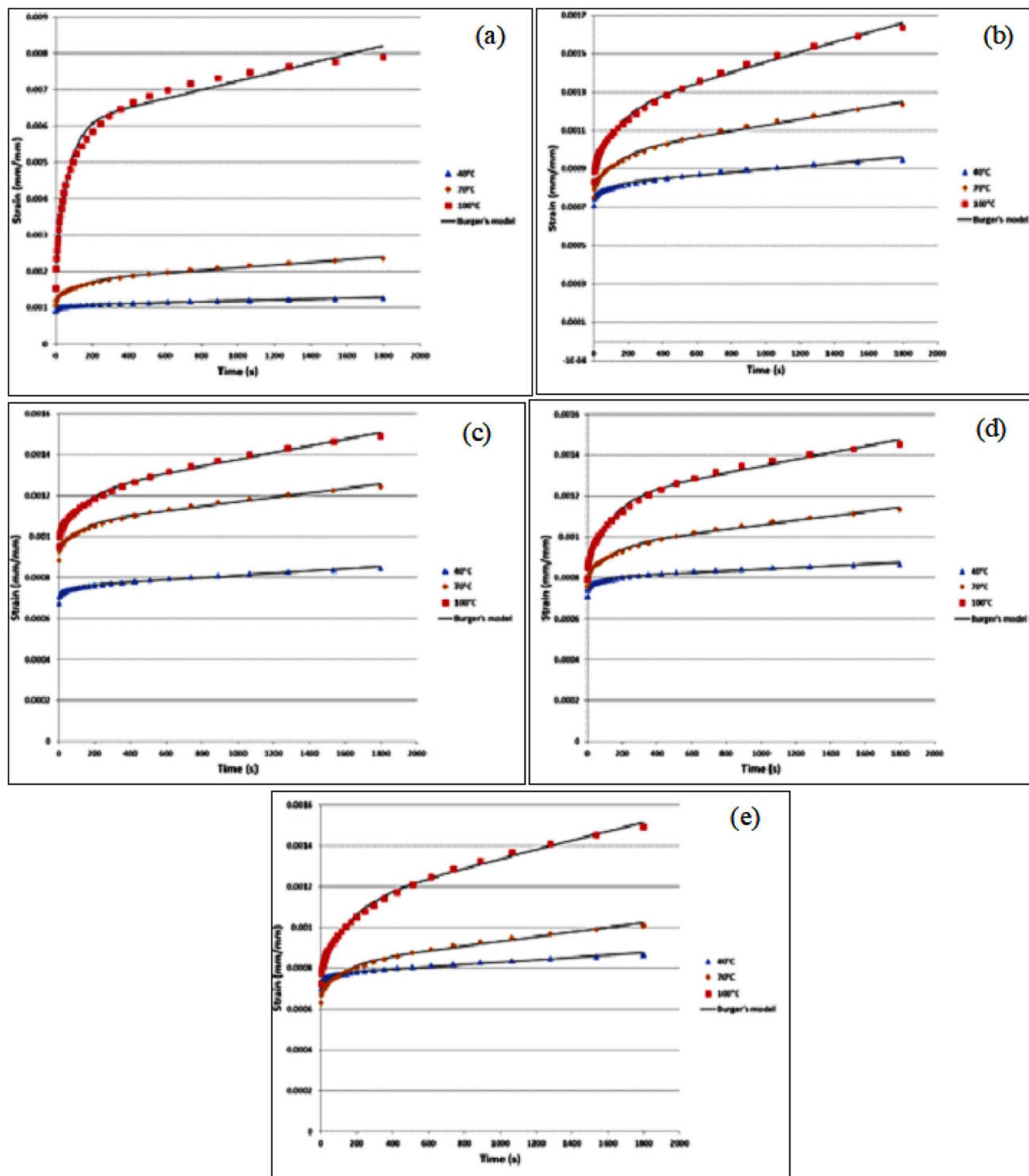


Fig. 9 Creep curves of (a) untreated, (b) enzyme-treated, (c) laser-treated, (d) ozone-treated, and (e) plasma-treated JFRCs. Reproduced from ref. 412 with permission from Elsevier, 2015.

elevated temperatures. Previous studies have demonstrated that the crystallinity index (CI) and cross-linking of cellulose have an influence on the thermal behavior of composites.⁶⁴²⁻⁶⁴⁴ Sinha and Rout⁶⁴⁵ reported that chemical surface modification increases the thermal stability of PFRCs. It was reported that the thermal resistance of JFRCs shows better stability than pure

polymers and other fibres. A decrease in resistance was observed with an increase in the content of fibres (wt%), which is consistent with the higher amount of polymer resin than jute fibres.⁵⁹ Also, the thermal properties of composites were investigated such as thermo-gravimetric analysis (TGA),^{56,59,61,353,354,356,368,376,560} differential scanning calorimetry

(DSC),^{353,356,363,376} dynamic mechanical analysis (DMA), heat deflection temperature (HDT),^{292,381} dynamic mechanical thermal analysis (DMTA),^{182,383,646–665} thermal aging,³¹¹ fire flow test³¹¹ by various researchers.

5.7.1 Thermal conductivity. Thermal conductivity, diffusivity, and specific heat capacity are the three major thermal-based properties of composites, which are required for effective heat transfer. Thermal conductivity is assessed according to the ASTM-E1530 standards using a Guarded Heat Flow Meter. The test sample with dimensions of 50 × 4 mm is mounted and a load of 10 psi applied. The heating element provided at the top and bottom maintains uniform heat transfer. A calorimeter is positioned to act as a transducer of the heat flow and the samples are tested in the temperature range of 30–130 °C. The difference in temperature is measured from the output of the heat flow transducer⁵⁴⁸ after thermal equilibrium is reached. The formula to determine one-dimensional heat conduction is as follows:

$$\text{Heat flux } q = \frac{k(T_1 - T_2)}{D} \quad (16)$$

where k is the thermal conductivity, $T_1 - T_2$ is the temperature difference, and D is the sample thickness. The thermal resistance and conductivity of composites can be calculated using the following equations:

$$\text{Resistance } R = \frac{(T_1 - T_2)}{q} \quad (17)$$

$$\text{Thermal conductivity } k = \frac{D}{R} \quad (18)$$

The specific heat capacity of the composites is determined by differential scanning calorimetry at 10 °C min⁻¹ according to the ASTM E1269-11 standards.⁵⁴⁸ Thermal diffusivity is determined using the following equation:

$$\text{Thermal diffusivity } \alpha = \frac{k}{\rho C_p} \quad (19)$$

where k , ρ and C_p are the conductivity, density and specific heat capacity, respectively.

5.7.2 TGA. TGA is conducted to assess the thermal stability and degradation profiles of composites. The thermogravimetric behavior of plant fibres directly depends on their chemical constituents.⁴⁵ This method can be employed to characterize composites that display weight loss or gain due to dehydration or decomposition.³⁸¹ The TGA of JFRCs was conducted using TG209F1 NETZSCH,^{353,354} Q50 TGA,³⁶⁸ Thermal Analyst 2100,⁶¹ TA Q500,^{59,356} SDT Q600,³¹⁹ Mettler Toledo,³⁴⁵ Shimadzu DSC 50,³⁷⁹ Universal V1.12E⁵⁶ and PerkinElmer^{346,362,376} instruments according to the ASTM E1131 standards³⁴⁵ in a complex O₂/N₂ atmosphere in the temperature range of 30–1000 °C at a heating rate of 10 °C min⁻¹.^{319,346,353,354,376,379} TGA was conducted under an N₂ atmosphere with a sample of weight 10–65 mg. The specimens were kept in a furnace and processing was done at the rate of 5–20 °C min⁻¹ in the temperature range of 0–900 °C (ref. 56, 59, 356 and 362) and the corresponding weight loss was

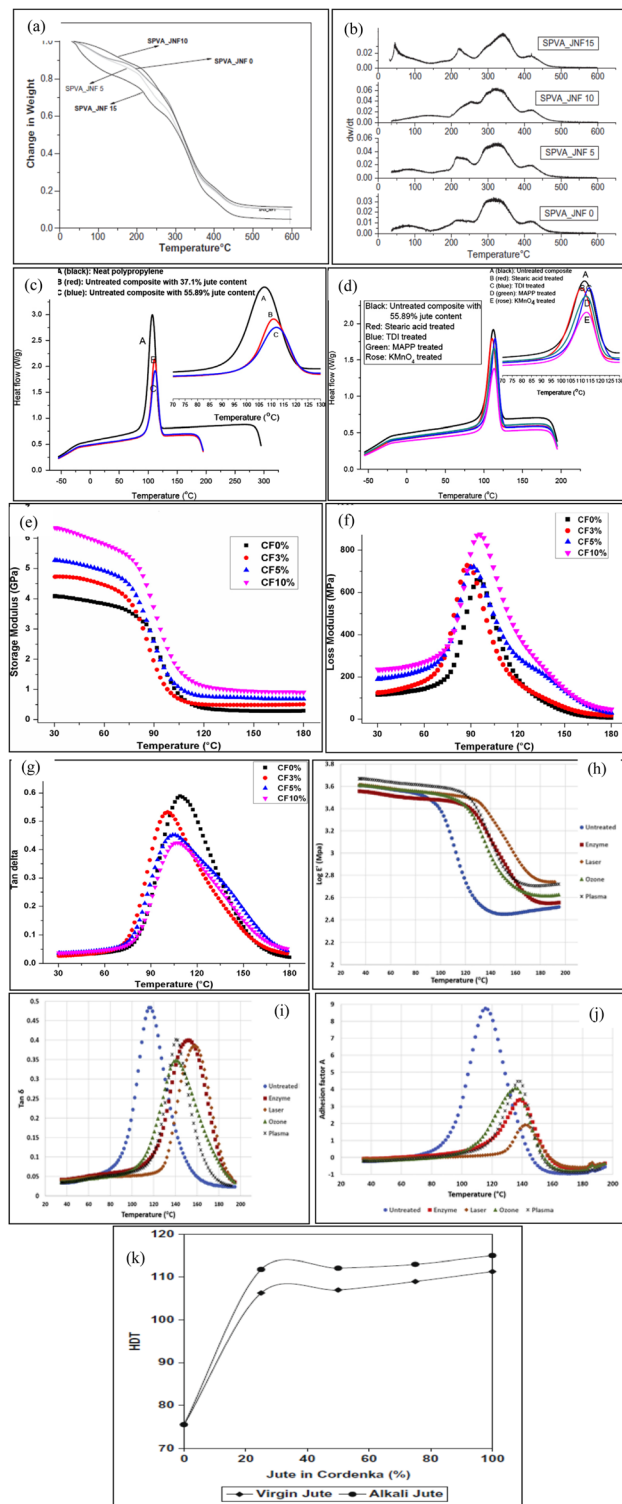


Fig. 10 Characterization of JFRCs: (a) TGA, (b) DTGA thermograms, (c) effect of fibre content on DSC, (d) effect of chemical treatment on DSC, (e–g) DMA curves of E' , E'' , and $\tan \delta$, (h–j) dependence of E' , $\tan \delta$, and adhesion factor on temperature, and (k) HDT curves, respectively.

recorded. The degradation nature of the JFRCs is presented in Fig. 10(a and b). The presence of jute fibres had a significant effect on the thermal degradation behavior, which shifted from

315 °C to 322 °C. In another sample, the degradation process was delayed, and the residue left at 600 °C was also higher than that of the other bio-composites, indicating the higher stability of jute fibre films than other combinations.³⁷⁶

5.7.3 DSC analysis. DSC is conducted to analyze the melting properties and crystallization nature of composites.³⁵⁶ The instruments employed include a DSC200PC NETZSCH,³⁵³ PerkinElmer Diamond DSC,³⁷⁶ DSC7 PerkinElmer,³⁶³ DSC8000 PerkinElmer,³⁴⁶ TGA/DSC1 STAR, Mettler Toledo,³⁴⁵ Shimadzu DSC50 (ref. 379) and TA Q1000 (ref. 356) for the DSC analysis of JFRC samples. DSC is performed according to the ASTM D3418 standards³⁴⁵ under an N₂ atmosphere in the temperature range of -50 °C and 600 °C at the heating rate of 5–10 °C min⁻¹. Also, 5–10 mg sample in aluminum discs is covered and fed in the calorimeter heating equipment.^{353,376,379} The heat flow is measured using indium before testing. DSC curves are obtained from the thermal cycle, which offer reliable results in terms of the melting (T_m) and crystallization (T_c) temperature.^{356,363}

Fig. 10(c and d) show the impact of fibre quantity and surface modifications on the DSC curves for the crystallization of a jute/PP composite. In the case of PP, crystallization occurred at 107 °C, while the temperature of the onset of crystallization was 118 °C. When jute fibres were incorporated in the resin, the crystallization temperature and onset crystallization of the composite increased to 111 °C and 119 °C, respectively. In the case of the untreated jute fibre composite, the crystallization temperature increased to 112.5 °C. This increased impact was due to the addition of the jute fibres, which acted as reinforcing agents and induced or favored the process of crystallization, leading to an increase in the crystallization temperature. The jute fibre reinforcement also favoured the formation of a transcrystalline layer on the surface, which increased the crystallization temperature, thereby supporting the crystallization. This was preceded by a decrease in the crystallization (DHC) value.³⁵⁶

5.7.4 DMA. DMA is performed over a broad temperature range for the determination of the viscoelastic properties, and particularly the glass transition temperature (T_g).³⁶² DMA is also employed for the evaluation of the relative stiffness and the damping properties of composites. DMA tests are conducted to track the storage and loss moduli and loss factor as a function of temperature. The test is conducted using a Rheometrics RDA-III,³⁶² Perkin DMA8000,^{515,556} DMA2980,³⁵⁸ DMA983,⁵⁴⁴ Q800 DMA,^{341,368,369,546} Mettler Toledo⁸³ and Eplexor® 150N⁶²⁸ according to the DIN 53457,⁵⁵⁶ ASTM D4065 (ref. 515) and ASTM D4065-01 (ref. 443) standards by various researchers. In the literature, DMA was performed with specimens possessing dimensions of 27.4 × 3.1 × 3 mm,³⁶² 50 × 10 × 3 mm,⁶²⁸ 60 × 6 × 3 mm (ref. 544) and 28 × 7 × 3 mm.⁵¹⁵ The temperature was set in the range of -120 °C and 200 °C at the heating rate of 2–3 °C min⁻¹ under an N₂ flow.^{546,556} Static and dynamic strain rates in the range of 0.05% to 0.2% (ref. 83, 358, 362, 412, 515, 544 and 628) were used at 3 °C min⁻¹ and 5 Hz.^{341,368}

The storage modulus (E'), loss modulus (E'') and $\tan \delta$ of JFRC specimens were evaluated by DMA at the heating rate of 5 °C min⁻¹ in the temperature range of 30 °C and 120 °C at a frequency of 1 Hz and 15 μm .⁴⁴³ E'' is the viscous component contribution of the material, representing the energy dissipated

by the device as heat during the process due to viscous movements in the material. E' was found to be high at room temperature, decreasing with an increase in temperature (Fig. 10(e)). In the case of the treated composites, E' was observed to be higher than the untreated composite. This was attributed to the stronger inter-laminar fibre/matrix bonding and chemical treatment. The higher E' with an increase in the content of nano-clay was observed to be an indicator of the higher crystallinity and brittle nature of the composite. A slight bump was observed at around 50 °C for the samples without nano-clay. The specimen entered the cold crystallization temperature range during the cycle, where there was an increase in the CI and a decrease in the E' value.⁶¹⁰

The shift in E' of different JFRCs is depicted in Fig. 10(e), showing that the E' values of all the specimens are almost the same, which decreased with an increase in temperature due to the change from the glassy to rough state. However, there was an increase in E' with an increase in the nano-cellulose content in both regions, which showed the superior strengthening effects of jute fibres compared against the above-mentioned temperature. In the glassy area, the constituents were highly immovable, compact, packed tightly and had strong intermolecular forces, which resulted in a high E' ; however, the intermolecular forces decreased with an increase in temperature and the constituents became mobile and lost their compact structure, resulting in a loss of stiffness, and thus E' . The uncoated JFRCs had the lowest E' value in the given temperature range, as shown in Fig. 10(e). The JFRC had an E' value of 4.05 GPa at 30 °C, whereas the composites with other combinations showed an average value of 5.5 GPa, resulting in a 16% to 56% increase in E' . In addition, E' increased from 0.3 GPa to 0.94 GPa at 150 °C, representing a 71% to 235% increase respectively. This shows the increase in the stiffness of the reinforcement in the rough surface region with an increase in the concentration of nano-cellulose above the content of jute fibres.⁵⁴⁶

Fig. 10(f) presents the E'' value as a function of temperature, which increased, and then decreased. The highest heat dissipation occurred at the highest E'' value is higher, reflecting the T_g . The sudden increase in E'' suggests the rapid increase in the free movement of the polymeric chains at higher temperatures, which is not possible at lower temperatures. This figure also shows an increase in E'' with an increase in the concentration of the nano-cellulose coating, reflecting the maximum energy dissipation with an increase in wear rate.⁵⁴⁶ The $\tan \delta$ of the coated JFRC is shown in Fig. 10(g), where peak $\tan \delta$ value was observed for high energy dissipation, while a decrease in the $\tan \delta$ peak was seen with an increase in the nano-cellulose concentration. The maximum peak of 0.59 for the composite was reduced to 0.42, reflecting a decrease of 40%, which suggests strong inter-molecular and physical interactions. This leads to excellent interfacial restrictions and low energy dissipation, given that E' was affected more by the increase in nano-cellulose concentration than E'' . Also, the width of the $\tan \delta$ peak was wider for the nano-cellulose-coated JFRCs. The increase in the width indicated an increase in the interface volume, reducing the movability of polymer particles in the

device and maximum segments following an increase in the content of nano-cellulose in the JFRCs.⁵⁴⁶

The E' of the JFRCs as a function of temperature is presented in Fig. 10(h), which shows a steady decrease in the E' of all the JFRCs. In the glassy region, a small variation in E' was seen, with the treated composites having higher values in the rub environment, which could be attributed to improvement in the fibre/matrix bonding at the interface, reduced molecular weight of the polymer particles, and excellent reinforcement of the surface-modified fibres. This resulted in an improvement in the thermal stability and strength of the material at higher temperatures.⁴¹² The $\tan \delta$ of JFRCs as a function of temperature is presented in Fig. 10(i). The unmodified JFRC was observed to have a higher peak than the surface-modified JFRCs. The tendency of the composites having weak fibre/matrix connectivity to dissipate high energy was seen compared to the composites with strong inter-laminar connectivity, *i.e.*, weak interface bonding. This resulted in higher damping,^{666,667} indicating the phenomenon of molecular relaxation in the interfacial region of the composites.⁴¹² Fig. 10(j) shows the low adhesion of the surface-treated JFRCs compared to the untreated JFRC, which shows an enhancement in the fibre-matrix bonding.⁴¹²

5.7.5 DMTA. DMTA was performed on SFRCs to investigate the effect of incorporating fillers, surface modifiers, chemical agents, compatibilizers, *etc.* on the fibre/matrix interface.^{646–657} Similar experiments were performed for natural fibres, given that there were no comprehensive studies on reinforcing filler.^{658–661} DMTA experiments were performed in the temperature range of 0 °C to 200 °C at a heating rate of 3 °C min⁻¹.³⁸³ The size of the specimen was 55 × 10 × 2 mm (ref. 182) and 53 × 13 × 1.5 mm (ref. 383) at a frequency of 1 Hz, and a relative strain of 0.1%. Ray *et al.*⁴⁴¹ examined the DMT characteristics of vinyl ester-based JFRCs and observed a change in T_g . Some researchers^{662,663} reported a similar phenomenon for compatible jute fibres and epoxy resin. Some other authors also reported the presence of a secondary transition at a temperature higher than T_g .^{664,665} This increase was thought to be correlated with the micro-Brownian movement of the immobilized polymeric molecules in close proximity to the outer surface. According to the test, the E' and $\tan \delta$ values are calculated at different temperatures and T_g values of the DMTA system.¹⁸²

5.7.6 HDT. The HDT test, which is also known as the heat distortion temperature test, is carried out according to the ASTM D648 (ref. 381) and DIN EN ISO75 (ref. 292) standards. Fig. 10(k) shows the HDT of the composites in relation of the fractions of jute fibre. The HDT value for 25% jute fibre increased to above 100 °C, and simultaneously, the impact energy loss was around 6%, retaining a value of 9 kJ m⁻². The enhancement in HDT was small for a higher concentration of jute, while the force of impact was appreciable. Jute fibres decreased the HDT to 22 kJ m⁻² due to the beneficial impact of their internal structure and mercerization on the HDT due to the enhanced properties.²⁹²

5.7.7 Thermal ageing. The thermal ageing of composites is due to its increasing use for structural applications and changes in environmental conditions. Micro-cracking is observed, which

is often a damaging characteristic that can be induced in polymers exposed to cryogenic conditions. Tensile strength tests were performed on specimens kept at 75 °C for 10 h and subjected to deep freezing at -75 °C for 6. Delamination growth, matrix cracking, and fibre failure are the most common modes of damage in thermal ageing. The cryogenic condition introduced matrix cracking and/or debonding at the interfaces. The fibre/matrix bonding was weak during cryogenic conditioning. At low temperatures, the first type of damage was usually matrix micro- and inter-laminar cracks. This is one of the reasons for the decrease in the tensile strength of composites when exposed to low heat. It is well known that heat conditioning at higher temperatures provides better bonding, and therefore enhanced mechanical properties are achieved, given that the fibre cross-linking is very high during heating at higher temperatures, increasing the mechanical properties of the composites. Both NFRCs and SFRCs exhibit similar behavior in thermal ageing processes.³¹¹

5.7.8 Fire retardant test. The fire-retardant characteristics of composites would easily diminish due to their ignitability. This is important for a reduction in the combustible properties of composites.⁵⁵⁴ In this case, a flow test is conducted according to the ASTM D635 standard and the rate of burning is evaluated from the test. Fire retardants such as Al₂O₃, MgO, and H₃PO₄ were used for enhancing the fire-retardant characteristics of JFRCs.⁵⁵⁴ NFRCs showed low burning rates compared to SFRCs; hence, the fire-retardant properties of NFRCs were observed to be much better than SFRCs. The diameter of natural fibres is higher than that of any synthetic fibre; therefore, the composite layer acts as an insulating material, thus slowing down the rate of burning of the materials.³¹¹

5.8 Acoustical properties

An acoustic emission (AE) test was conducted on JFRCs by positioning sensors at both ends of the specimen to allow linear localization of the acoustic signals. The experiments were conducted based on the ASTM E976 standard.³³⁶ The detection point was localized between the sensors to avoid disturbances from the detection of the acoustic signals. The sensors used for the AE resonated at a frequency of 150 kHz. The sensors were mounted on the specimen surface to allow linear localization, while the two other sensors acted as guard sensors to distinguish AE and noise signals.⁵⁴² Implementation of the fragmentation approach with non-destructive AE helped to determine the micro-failure mechanism of the specimen. Fig. 11 displays the AE signal strength and energy of untreated, alkaline, and silane-treated JFRCs. The AE signals indicated that the energy was very low before the boiling water test due to the occurrence of a micro-failure cycle at the swelled fibrils after the boiling water test. With an increase in the AE energy, its amplitude and signal strength were seen as a result of the chemical surface modification of the jute fibres. The results obtained for silane-modified jute fibre after the boiling water test were compared to that of other combinations. The AE signal strength and its amplitude were high compared to the other combinations after the boiling water test.⁶¹

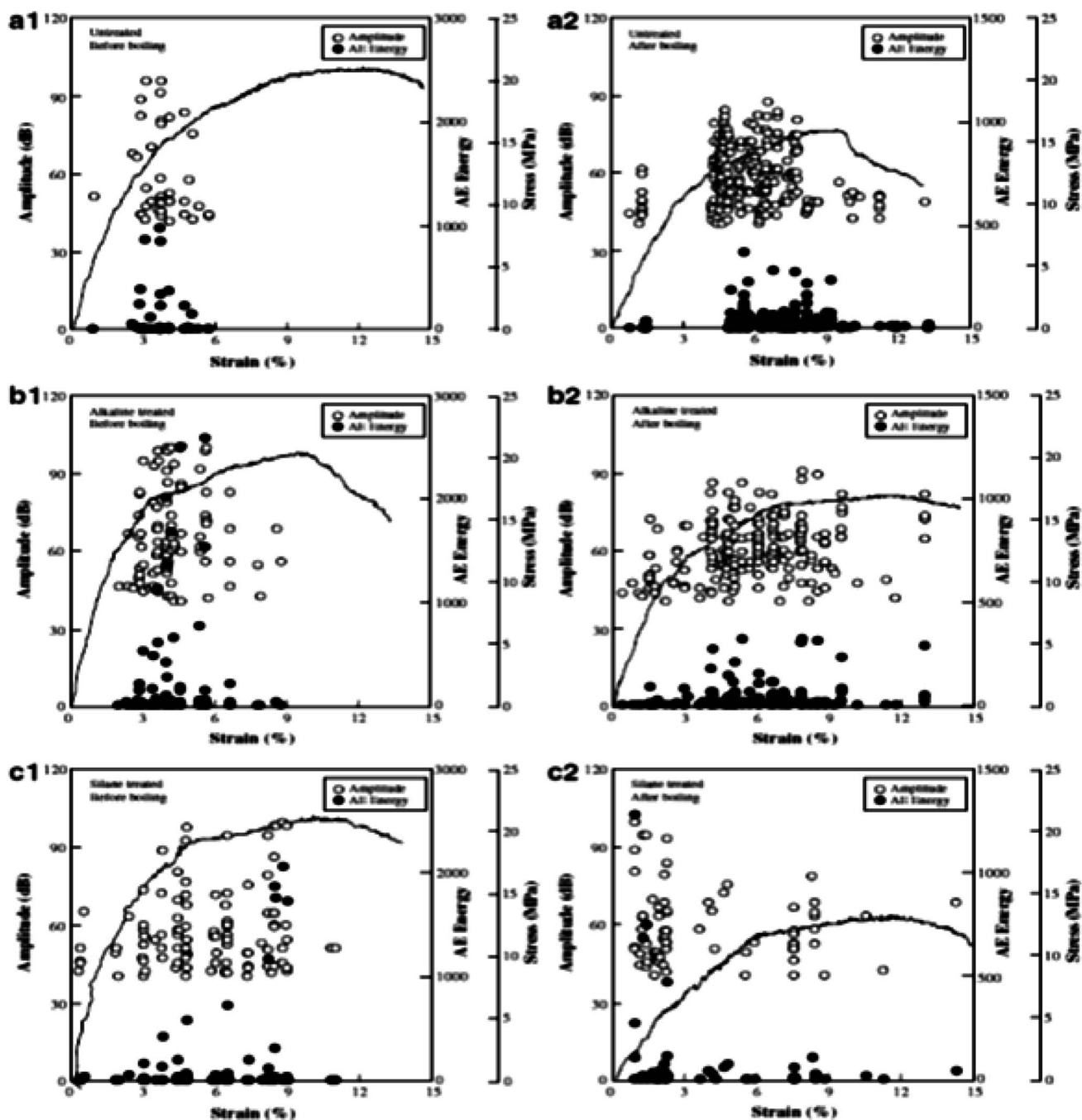


Fig. 11 AE amplitude of untreated and chemically treated JFRCS. Reproduced from ref. 61 with permission from Elsevier, 2008.

5.9 Wear properties

The objective of the Green Tribology Congress held in Japan in 2009 urged materials science researchers to focus on reducing the dependency on synthetic materials and minimizing the wastage of materials due to wear.^{16,668} In this case, PFRCs can be used as components, which exhibit good tribological properties in many cases due to their effect on wear and tear, depending on the material selection and processing conditions.^{669–674} An abrasive test was conducted to study of the strength of the bonding at the interface of composites on their performance

through use of a pin-on-disc apparatus according to the ASTM G65 standard.⁶⁷⁵ Wear test specimens with dimensions of $3 \times 3 \times 25$ mm were abraded against a spinning disc, on which an abrasive paper was pasted. The experiments were carried out in various cycles with the sliding distance corresponding to 6–200 m by various researchers. A sliding speed of 0.418 m s^{-1} was used, and the weight loss was calculated at the end of each series of 100 cycles using a physical balance.³⁶⁸

Abrasive powder was fed into the contact surface, and then examined. The sample was loaded against the wheel using

a lever and fine sand particles were added.⁵⁴⁹ The disc rotated with the aid of a DC motor at a speed of 600 rpm. The disc surface acted as a counterpart and was finished with a roughness of 0.6–0.7 μm by abrasion from a 1200 SiC layer. Measurement of the abrasion loss was performed using an electronic weighing machine with a precision of ± 0.001 mg.⁶⁷⁶ In another experimental study, an abrasion tester was used for two-body abrasive wear tests.⁶⁷⁷ A $45 \times 35 \times 5$ mm specimen was made to slide against a spinning drum, on which an abrasive sheet was placed, and readings were taken for four period sets. A sliding velocity of 2.56 m min^{-1} was applied, and the abrasion loss was estimated when 100 cycles per set were completed.³⁷⁵ The pre- and post-experiment weight of the specimen was reported *via* an electronic mini-balance that had a minimum count of 0.01 mg.³⁸⁹ The wear rate of the specimen was calculated using the following equation:

$$W = V/\rho D \text{ (mm}^3 \text{ mm}^{-1}\text{)} \quad (20)$$

where V is the weight loss, ρ is the density of the material and D is the sliding distance. The specific wear rate was as follows:

$$\text{Specific wear rate}(K_s) = \frac{\Delta m}{\rho L F_n} \text{ (mm}^3 \text{ mm}^{-1}\text{)} \quad (21)$$

where Δm is the mass loss, ρ is the density, L is the sliding distance, and F_n is the applied load.

The wear rate *vs.* sliding velocity of the JFRCs is presented in Fig. 12(a), showing an increase in the common wear rate of the JFRCs with an increase in the sliding velocity. A decrease in the wear loss was observed with an increase in the sliding speed due to the surface contact between the specimen and wheel.³²⁷ The JFRC with 36 wt% fibre loading showed lower wear loss than other combinations. The effect of load *vs.* specific wear rate is presented in Fig. 12(b), which shows that under all operating conditions of the plain epoxy specimen, it displayed a higher specific wear than the other specimens, increasing with an increase in the content of fibres (wt%). However, the impact of load on the wear rate of the JFRCs showed an irregular trend.

The same 36 wt% composite exhibited better wear characteristics than the other samples under the standard load conditions, where the minimum specific wear rate was obtained. The wear rate of the JFRCs was observed to be high at a specific load due to the fact that the resin was supported by a low volume of fibres, and the depth penetration of the abrasive particles could also be a reason for this. Ploughing or wedge forming could be the potential wear mechanism for reduced wear loss and addition of the fibre to the resin allowed a decrease in losses due to wear.³¹³ The 36 wt% fibre-loaded composite showed the minimum wear rate at various sliding speeds and regular load under stable conditions due to the strong fibre–matrix interfacial bonding. The average wear rate of the 48 wt% composite was higher than that of the 36 wt% sample due to the inadequate fibre impregnation in the former, resulting in poor interfacial bonding.

5.10 Water/moisture absorption properties

The major drawback of NFRCs is their poor resistance to moisture absorption.⁶⁷⁸ It is known that these composites are sensitive to moisture, which results in the swelling of their surface.⁶⁷⁹ This is even more challenging in plant fibres, which have low water-absorption resistance^{456,680–683} due to their hydrophilic characteristics and chemical composition, causing them to retain hydrogen water molecules.^{132,160} The intensity of moisture absorption depends on the fibre surface, substrate, fibre/matrix interface, relative humidity and surrounding temperature. The hybridization of plant fibres with less water-intake conventional fibres has an effective impact on the resulting composite materials,^{84,684–686} thereby allowing a reasonable performance. Factors such as fibre volume, reinforcing type, fibre composition, processing temperature, exposed surface area, polarity, degree of cross-linking and crystallinity affect their water-absorption nature.^{9,687–690}

Moisture absorption tests on JFRCs were conducted according to the ASTM D570-98,^{381,548,550,556,574,691} ASTM D570-99,^{340,342,346} ASTM D570,³⁷⁴ ASTM D2495-07,⁴⁴³ and IS:2380-77

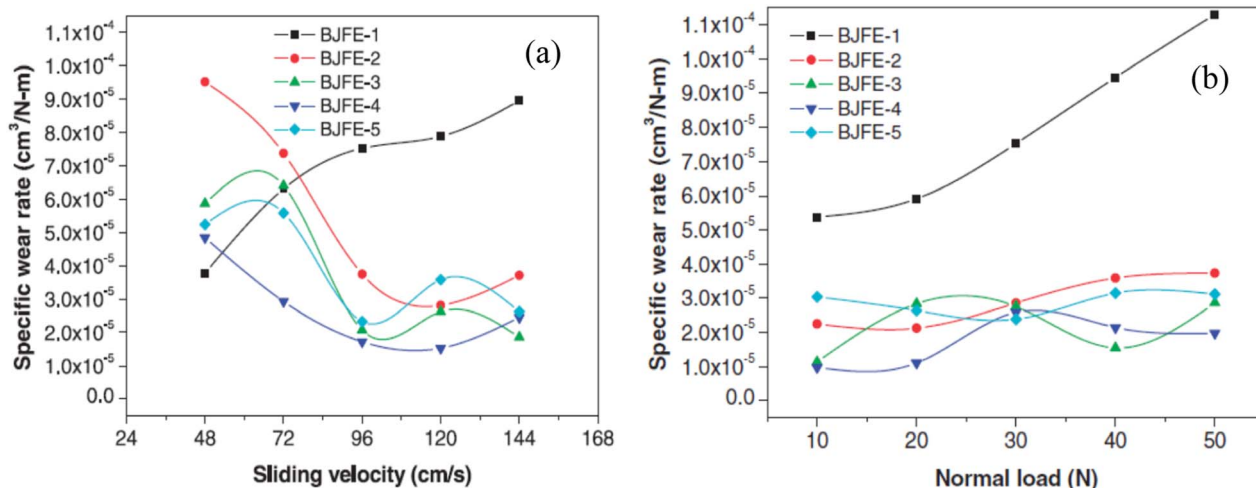


Fig. 12 Wear properties of JFRCs (a and b) effect of sliding velocity and normal load on the specific wear rate.

(ref. 608) standards. The samples were prepared with dimensions of $100 \times 13 \times 3$ mm,⁴⁴³ $40 \times 10 \times 0.5$ mm,³⁴⁰ $39 \times 10 \times 4.1$ mm (ref. 342) and $80 \times 80 \times 2$ mm (ref. 59) for water absorption studies. The samples were dried for 5 h at 105°C and their weight recorded. Distilled water was poured into a desiccator for the creation a humid atmosphere and the humidity was found to be 98% after 12 h. Then, the samples were placed in the desiccator and their weight recorded at regular intervals.⁴⁴³ Subsequently, the specimens were dried at 105°C , cooled with silica gel in a desiccator, and their weight recorded. Next, they were soaked in distilled water for 2 h. Afterwards, the excess water on their surface was removed using a soft cotton cloth. Finally, the weight the specimens was recorded.³⁴²

In another study, the specimens were first dried at 70°C and their weight recorded using a weighing balance at a precision of ± 0.01 g. Then, the specimens were immersed in water and left

for a day. They were taken out at frequent intervals and their wet weight recorded.³⁴⁰ In another study, jute fibre was cured for 4 h at 99°C , and then its moisture absorption was measured at 23°C .¹⁰⁵ The weight gain of the composite specimens submerged in water at 23°C was taken for water absorption.³⁸¹ The JFRCs were subjected to water absorption experiments through immersion in sea water, clean water, and acidic water. Different parameters were investigated including the diffusion coefficient and overall moisture quantity using the absorption curves.³³⁷ The strength in each case was found to be inversely proportional to the percentage water absorption.⁵⁶⁰ A water absorption analysis was performed for jute fibres and for the JFRCs. The samples were kept in water for 5 h at 23°C , taken out and their weights recorded.³¹⁷ The difference in weight was taken for the absorption of moisture.⁵⁷⁴ The moisture content or percentage increase in weight was determined as follows:^{315,336}

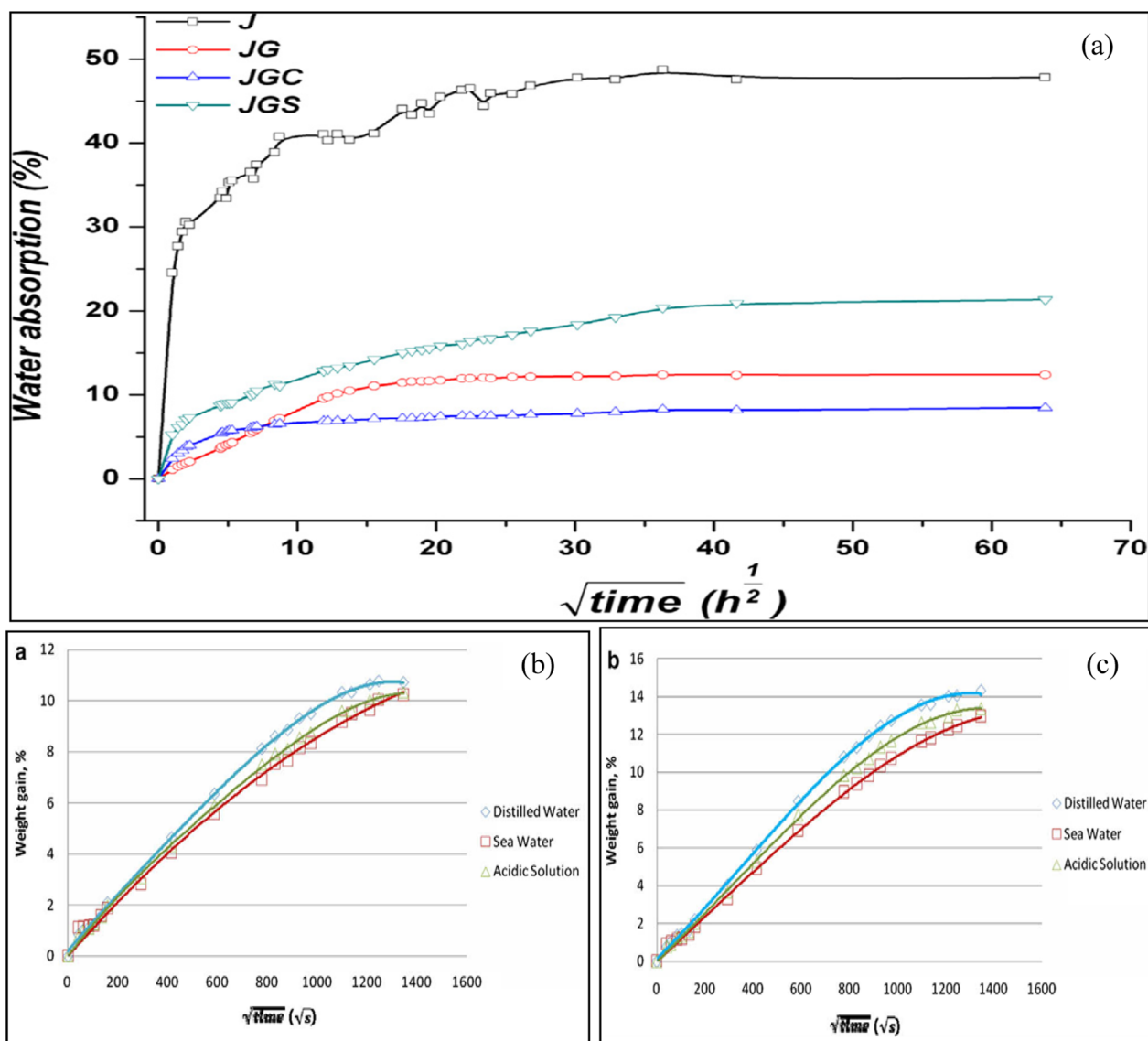


Fig. 13 (a) Water absorption curves of JFRCs and (b and c) weight gain of JFRCs after exposure to distilled water, sea water and acidic solution after flexural and compression tests, respectively.

$$\text{Water absorption (\%)} = (W_1 - W_2/W_2) \times 100 \quad (22)$$

where W_1 is the weight after immersion and W_2 is initial weight of the specimen. The Fickian diffusion coefficient was determined using the following equation:

$$D = \pi \left(\frac{kh}{4M_m} \right)^2 \quad (23)$$

where D is the diffusion coefficient, M_m is the moisture content, h is the thickness of the laminate and k is the slope of the plot of $M(t)$ versus $t^{1/2}$, as determined using the following equation:

$$k = \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \quad (24)$$

The corrected diffusion coefficient (D_c) is expressed as:

$$D_c = D \left(1 + \frac{h}{l} + \frac{h}{n} \right)^{-2} \quad (25)$$

where h is the thickness; l is the length and n is the width of the specimen.

The curves for the absorption of water by the JFRC are shown in Fig. 13(a), displaying the proportion of water consumed versus immersion time. It can be observed that the water-absorbing nature was very quick until reaching a saturation point, after which the absorption rate showed a decreasing trend.³³⁶ The water absorption curves for the JFRCs reported by other researchers are shown in Fig. 13(b) and (c). The samples were submerged in normal water, water with pH 8, and acidic water with pH 3. The curves showed an increase in absorbed water content following an increase in immersion time. The JFRCs followed pseudo-Fickian behaviour, where the gain in weight after the initial increase never reached equilibrium.⁶⁹² These composites are known to have a higher coefficient of diffusion and overall moisture content if submerged in purified

water, followed by pH 3 acidic water. In contrast with sterile water, a decrease in the value of D and a decrease in moisture content were observed when the specimen was kept in water with pH 8 and pH 3 acidic solution. This is due to the presence of large molecules in pH 8 water, which delay the diffusion time, resulting in the minimum kinetic absorption parameter.⁶⁹³ Acidic water at pH 3 did not result in greater corrosion than purified water.⁶⁹⁴

5.11 FEA

Modeling and simulation are important processes used for the enhancement of design and analysis of the performance of NFRCs.¹⁹ A significant numerical and confirmatory approach is numerical analysis or FEA for the optimal solution to a variety of design problems.³³⁸ This was performed to determine the natural frequency of JFRCs^{553,695} under the pure opening mode, mixed mode, and pure shearing mode fracture power. This analysis was carried out using the ANSYS Work-bench commercial FEA programme. Rizal *et al.*⁶⁹⁵ performed FEA on JFRC plates and found that the frequency of their vibrations is critical for the identification of their Young's modulus, rigidity modulus and Poisson's ratio. The vibration modes and the natural frequency of the JFRCs are presented in Fig. 14,⁶⁹⁵ showing the natural frequency of 297.77 Hz and 222.25 Hz in the flexural and torsional mode, respectively.

ANSYS was used to test the thermal conductivity of the composites. Coding was done in APDL (ANSYS Programming Design Language), which delivered the output for the conductive heat transfer of the composites. 3D cylinders-in-cube physical models were used for the simulation of the unit cells of the composites at various contents of the fibres (wt%). A one-axis aligned orthogonal coordinate scheme with the orientation of the fibre was used in the study of the micro-mechanics of fibre-reinforced materials. The diameter of the fibre was

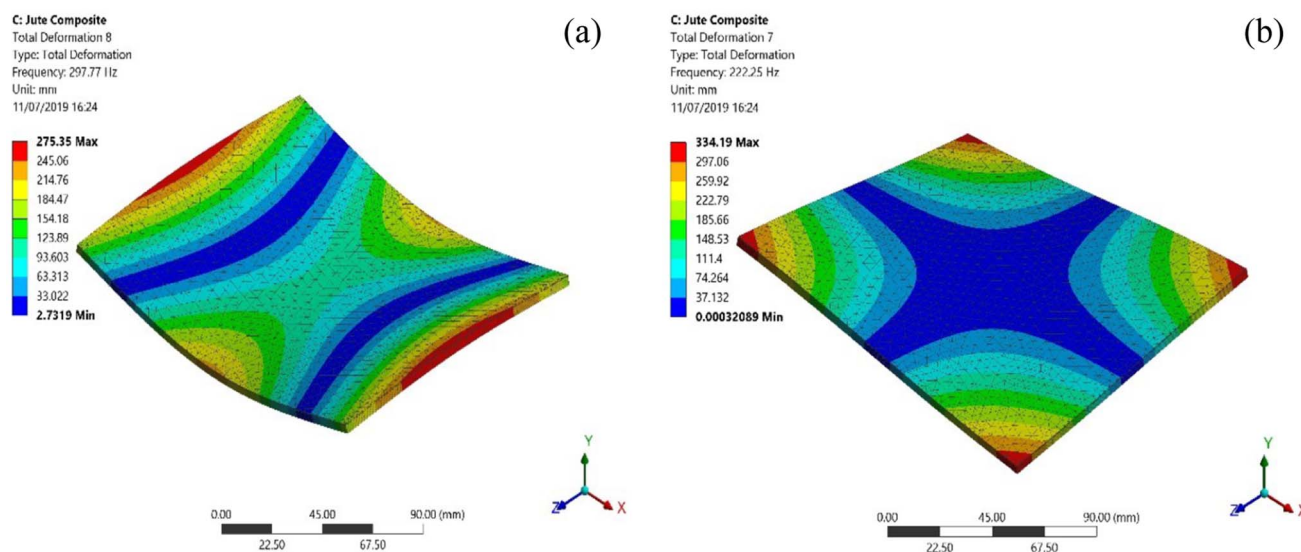


Fig. 14 Simulation results of vibration characteristics from FEA: (a) flexural mode and (b) torsional mode. Reproduced from ref. 695 with permission from AIP Publishing, 2019.

measured, which matched the fibre load from 0–40 wt%. For the discretization of the constituents, a 3D quadratic brick element, SOLID90, was used in the evaluation of the thermal properties.⁵⁴⁸ FE models for jute/glass fibre hybrid composites were created using the ANSYS19 software for the determination of the J integral and stress intensity factor. The FEA results were observed to be closely correlated with the experimental results.⁵³⁰ The thermal expansion coefficient of JFRCs was evaluated by FEA using the representative unit cell method. Also, a hexagonal array unit cell was used for the study of the contiguity of JFRCs.³³⁸ The results showed that the coefficient of thermal expansion increases with decrease in the fibre content.

A continuum damage mechanics-based mesoscale model was developed to describe the in-plane tensile response in NFRCs. These composites demonstrate a nonlinear response, with an initial rapid stiffness degradation rate, which eventually decreases, unlike SFRCs, where their stiffness degradation tends to be constant and linear. The elementary fibres were treated as composites, idealised as cylindrical tubes of varying diameters, and reinforced by microfibrils at variable orientations, as governed by the classical laminate theory.⁶⁹⁶ Andersons *et al.*⁶⁹⁷ proposed a semi-empirical model, whereby tensor-based orthotropic stress–strain relationships were made to fit experimental observations of NFRCs, and the classical laminate theory was employed to simulate the laminate tensile response. The authors showed that a purely macroscale analytical approach can reasonably reproduce the nonlinear behaviour in NFRC unidirectional laminates. Panamoottil *et al.*⁶⁹⁸ demonstrated a ‘hierarchical’ approach to simulate the tensile response of a single resin-impregnated natural fibre. Analytical microscale models were developed separately for elementary fibres, resin, and the fibre–matrix inter-phase layer, which were then combined in a finite element based ‘unit model’ of a fibre yarn impregnated and surrounded by matrix.

Poilane *et al.*⁶⁹⁹ developed a viscoelastoplastic model for fibre-direction-only tensile response in a single-ply NFRC. The strain response was considered split into pure elastic, viscoelastic, and viscoplastic components. Based on observations from creep and repeated load-unload tests, the authors proposed free energy and dissipation potentials that capture plastic yielding, temperature dependence and strain rate effects in the fibre direction. Sliseris *et al.*⁷⁰⁰ proposed micro-mechanical models within a thermodynamic framework for NFRCs under tensile loading. In this model, the fibre length and diameter were randomly distributed, and separate constitutive laws were defined such as elementary fibre, defected regions of fibre, and regions between elementary fibres and resin. The fibres were modelled simply as linear elastic, but the defected fibre and intra-bundle regions were modelled as brittle materials with linearly degrading stiffness after a specified threshold. The matrix was modelled with constant stiffness and von Mises plasticity with isotropic hardening. In the case of the woven fabric model, both the fibre and matrix were governed by nonlinear, isotropic hardening plasticity laws, but with no state variable laws that would permit any degradation of the material properties. Both models were executed *via* a finite element-based representative volume element loaded in tension. The

models captured the initiation and progressive evolution of damaged zones, and the representative volume element response closely reproduced the experimentally observed nonlinear stress–strain response.

5.11.1 Dynamic relaxation method. In the dynamic relaxation method (DRM), the equilibrium equation is converted into the dynamic equation. The damping term is expressed in the finite difference, and the solution is obtained through the iteration process. The time increment and the optimum damping coefficient are used to stabilize the solution. The stiffness matrix, applied load, boundary conditions, and mesh size of the specimen are the main variables that affect the solution.

5.12 LCA

The aim of LCA is to determine the environmental impact of using plant fibres on the surroundings.⁷⁰¹ The consumption of energy during the fabrication of bio-composites increases their environmental impact. This can be rectified through assessment of the life-cycle and service life simultaneously during the fabrication of the composite. It is also useful in minimizing the environmental effects during the processing of materials.¹²⁰ LCA was developed for the analysis of the processing of PFRCs and improvements in the environmental impact of the reaction were found.⁷⁰² Detailed analysis outside the system boundaries is required for the consequences of LCA if government policies permit the implementation of bio-based materials. A comparative LCA for a bio-based *versus* oil-based polymer composites was conducted by Rosa *et al.*⁷⁰³ This study was useful for the analysis of composite processing, ecological effects induced by fibre extraction and the consideration of bio-based and polymer-based resins. LCA on processing of poly(3-hydroxybutyrate) was conducted for the evaluation of its green-house gas emission, acidification and eutrophication potential. The results showed that parameters such as raw material selection and choice of methodology have an effect on the variability of the process.⁷⁰⁴

Alves *et al.*⁷⁰⁵ explained various ecological, social and economic merits of replacing synthetic fibres with jute fibres in an automotive structural front bonnet through LCA. The feasibility study of this application for environmental improvements was made as a case study designed by them. Environmental characterization was carried out with the use of LCA and a significant improvement in the environmental performances was seen. They also performed a quantitative analysis on the social and economic impacts and found that JFRCs performed better in both categories compared to SFRCs. Pandita *et al.*⁷⁰⁶ conducted LCA experiments on the processing of jute/glass fibre composites using the GaBi 4.3 software. The energy consumed during the processing of the jute/glass fibres was determined to be around 33 MJ kg⁻¹ of fibres. With the positive energy adjustment as a result of the use of plant fibres, the energy consumed to process 1 kg of jute was subtracted with the lowest energy balance of 8.7 MJ kg⁻¹.⁷⁰⁷

5.13 Spectroscopy studies

5.13.1 FTIR spectroscopy. FTIR spectroscopy is a useful tool for the identification of the functional groups present in

composites based on the wavenumber of the peak of the surface molecular layer with respect to its transmittance.⁷⁰⁸ FTIR spectrometers including Tensor 27 Bruker,^{327,353} Shimadzu FTIR81001,^{342,345,356} PerkinElmer,^{346,539} PerkinElmer 1720X,^{362,378} Nexus 870,^{354,371,613} ABB Bomen FTLA2000-102,³⁸⁵ Nicolet 6700,³⁶⁸ Nicolet iN10 (ref. 319) and Nicolet 10DX⁵⁶ instruments were used in the analysis of composites. The spectrum of the sample was recorded with scans in the range of 32–64.^{319,327,346,353,354,362,378,539,613} The FTIR spectra of KBr-coated JFRC samples were examined in the wavenumber range of 4000–400 cm^{-1} at a resolution of 2 cm^{-1} . The fibres were cured at 85–105 $^{\circ}\text{C}$ for one hour, and then the samples were coated with KBr for testing.^{354,613} FTIR measurements on oven-dried JFRC pellets were carried out at 105 $^{\circ}\text{C}$ for 1 h. Also, the FTIR spectra of untreated and chemically treated JFRCs were recorded.³⁴² Analyses of silane-treated fibres showed a peak value of 1100 cm^{-1} . The peaks near 1100 cm^{-1} indicated the formation

of Si–O–C bonds, showing the reaction of the silane coupling agent with the hydroxyl groups in the fibres.³⁸⁶

The FTIR spectrum of jute fibres presented in Fig. 15(a) shows the –OH stretching bond, indicating the presence of hydroxyl elements in the fibres.³⁷³ The peak at around 2900 cm^{-1} is attributed to the C–H bonds, while the peaks at 1600, 1400, and 1060 cm^{-1} represent –OH bonding, –CH₂ bonding, –C–O pyranose, and glycosidic C–H bonding, respectively. A broad absorption band was typical in all the spectra in the range of 3200–3600 cm^{-1} , corresponding to the O–H stretching (Fig. 15(b)).⁷⁰⁹ Alkali-treatment caused a reduction in the O–H stretching, resulting in more O–H groups accessible for the acetic-acid reaction. At around 2950 cm^{-1} ,⁷¹⁰ the C–H stretching frequency of the methyl groups and methylene was observed. However, the intensity of this peak decreased upon surface treatment. Hemicelluloses are the key elements of fibre alteration, showing a peak at around 1730 cm^{-1} for the C=O

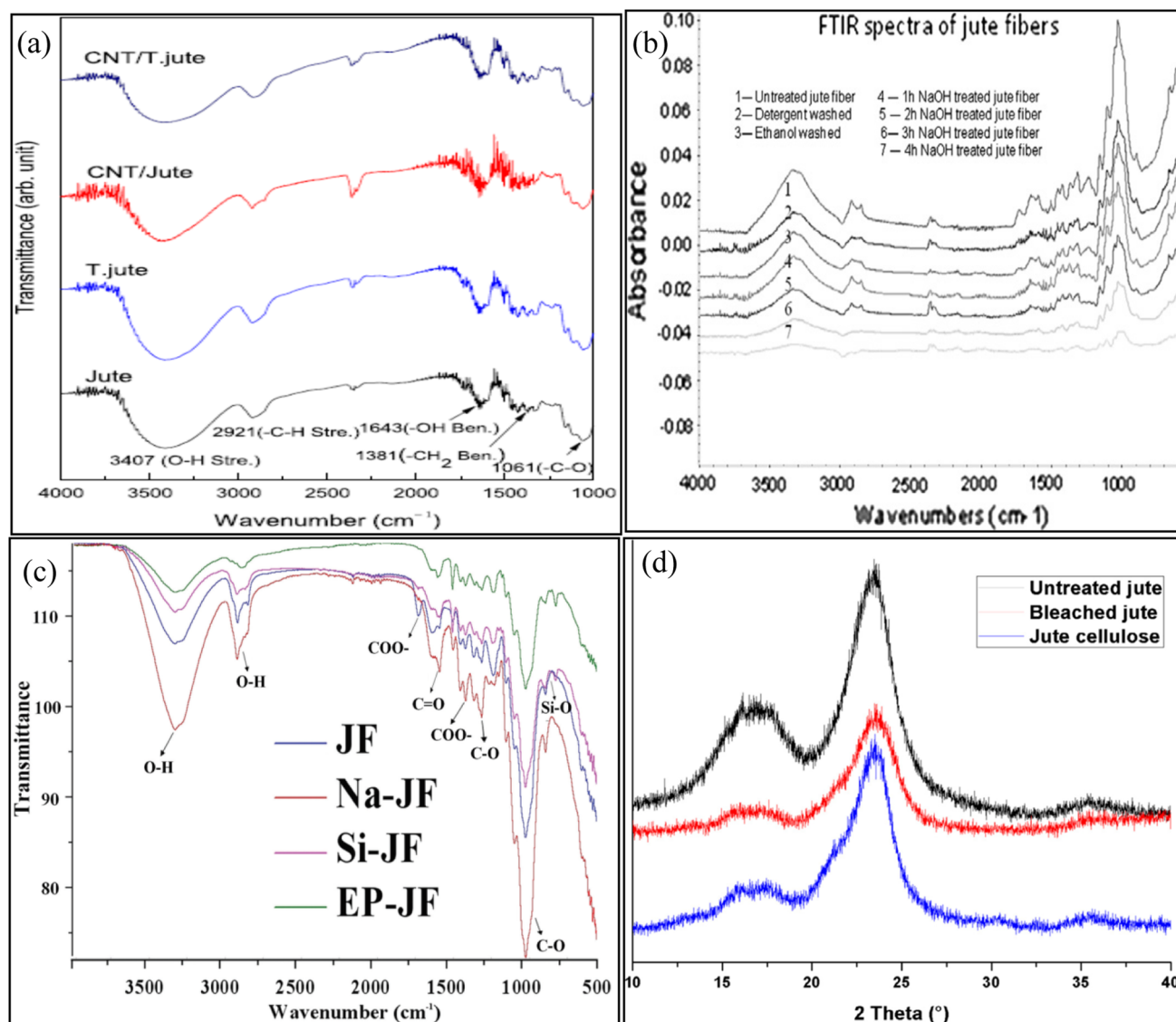


Fig. 15 FTIR spectrum of (a) raw and (b) chemically modified jute fibres. Reproduced from ref. 709 with permission from Elsevier, 2008. (c) ATR-FTIR spectra of raw and chemically modified jute fibres and (d) XRD patterns of jute fibers and cellulose nano-fibrils.

bonding of carboxylic acid and hemicellulose constituents. In the case of the alkali-treated fibres, they were dissolved in the chemicals, and thus no peaks were observed for the C=O stretching. Lignin⁶¹¹ is responsible for the absorbance band at around 1464 cm⁻¹. This peak was reduced by chemical treatment, indicating the extraction of lignin from the surface-modified fibres.

An absorption band at around 3430 cm⁻¹, which is characteristic of the stretching vibration of -OH bonded with hydrogen, was common in all the spectra. In the spectrum of the detergent washed and dewaxed jute, the peak at 1740 cm⁻¹ can be attributed to the C=O stretching vibration of hemicellulose. Alternatively, the sharp intensification the ester group appeared in the case of the MMA-grafted jute at 1743 cm⁻¹. The grafted jute fibre showed an intense peak at 2244 cm⁻¹, corresponding to the nitrile group, whereas the cyano-ethylated fibres show a low-intensity peak. This can be explained by the migration of many acrylonitrile monomers in the chain in the AN-grafted jute with the addition of only one AN molecule in the cyano-ethylated samples per group of cellulose macromolecules in -OH. The positive Lassaigne Test confirmed the presence of N₂ in the cyano-ethylated specimens.⁷¹¹ A similar observation was recorded for cyano-ethylated jute.⁴³⁶

5.13.2 Attenuated total reflectance (ATR) FTIR spectroscopy. ATR-FTIR was applied for the identification of the functional groups in jute fibres. The ATR technique was used to test samples in different states without any preparation.³⁵⁶ This testing practice in combination with infra-red light permits the direct examination of composites without prior preparation.^{358,712,713} A variety of materials is employed in ATR testing, including the widely used germanium, diamond, platinum and ZnSe.³⁵⁷ ATR-mode spectrometers including Bruker Vertex70,^{341,374} Shimadzu FTIR81001,^{357,358} and Nicolet 6700DX⁴⁴³ instruments have been used by different researchers. A total of 64 scans was performed in the wavenumber range of 400–4000 cm⁻¹ at a resolution of 4 cm⁻¹.^{341,356–358,374,443} The spectra were standardized at 895 cm⁻¹ from the vibration provided there was no subsequent shift in the peaks.³⁴¹ At intervals of 60 min each spectrum was captured using 32 scans.⁴⁴³ The ATR-FTIR spectra of jute fibres are presented in Fig. 15(c), where the increments in the absorption peaks for NaOH-treated jute fibres can be clearly observed. The silane-treated jute fibres were produced a lower transmittance due to the changes in the hydroxyl groups of cellulose and lignin. These results proved the occurrence of changes on the surface of the fibres due to the chemical modifications.³⁷⁴

5.13.3 XRD studies. The structural features of jute fibres and JFRC samples were investigated *via* XRD analysis. The dried samples were filled in the rectangular hollow region of the sample holder.³⁵³ The XRD patterns were recorded using an Ultima,^{327,353,613} Bruker D8 Focus,³¹⁹ PANalytical X'Pert PRO MPD⁵⁴⁶ and Bruker AXS³⁹⁰ X-ray diffractometer in different studies. These instruments produce X-rays using an Ni filter from CuK α radiation. The CuK α radiation was used to record the XRD patterns at a scan rate of 1° min⁻¹ (ref. 327 and 613) for 2 h in the 2 θ range of 10 °C to 60 °C. A TG analyzer was used to investigate the thermal decomposition in the temperature

range of 30–500 °C at a heating rate of 10 °C min⁻¹. The prepared specimens were measured at 200 mL min⁻¹ in fresh ambient air. A sample mass of 10 mg was taken for every experiment.³⁹⁰ The size of the crystallite was calculated using the following formula:

$$\text{Crystallite size} = 0.9\lambda/(\beta \cos \theta) \quad (26)$$

The percentage CI was estimated using the following relation:

$$\% \text{ of CI} = (I_{002} - I_{\text{am}})/I_{002} \times 100 \quad (27)$$

where I_{002} represents the intensity of the (002) reflection from the crystalline cellulose lattice and I_{am} represents the intensity of the peak from the amorphous portion of the fibre.⁷¹⁴ The XRD patterns of jute fibres presented in Fig. 15(d) display diffraction characteristics of semicrystalline materials with small amorphous peaks. Typical well-defined peaks were observed at 2 θ values of 16° and 23°.⁵⁴⁶

5.13.4 X-ray photoelectron spectroscopy. XPS assessments are performed using a spectrophotometer fitted with a monochromatic X-ray source. Several spectrophotometers operate at 1486.6 eV using AlK α radiation. The energy scale is set to a value of 83.98 eV with the help of Au photoelectrons. The spectra are captured at 160 eV in the binding energy range of 0–1400 eV. This determination is done using the ratio of the supplied oxygen to carbon. The chemical states on the surface of the sample are determined by fitting the curve of carbon and oxygen. The model is equipped with a function of the Lorentzian–Gaussian mix Voigt profile, and a program is used for fitting the non-linear least squares curve.³⁴¹

5.14 Morphological property analysis

5.14.1 Scanning/field emission scanning electron microscopy analysis. Scanning electron microscopy (SEM) is a method for the examination of the fibre/matrix adhesion based on surface morphology at the microscopic level. Specifically, the surface morphology of broken composites shows the degree of fibre/matrix adhesion.⁷¹⁵ The SEM specimens are sputtered with gold/palladium/platinum before their evaluation to reduce/avoid electro-static charging during examination.^{41,56,81,296,302,315,319,327,336,339,344,345,348,353–355,362,368,381,387,389,390,412,515,539,544,546,556,574,607,608,613,628} In the literature, images were captured using Philips FEG XL30,^{41,389,390} XL20,⁵⁶ Philips 501,⁶⁰⁸ Hitachi S-4500,⁹⁹ TM3000,^{296,319,379} TM3030,³⁴⁶ S-3400N,³⁷⁸ SU1510,³⁶⁹ Jeol JSM7600F,³⁴⁰ JSM-5600LV,^{356–358,371} JSM-6480LV,^{315,549,550,574} JSM-5610,³⁸⁷ JSM-6701F,³⁴² 35CF,^{375,628} JSM5800,³⁶² JSM6060,³³⁹ JSM840,³⁸⁵ JSM6400,^{344,374} JSM7001FD,⁸¹ TM3030,³⁴⁵ LEO 440,⁵¹⁵ Leo Supra 50VP,⁸³ Quanta 200F,⁵³⁹ Vega LSV,^{353,354,613} Vega-Tescan TS5130,⁵⁴⁶ Zeiss EVO MA,^{302,348,368} FESEM Zeiss Auriga,³³⁶ Zeiss DMS960,⁶⁰⁷ Siemens XL30,⁵⁵⁶ DXS-10ACKT,³⁸¹ TS5130-Tescan,⁴¹² and LEICA S440 (ref. 544) microscopes by various researchers. The morphologies of the composites were observed at a 5 kV,^{339,379,556} 10 kV,^{81,344,368,381,515} 15 kV,^{296,348,385,387,539} 20 kV,^{56,412,544,574} 25 kV (ref. 302, 336 and 607) and 30 kV (ref. 41, 378 and 546) acceleration voltage.

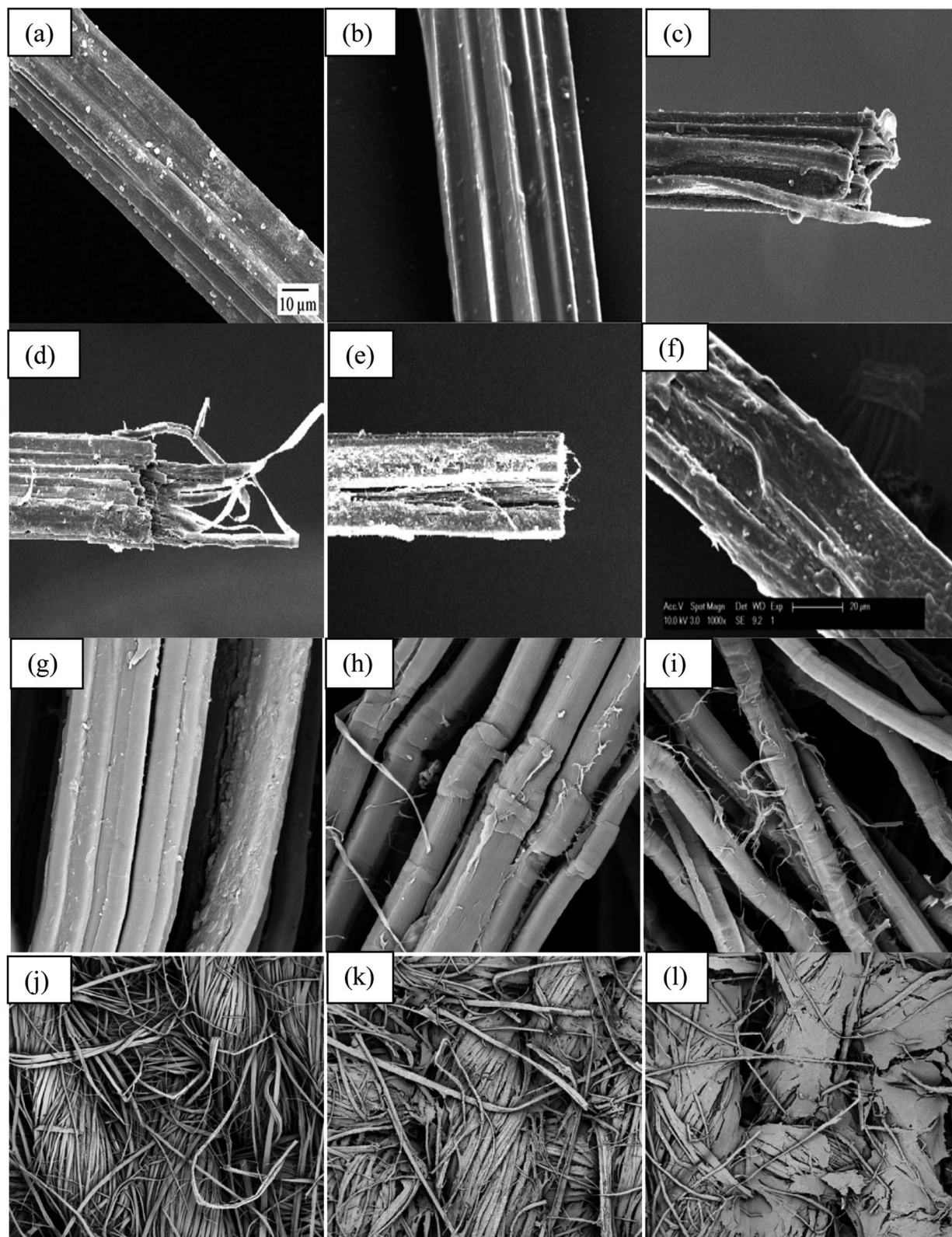


Fig. 16 Morphology of jute fibre: (a) axial direction. Reproduced from ref. 61 with permission from Elsevier, 2008. (b) Longitudinal direction. Reproduced from ref. 348 with permission from Elsevier, 2016. (c) Failure of fibre in axial direction. Reproduced from ref. 61 with permission from Elsevier, 2008. (d) Failure of alkali-treated fibre in axial direction. Reproduced from ref. 61 with permission from Elsevier, 2008. (e) Failure of silane-treated fibre in axial direction. Reproduced from ref. 61 with permission from Elsevier, 2008. (f) Surface defects. Reproduced from ref. 343 with permission from Elsevier, 2013. (g) Raw fibre, (h) alkali-treated, (i) bleached, and surface coated jute fibres at: (j) 0 wt%, (k) 2 wt%, and (l) 5 wt% of nano-cellulose.

Fig. 16(a–f) show the SEM images of jute fibre morphology in the longitudinal direction and its failure modes. Generally, the longitudinal shapes of the jute fibres are not uniform along

their length.⁶⁴ Fig. 16(c–e) demonstrate the micro-failure mode under axial load for raw, chemically treated, and silane-treated jute fibres, respectively. This can be affected by tensile stress

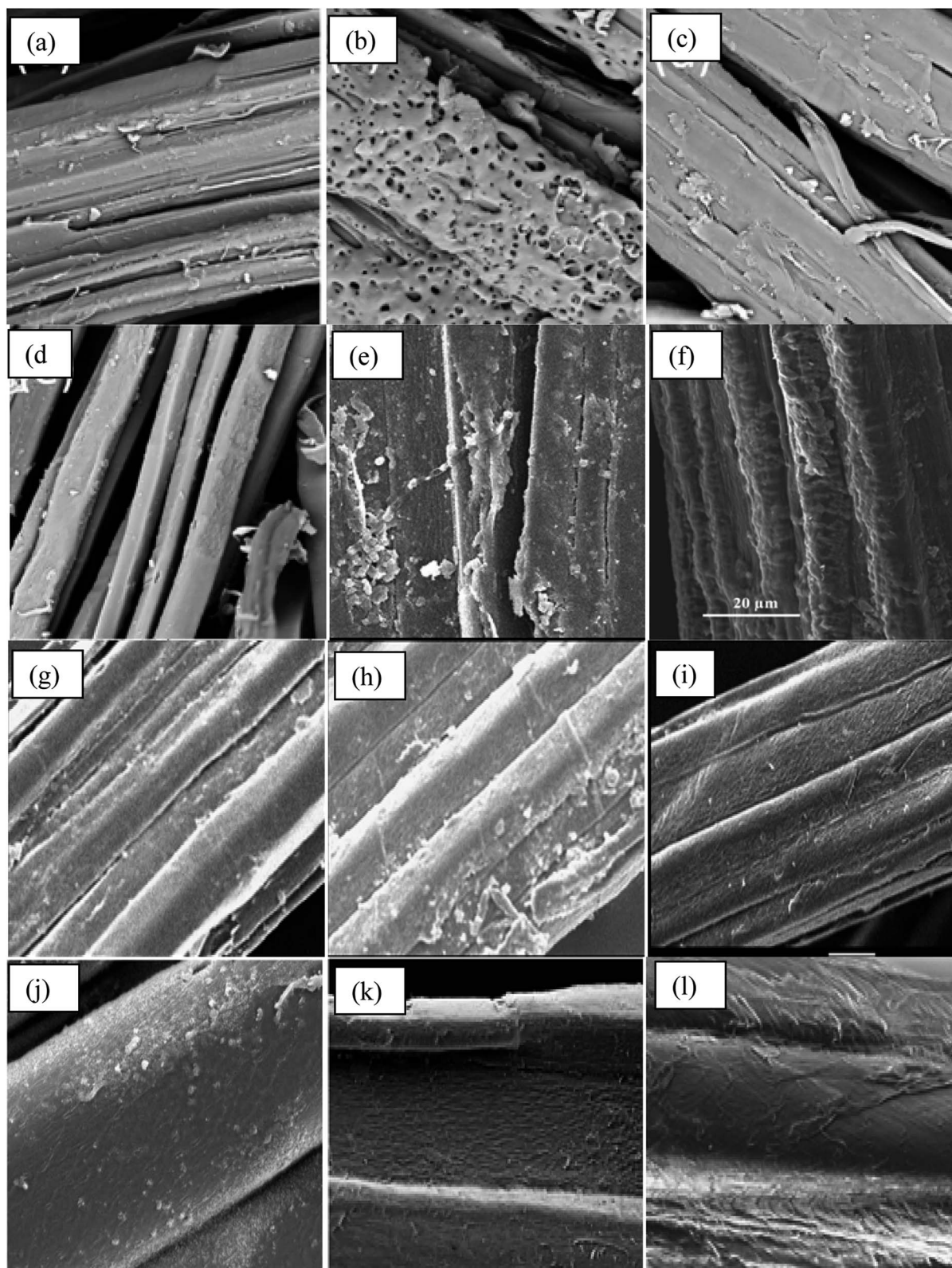


Fig. 17 SEM images of modified jute fibres: (a) enzyme, (b) laser, (c) ozone, (d) plasma, (e) KMnO_4 , (f) immersed in cement paste for 90 days, (g) detergent washed, (h) ethanol washed, (i) detergent + ethanol washed jute fibres, (j) 3% NaOH, (k) 5% NaOH, and (l) 7% NaOH treated fibres.

prior to the failure in each case before the boiling water test, while the micro-failure mode after the test can be affected by the low tensile strength due to the fibrils swelling in the fibres. In

the silane-treated sample, the fibres did not show any swelling following the water absorption due to the strong bonding between their adjacent layers.⁶¹ The surface defect due to the

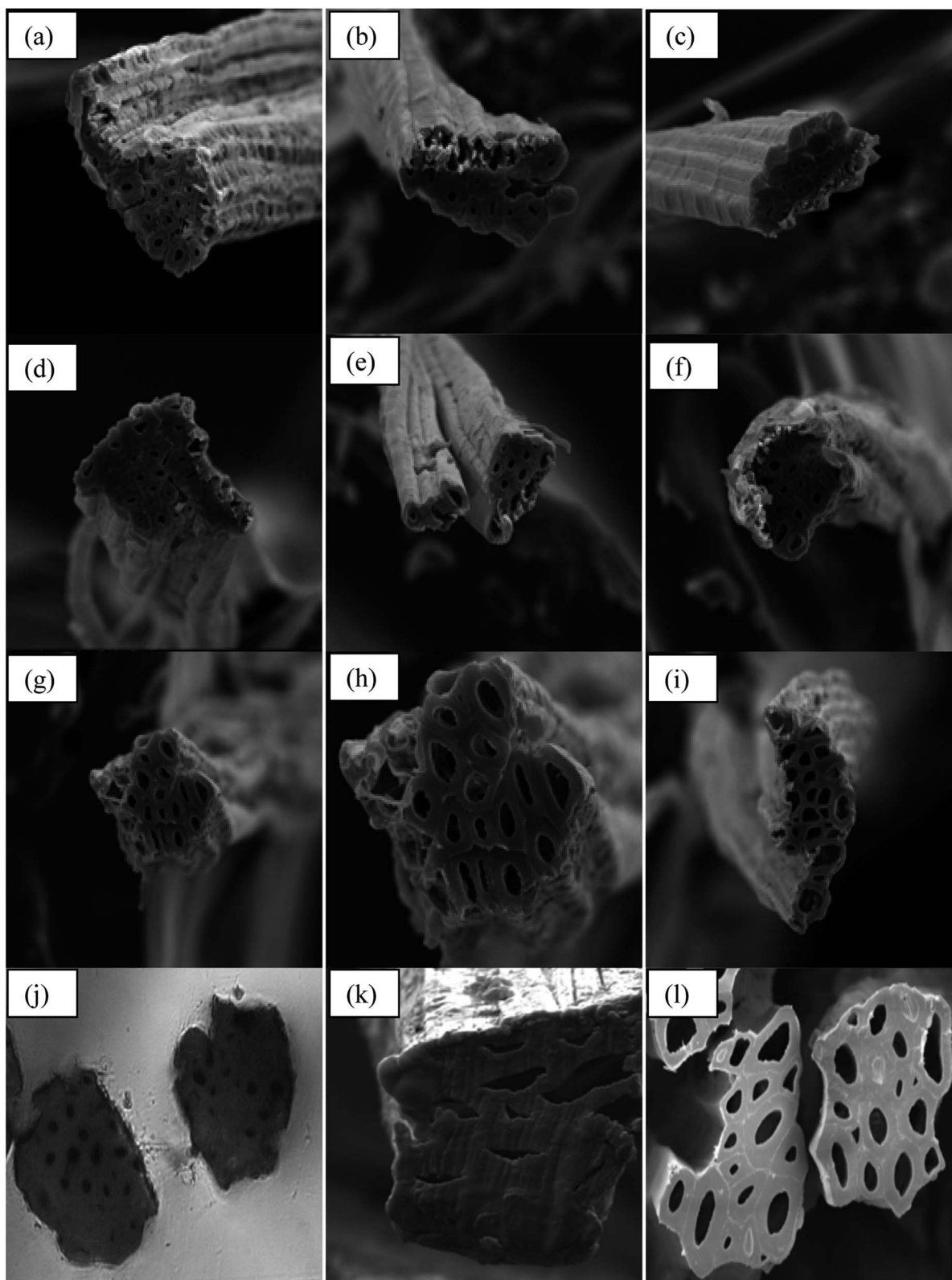


Fig. 18 SEM images of the cross-section jute fibres at their failure modes. Compiled from ref. 61 with permission from Elsevier, 2008,²⁹⁶ with permission from Elsevier, 2016,³⁴³ with permission from Elsevier, 2008, and³⁴⁸ with permission from Elsevier, 2016.

concentration of chemicals is shown in Fig. 16(f).³⁴³ Fig. 16(g) shows bundles of jute fibres combined by their constituents. Following surface modification, the fibres were observed to split due to the degradation of the surface mesh structure, and whole portions of the cellulose and non-cellulose materials were removed, as observed in Fig. 16(h). The fibres were further isolated into individual fibres due to the chemical treatment and surface fibrillation of the fibres was possible, as shown in Fig. 16(i), which may be due to their further realignment following bleaching treatment. The morphological changes occurring after the nanocellulose coating on the jute surface are depicted in Fig. 16(j–l). The nanocellulose precipitated on the fibre, forming a coating. The thickness of the coating slowly increased with the an increase in the concentration of nanocellulose, as evident from the images.

The changes in the fibre surface following various surface treatments are described in Fig. 17(a–f), demonstrating major texture changes in surface after treatments. Fig. 17(a) shows the raw and fragmented surface morphology of the enzyme-treated fibres, arising as a result of the removal of the binding constituents from the fibres. Fig. 17(b) shows the thermal degradation following the laser-treatment of the fibres, resulting in a rough and porous surface. The crack growth and increase in surface roughness of the jute fibres treated with ozone are visible in Fig. 17(c). Plasma treatment induced a small increase in the surface roughness of the fibre (Fig. 17(d)).⁴¹² Fig. 17(e) displays the surface of the KMnO_4 -treated jute fibre. The images clearly show that the fibre surface become rough after KMnO_4 treatment compared to the raw fibre surface. This rough surface initiated the formation of fibre–matrix

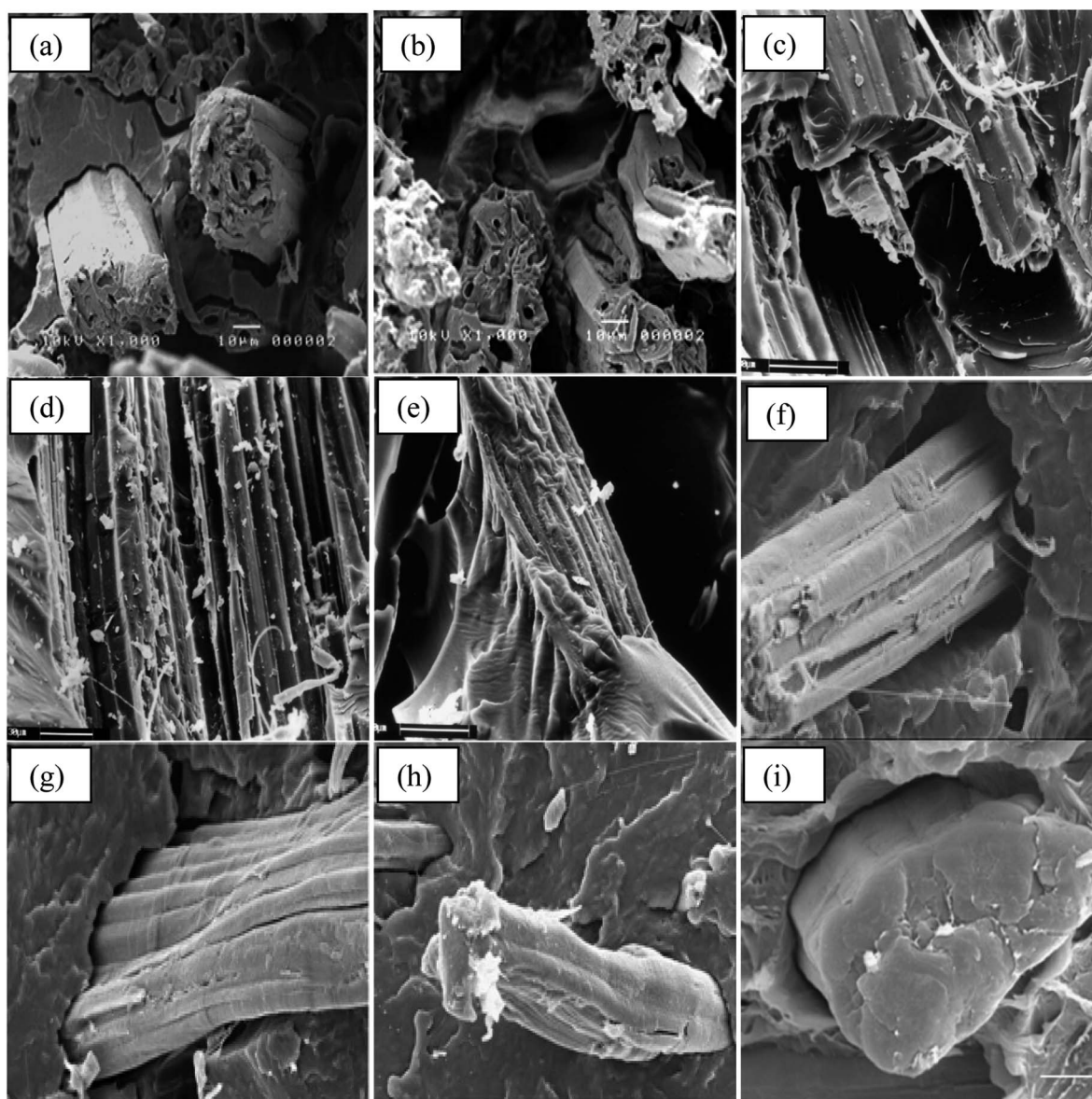


Fig. 19 SEM micrographs of fractured surfaces of JFRCS: (a) raw fibre, (b) KMnO_4 treated, (c) fibre breakage from the matrix, (d) resin layer on the fibres, (e) bonding between the fibre and the matrix, (f–h) effects of surface modification with different fibre contents on tensile properties and (i) fractured surfaces of JFRCS.

interlocking, which yielded better properties.³⁵⁷ Fig. 17(f) displays the micrograph of the chemically treated jute fibres soaked in cement paste. As the surface of the jute fibres became

degraded, the key explanation for the decomposition of the fibres in chemical medium is Ca^{2+} fastening on the fibre surface, whereas when the surface for the fibre is coated with

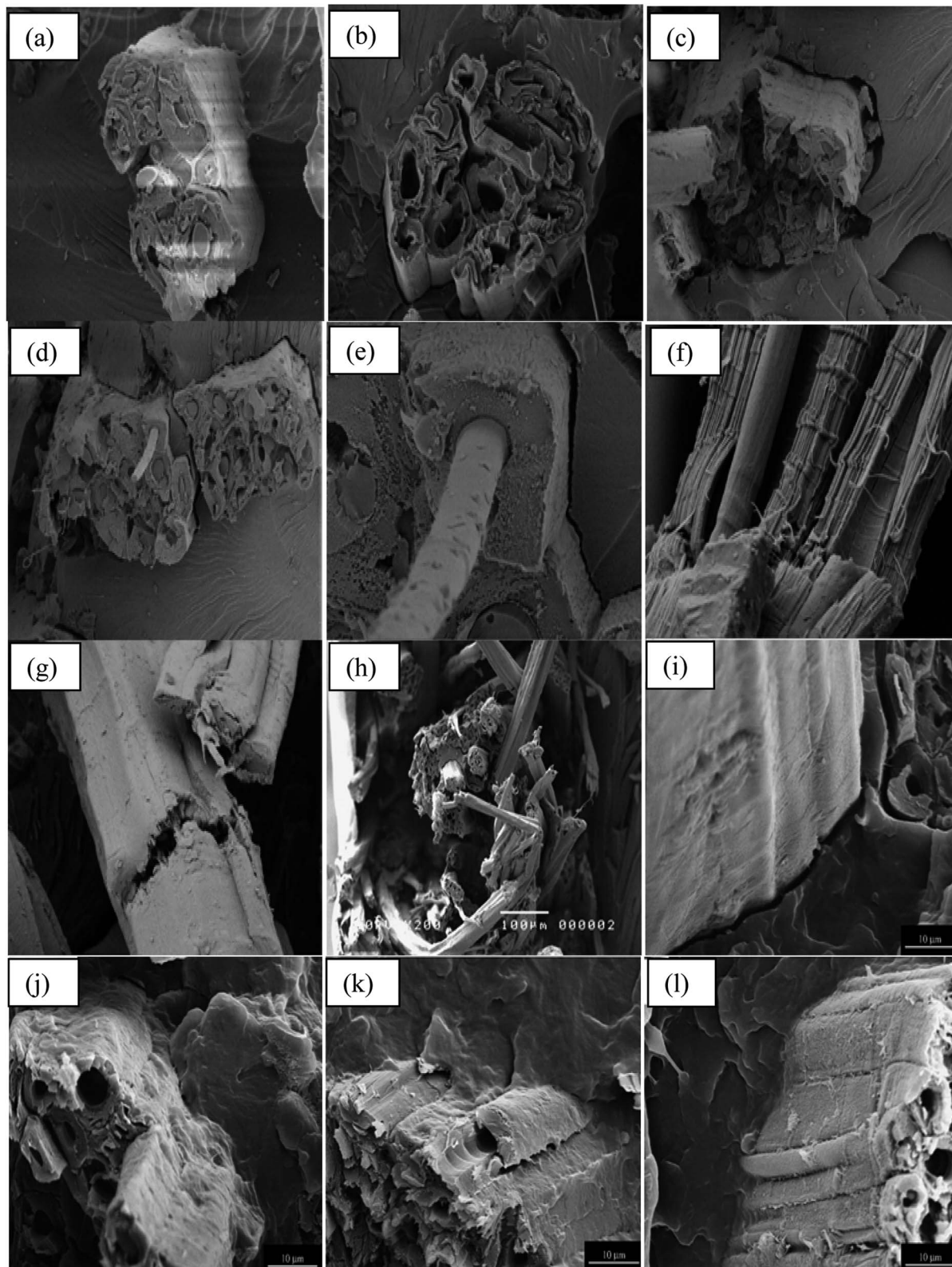


Fig. 20 SEM micrographs of fractured surfaces of (a–g) JFRCS, (h) KMnO_4 -treated and (i–l) Epolene G3003-treated JFRCS.

polymer, the Ca^{2+} fastening tends to be reduced.³²⁷ The surfaces of the chemically modified fibres analyzed through SEM images showed comparatively rough surfaces due to the treatment (Fig. 17(g–i)). The chemically treated fibres provided cleaner and rougher surfaces due to the removal of impurities, non-cellulosic constituents, inorganic substances, waxes, *etc.* The rough surface texture and defibrillation were also a result of the stronger fibre-to-matrix interaction with a wider surface area.⁴⁴³ The removal of waxes and other impurities made the fibre cleaner and increases its bonding strength, as confirmed by the images presented in Fig. 17(j–l).³⁷¹ Fig. 18 displays the results of the cross-sectional micro structural analysis carried out on jute fibre. It highlights an irregular geometric cross-section characterizing jute fibre, which is usually not circular, and the diameter is not uniform throughout the length of the fibre.

Moreover, along its longitudinal axis, the fibre cross-section is not constant.³⁴⁸

The fractured surface of the unaltered JFRCs is presented in Fig. 19(a). The fibre that emerged from the matrix confirmed the weak fibre–polymer bonding, resulting in the formation of voids in the matrix. The better adhesion prevented fibre pull-out and debonding. The SEM micrograph in Fig. 19(b) demonstrates the increased adherence of the fibres to the resin as a result of their successful bonding at the interface, resulting in reduced fibre pull-out by KMnO_4 treatment.³⁵⁷ The fibre breakage and pull-out from the matrix are shown in Fig. 19(c), with no resin binding to the fibres (Fig. 19(d)). Clear adhesion on the surface of the jute fibre between the latex and the resin is evident in Fig. 19(e), where the latex-coated jute fibres are strongly adherent to the resin.⁵⁴⁴ The SEM images of the surface-modified JFRCs with different

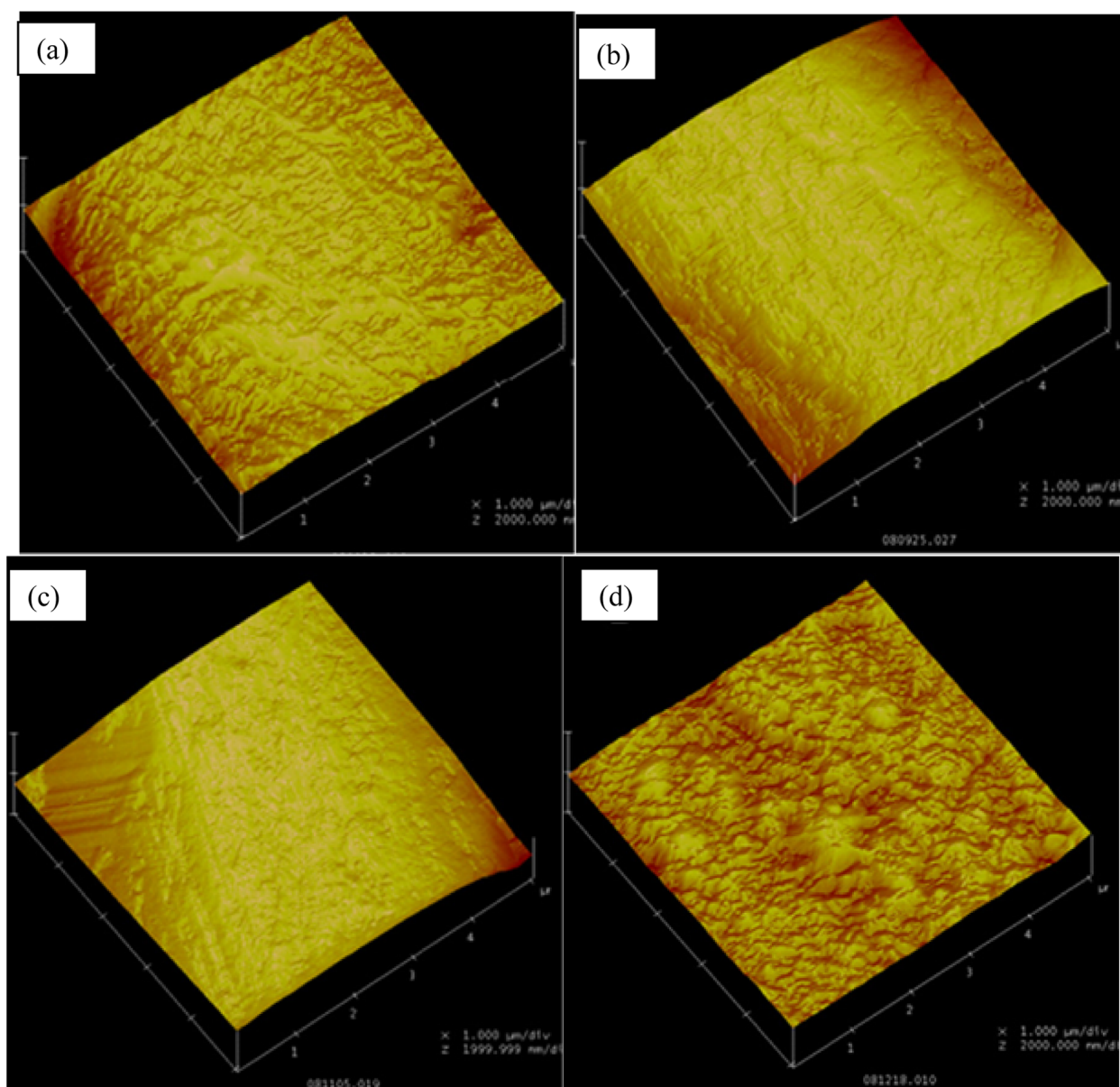


Fig. 21 Typical SEM contact mode images of jute fibres: (a) raw, (b) He treated, (c) He/acetylene treated and (d) He/ N_2 treated. Reproduced from ref. 341 with permission from Elsevier, 2011.

fibre contents are shown in Fig. 19(f–h).³⁷¹ The SEM findings, as observed from the fractured specimens after the tensile examination in Fig. 19(i), support the enhanced interfacial adhesion.⁶⁰⁷

The water absorbed in fibres establishes hydrogen bonds, which minimize the fibre–matrix interactions. The interfaces of the virgin composites tended to be intact, showing pull-out fibres (Fig. 20(a)). Fibre/matrix failure was evident in the images after ageing (Fig. 20(b–e)), where the fibres removed during tensile testing are visible. Fibre degradation as the result of moisture content was confirmed by the existence of fibrillation and micro-cracks (Fig. 20(f–g)). It was also observed that the lumen size of the fibres in the JFRCS appeared to be fully occupied with the matrix (Fig. 20(d and e)).³³⁶ Fig. 20(h–l) display the SEM images of the fractured JFRC surface. The SEM image of the KMnO_4 -treated surfaces of the broken composites is depicted in the Fig. 20(i), showing a decrease in fibre pull-out after chemical surface modification.³⁵⁸ A small gap between the fibre and resin and slight adherence of the resin on fibre were also seen. This is an indication of the saturation of the fibre surface –OH groups, requiring a higher concentration of compatibilizer, and accordingly the interface strength can be improved further.⁶²⁸ This is due to the changes in the moisture content and the stress produced at the joint due to the thermal expansion coefficient of the resin and failure at the interface of the fibre/resin bonding.⁷¹⁶

5.14.2 Scanning probe microscopy analysis. Topographic images of jute fibres were obtained through use of silicon nitride pyramid tip probes with a spring constant of 0.12 N m^{-1} and a contact mode digital instrument. Single fibres were taken and fixed on both ends on a glass slide. The test was conducted at five different spots on the fibre surface to capture the fibre surface topography.³⁴¹ The typical photographs of the position of the jute fibres are shown in Fig. 21. The untreated fibres displayed some degree of ruggedness, as seen in Fig. 21(a). Alternatively, the jute fibres treated with He and acetylene plasma showed with a smoother surface. However, an increase in roughness was observed when He/ N_2 plasma treatment was performed. The roughness was found to have a very broad statistical variability due to the inhomogeneous existence of the jute fibres.³⁴¹

5.14.3 Optical microscopy analysis. The micrographs captured through use of optical microscopy are shown in Fig. 22. The images for the samples of coated jute fibres showed better matrix blends and enhanced impregnation within and outside the jute fibres compared to the non-coated specimens.³⁶³

5.14.4 Polarized optical microscopy (POM) analysis. The reinforcement type in a composite has a major effect on its surface morphology. Jute fibres were reinforced with PP films and processed using a precisely controlled iso-thermal process,

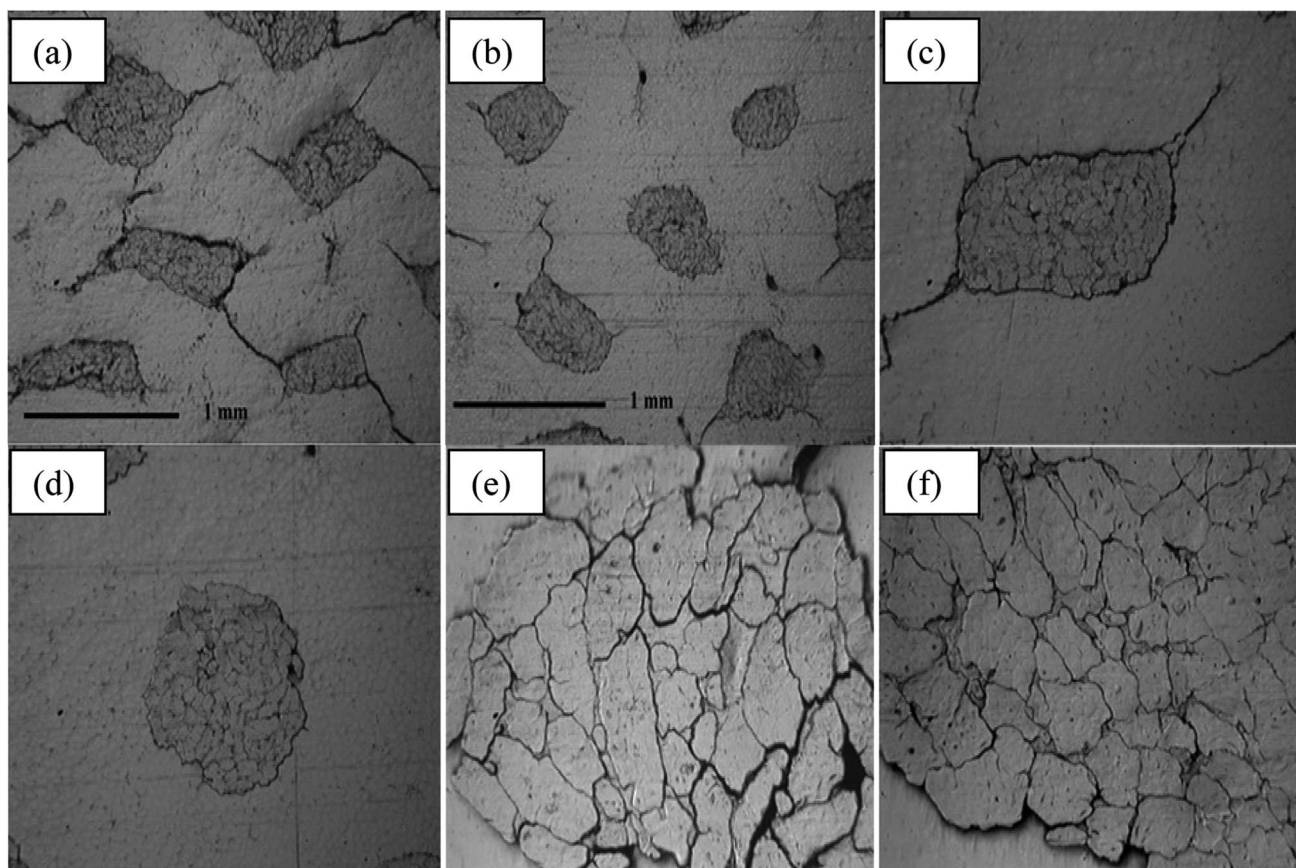


Fig. 22 Optical images of jute micro-braid JFRC specimens: (a, c and e) uncoated and (b, d and f) PP/PVA coated. Reproduced from ref. 363 with permission from Elsevier, 2006.

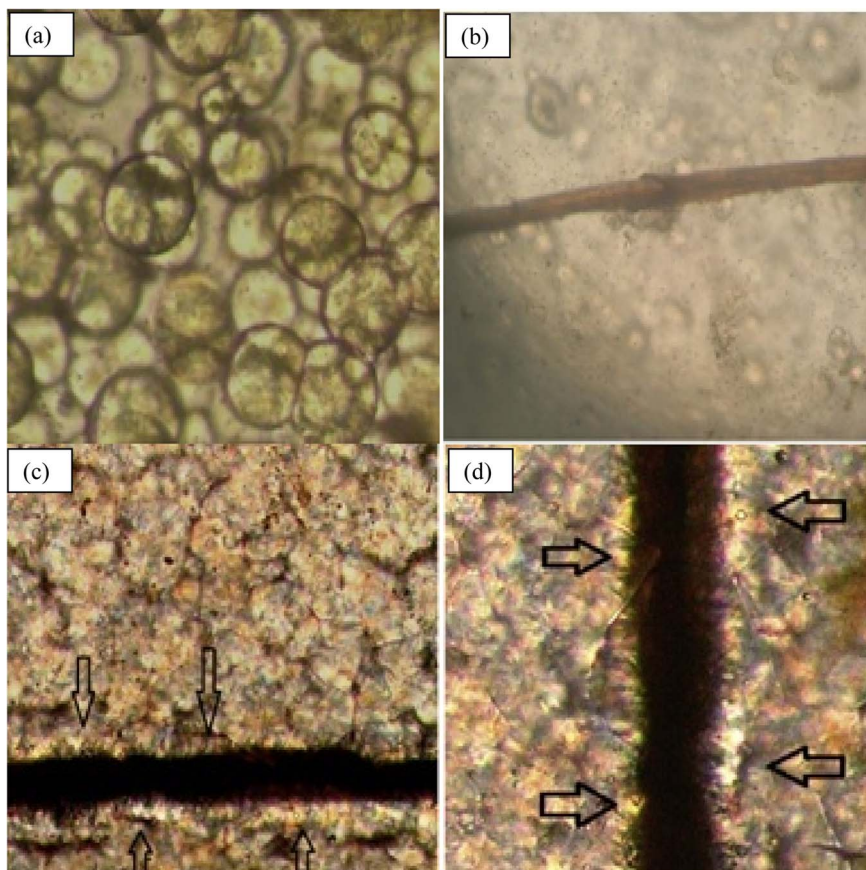


Fig. 23 POM images of: (a) neat PP, (b) jute fibre in resin melt, (c) nuclei-growth initiating and (d) formation of transcrystalline layer. Reproduced from ref. 356 with permission from Elsevier, 2013.

and then cooled.⁴⁹⁴ Fig. 23 shows the POM photographs of isothermally crystallized samples,³⁵⁶ indicating an increase in the thickness of the transcrystalline layer with time. The growth of the transcrystalline layer eventually comes into contact with the spherulite boundary in the bulk. Another explanation for the formation of these layers is based on the thermal stress produced at the interface. The load-induced polymer chains serve as nucleation points.⁷¹⁷

5.14.5 Atomic force microscopy (AFM) analysis. The AFM technique is very sensitive to the tip-sample interaction due to the bonding difference, and also the contact area of samples, and thus can map topographical features. The 3D surface topography and root-mean squares of jute fibres were examined using AFM analysis. The fibres were immobilized, and scanning was performed in tapping mode with a silicon cantilever. The scanning rate was set at $5000 \times 5000 \text{ nm}^2$ and at a frequency of 2.0 Hz.³⁶⁹ Using an atomic force microscope at a resonance frequency of 300 kHz with a voltage of 220 mV, fracture surfaces were examined after the tests.¹⁰⁵ A microscope with a Nanoscope controller was used to observe the dispersion of jute nano-fibrils in the matrix. The images were captured using a phosphorous-doped silicone tip in tapping mode with a nominal frequency of 312 kHz.³⁷⁶ Investigation of the jute nano-fibril dispersion into composites was done by AFM analysis which was observed to be uniform, as shown in Fig. 24.

The jute fibre surface was rough, irregular and covered with impurities, as visible from the magnified images (Fig. 24(c)). The AFM image presented in Fig. 24(d) shows the natural surface of the jute fibres, with the roughness minimized possibly due to the enzymatic dislocation and redistribution of lignin. Several lignins in the jute fibres were reduced to low-molecular-weight fragments at the start of laccase treatment and dissolved in the aqueous media. Laccase-mediated lignin polymerization was predominant at the later stage of the reaction, and the dissolved lignins were bound on the fibre surface.³⁶⁹ Similar results were also seen in the AFM topography of the fibres on a smaller scale (Fig. 25). The phase picture also displays some 200 nm structures. The images show the details of the topography due to the surface nature of the fibre or larger topographical structures.³⁸² An AFM study was carried out on the broken surface of the fibres. The picture shows a waxy surface with compact structures. The occurrence of failure in the inner layer of the composites was likely due to the covering constituents such as fat, lignin, pectin and hemicellulose.³⁸²

5.15 Structure-process-property relationships

The structure of plant fibres is based on the hierarchical arrangement of fibrils and the composition of the constituents present in the fibre with different proportions.⁷⁰ Structure-

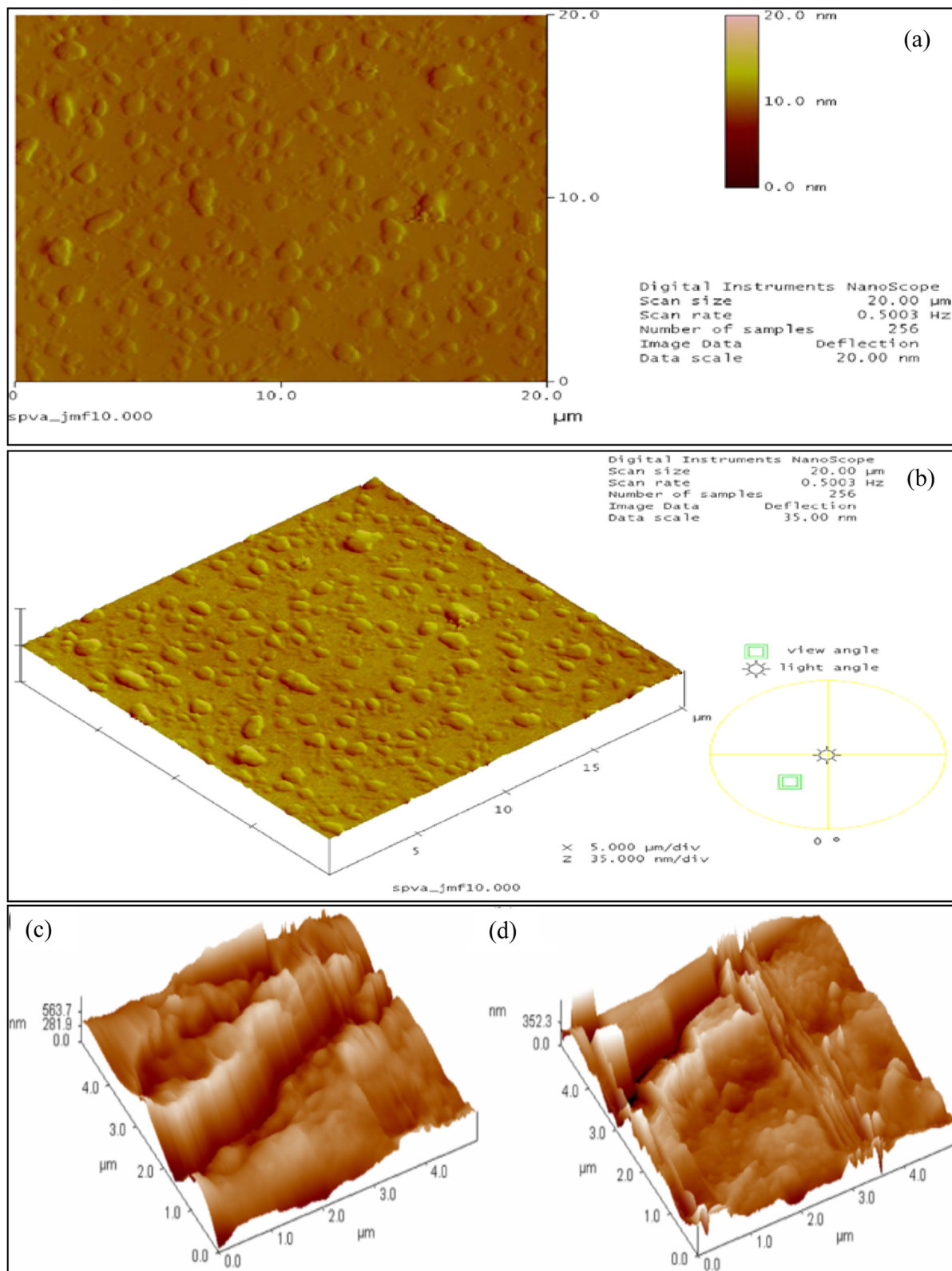


Fig. 24 AFM images of (a) 2D, (b) 3D, (c) untreated, and (d) laccase-treated jute fibres.

property relationships were developed for a description of the characteristics of jute fibres.¹⁵⁵ A relationship between structure and property was defined by the thermal stability of the

material.⁴⁵ The relationship between the mechanical property and structure of plant-based fibres can be described using the following relation:¹⁵⁵

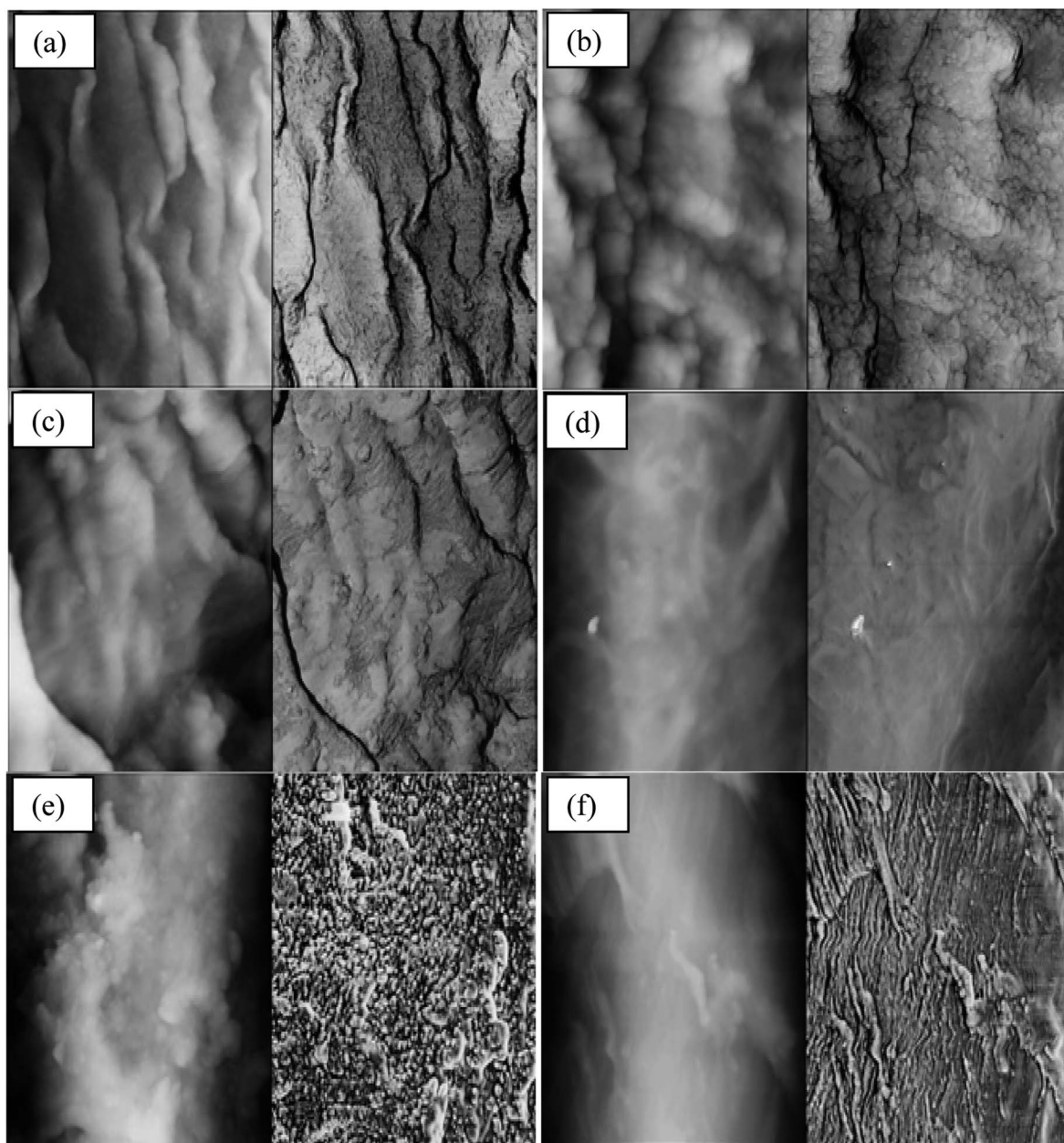


Fig. 25 AFM images of (a) untreated, (b) NaOH-treated and (c) NaOH/(APS + ED)-treated jute fibres. AFM topography and phase images of (d) untreated, (e) NaOH-treated and (f) NaOH/(APS + ED)-treated jute fibre surfaces.

$$\sigma \propto C f_r^i \quad (28)$$

where C is the crystallinity ratio, f_r is Herman's factor and i is an exponent.

An increase in the tenacity of the chemically modified jute fibres with an increase in physical parameters was found according to the above-mentioned equation. Correlation with a factor of 87% was achieved. The degree of polymerization of the matrix has little influence on the structural parameters. The structure–property relationship of polymer-based JFRCs was analyzed by Sengupta.⁵⁵⁵ The analysis showed the random orientation of non-woven JFRCs, indicating better properties than woven JFRCs. The impact strength and moisture-

absorbing nature of multi-layered JFRCs were also observed to be higher compared to single-layered JFRCs.

6. Machining characteristics of JFRCs

The need for machining operation of a certain degree is important in the production cycle of composites. Removal of the excess materials for the establishment of the exact shape and size of the finished goods is a subtractive process. One of the main machining operations performed in composite laminates⁷¹⁸ is drilling, while milling is a significant machining operation that can produce the required shape, size and tolerances.⁵²⁹ Machining of NFRCs is a complex process due to their

high abrasiveness, anisotropic nature and heterogeneous structure.⁷¹⁹ During machining,⁷²⁰ issues such as fibre peel-up, pull-out, debonding, spalling, and fuzzing could be identified. An analysis conducted by Nassar *et al.*⁷²¹ on the machinability of NFRCs helped in the identification of the key factors affecting the quality of machined parts. The correlation among cutting velocity, thrust force and feed rate for the drilling of PFRCs was reported by Chandramohan and Marimuthu.³⁹² They found no remarkable effect of velocity on thrust force, and with an increase in cutting speed at lower feed rates, but a decrease at a higher feed rate. The following equation can help measure the surface damage of a drilled hole (delamination).

$$\text{Delamination factor } F = D_{\max}/D \quad (29)$$

where D_{\max} is the maximum hole diameter and D is the diameter of the drill.

Yallem *et al.*⁷¹⁸ conducted experiments on the impact of machining parameters on the output responses during drilling of JFRCs. They observed a close relation between the geometries of the tool and the delamination of the hole, and parabolic drills produced holes of better quality than solid drills with lower thrust forces. Celik *et al.*⁵²⁹ examined the impact of cutting parameters on the thrust force, delamination and surface roughness during milling of JFRCs and found that the cutting speeds and feed rates were highly affected by the thrust force, delamination and surface roughness. The effect of cutting parameters on the thrust force and torque in JFRC milling was investigated and it was found that the cutting speed and feed rate were highly influencing parameters, while the depth and feed rate were the dominant variables for torque.^{722,723}

Using the grey relational analysis (GRA) technique in combination with the principle of component analysis, Sankar *et al.*⁷²⁴ optimized parameters such as speed, feed, cut depth and fibre angle in the machining of JFRCs. They also found that the optimal solution was obtained by the use of combined techniques. The surface roughness and material removal rate were optimized in the machining of jute/flax composites by using the Taguchi technique and ANOVA. The results showed that the spindle velocity and drill diameter are the most influential parameters for surface roughness and material removal rate.⁷²⁵ GRA and ANOVA were used in the test of the influence of treatment on the delamination factor of JFRCs.³⁶⁷ It was observed that alkali-treatment decreased the delamination at the exit and the feed rate was the most influential factor. Celik and Alp⁷²⁶ investigated the importance of milling parameters on the cutting force, delamination, vibration intensity and surface roughness. They found that an increased spindle speed produced an increase in the vibration and delamination responses and reduction in surface roughness. They also found an improvement in all responses with an increase in the feed rate.

7. Applications of jute fibres and JFRCs

7.1 Applications of jute fibres

➤ Bulk packaging for agricultural commodities.

➤ Food, garment, and non-textile industries as raw materials.

➤ Making raw cotton bales, wrapping cloth and coarse cloth.

➤ Making window curtains, chair covers, floor carpets, floor mats, hessian cloth, handbags, ornaments, fancy items, technical clothing, *etc.*

➤ Use of jute plant butts for weaving low-priced cloth.

➤ Store seeds, prevention of weeds and application of slow fertilizer to the seedlings.²⁹¹

7.2 Applications of JFRCs

Bio-based composite materials are emerging as viable alternative reinforcements to SFRCs in several engineering applications.^{297,727} NFRCs are cost-effective products; hence, numerous applications in various fields in the form of furniture, roof panels, ceilings, partition walls, packaging materials, automotive, and railway compartment interiors and storage silos have been reported. These composites are also used in non-structural auto body parts such as door panels, package trays, hat racks, instrument panels, internal engine covers, sun visors, boot liners, oil air filters, seat backs and exterior underfloor paneling.⁵⁶⁸ PFRCs are potential materials for biomedical and packaging applications.^{728,729} A report on Audi car door panels replacing conventional breaking panels with NFRCs showed a decrease in the energy consumption by 45% and CO₂ and methane emissions.⁸² Plywood, fibre boards of medium density, artificial boards, building components and plush doors were made from JFRCs.²⁹¹ These composites have been applied in the biomedical field such as tissue engineering, diagnostics, drug delivery, and wound healing.⁷³⁰ Other applications of JFRCs include lampshades, shopping bags, paper-weights, helmets, toilet closets, bath devices, electrical fittings, covers, hoses, household post-boxes, milk-boxes, fancy tiles, food containers, temporary ceiling panels, bio-gas storage devices, and movable and pre-made buildings for use in times of natural disasters.^{5,731-733}

One of the promising applications of JFRCs is based on their ability to be shaped into a variety of complex parts by harnessing their potential as reinforcement, *i.e.*, making various products such as building components,^{520,537} food grain storage units,⁵²⁰ models of low-cost housing units,^{520,521} roofing,⁵¹⁹ wood replacements,^{521,522} and pipes.⁷³⁴ In ancient times, JFRCs were used for making cords, belts, hessians, sack cloths, mats and tapes.²⁹¹ They were also used in the manufacture of building materials, thin sheets, window frames, vehicle shutters, goods packaging, geo-textiles, printed circuit boards, absorbent material, household accessories, bio-degradable shopping bags, *etc.* Some of the applications of JFRCs are presented in Fig. 26.

7.3 Comparison of properties and applications

Plant-based fibres are environmentally sustainable substitutes to synthetic fibres, and thus used as reinforcements in polymer matrix composites. However, the mechanical properties of these fibres and their composites in load-bearing components and structural applications are limited. A comparison of the

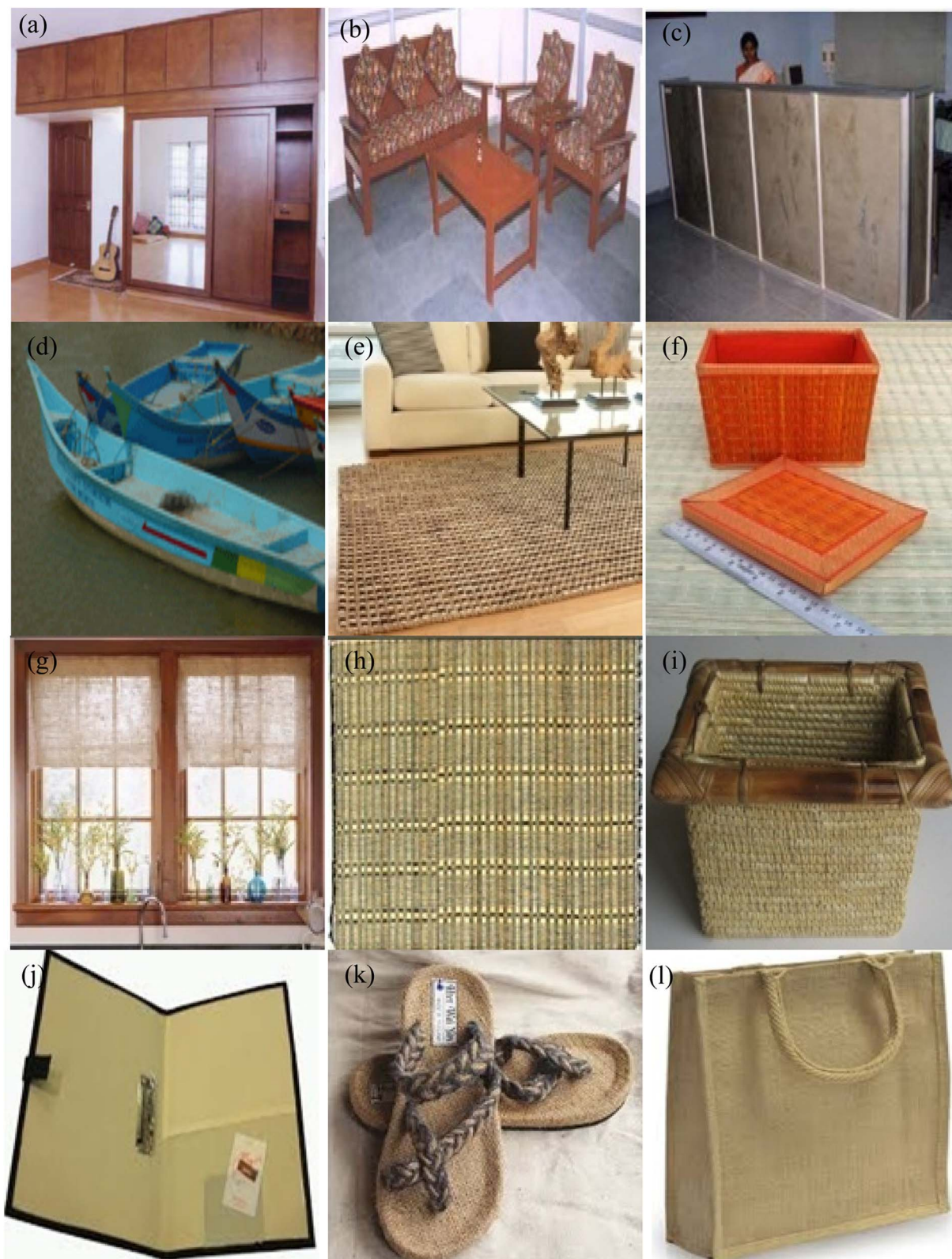


Fig. 26 Applications of JFRCs: (a) doors, (b) furniture, (c) partition boards, (d) fishing boat, (e) rugs, (f) boxes, (g) curtains, (h) mats, (i) tray, (j) stationery, (k) footwear and (l) bag.

Table 9 Comparison of the properties and applications of jute fibres with other plant-based natural fibres

Fibre	Density (g cm ⁻³)	Tensile strength (MPa)	Tensile modulus (GPa)	Specific modulus (approx)	Elongation (%)	Applications	Ref.
Abaca	1.5	400–980	6.2–20	9	1.0–10	Twines, ropes, <i>etc.</i>	11, 132 and 735
Alaska	—	271–733	3.2–49.5	—	0.6–1.7	—	736
Alfa	0.89	35	22	25	5.8	Paper, construction, <i>etc.</i>	737
Areca	0.7–0.8	147–322	1.12–3.15	—	10.2–13.15	—	78, 132 and 134
Bagasse	1–1.5	200–300	16–28	19	1	Bio-fuel, pulp, paper, construction, fencing, panels, railing systems, decking and window frame	11, 136, 145 and 738
Bamboo	0.5–1	200–750	10–30	24	2.2–3.5	Construction, furniture, textile and paper	132, 134 and 739
Banana	1.4	450–550	10	10	2–8	Food container, textile, <i>etc.</i>	132 and 140
<i>B. mori</i> silk	1.5	200–210	6	—	18–19	—	78
Coir	1–1.5	100–220	3–7	4.5	10–50	Floor mats, door mats, storage tanks, packaging, helmets, mirror casing, roofing sheets, brushes and mattresses	11, 132, 133, 136 and 140
Cotton	1.2–1.8	300–790	5–13	8	4–12	Textile, goods and cordage and furniture	11, 132, 133, and 140
Curaua	1.5	100–1000	12–100	40	1–5	Car interior, furniture and acoustic insulation components	25, 132 and 740
Elephant grass	817	185	7.4	—	2.5	—	741
Flax	1–1.4	350–1850	28–100	50	1–3.5	Composite, panels, fencing, fork, laptop cases, window frame, tennis racket, bicycle frame and textile	11, 132, 133 and 138–140
Hemp	1.2–1.8	300–870	25–85	48	1.1–4	Construction, food, papers, medicine, fuel and textiles, cordage, furniture, packaging, geo-textiles and electronics	11, ¹³² ¹³³ , ¹³⁹ and ¹⁴⁰
Henequen	1–1.3	450–600	10–17	10.5	3.5–6	Textile industries	11, ¹³² ¹³³ and ¹⁴⁰
Helicteres isora	—	544–1111	23.3–71.7	—	0.6–1.7	—	736
Jute	1–1.5	480–620	—	—	4–6.5	—	736
Jute	1.2–1.5	300–920	10–80	32	1.2–2	Construction, automotive, textile, building panels, packaging, door frames, geo-textiles and door shutters	11, 46, 132, 133 and 140–142
Kenaf	1.55	200–900	15–50	25	1.4–3	Construction, paper, mobile cases, bags, insulations, packaging, and textiles	11, 23, 132, 133, 139 and 140
Nettle	—	680	40	—	2	Food, medicals, cosmetics, and drug industries	143 and ⁷⁴²
Oil palm	1–1.6	80–248	0.5–3.2	2	17–25	Palm wine, food, medicine, boiler fuel, roofing, building materials, door frame and windows	11, 132, 133 and 743
Niagara	—	400–741	16.7–45.6	—	0.6–1.7	—	736
Oliver	—	461–899	20.9–55.5	—	0.8–1.7	—	736
PALF	0.8–1.6	180–1627	1.44–82.5	35	1.6–14.5	Textiles, carpets, mops and curtains	132, 140 and 743
Petiole	690	185	15	—	2.1	—	741
Piassava	1.4	134–143	1.07–4.59	2	7.8–21.9	—	744
Rachilla	650	61	2.8	—	8.1	—	741
Rachis	610	73	2.5	—	13.5	—	741
Ramie	1.0–1.55	180–1627	1.44–82.5	35	1.6–14.5	Textiles, packaging, fishing nets, household furnishings and paper manufactures	11, 132, 133, 139 and 140
Root	1150	157	6.2	—	3	—	745
Sea grass	1.5	453–692	3.1–3.7	—	13–26.6	—	741

Table 9 (Contd.)

Fibre	Density (g cm ⁻³)	Tensile strength (MPa)	Tensile modulus (GPa)	Specific modulus (approx)	Elongation (%)	Applications	Ref.
<i>Sansevieria ehrenbergii</i>	0.88	50–585	1.5–7.67	—	2.8–21.7	—	741
<i>Sansevieria trifasciata</i>	0.89	526–598	13.5–15.3	—	—	—	741
<i>Sansevieria cylindrica</i>	0.915	585–676	0.2–11.2	—	11–14	—	741
Sisal	1.33–1.5	363–700	9.0–38	17	2.0–7.0	Construction, automobile, doors, roofing sheets, paper and pulp, and shutting plates	11, 132, 136, 140, 144 and 743
Snake grass	0.887	278.82	9.71	—	2.87	—	741
Spata	0.69	75.6	3.1	—	6	—	741
Spider silk	—	870–970	12	—	17.5	—	78
Talipot	890	143–294	9.3–13	—	2.7–5	—	745
Tussah silk	1.3	250	6	—	34	—	78

properties and applications of different plant-based natural fibres is presented in Table 9.

8. Opportunities and challenges

In recent times, several challenges have emerged with the exploitation of petroleum products such as high cost and degradation of the environment. Consequently, researchers have focused on developing bio-composites as a replacement for petroleum-based products. The extensive overview of jute fibres and their composite processing, properties and characterization described in this review offer insight into opportunities for the researchers in this field. Future research on jute fibres and their biocomposites should focus on the following aspects:

➤Significant research is needed for the elimination of deficiencies seen in JFRCs including fibre uniformity; mechanical strength; interfacial bonding and reduced susceptibility to moisture; bio-degradability characteristics; manufacturing methods to increase fibre content and fibre orientation; and delamination behavior to optimize efficiency.

➤Jute fibres have a hexagonal cross-section, varying chemical composition and fibre structure, resulting in poor bonding to typical resins. Swelling and microbial attack make jute fibres vulnerable to dimensional instability. Exploration of the possibilities of using surface treatments to reduce these limitations of jute fibres is necessary. To make cost-effective and eco-friendly JFRCs, surface modification techniques must be improved. Also, research must be carried out to predict the appropriate modification techniques for suitable composite application.

➤The anisotropic nature of jute fibres and their composites may cause dimensional changes during machining. Care is required in the selection of the cutting conditions, machining parameters, tool dimensions, tool geometry, etc.

➤Modern methods for providing inter-ply fibre reinforcement have been developed using techniques such as knitting,

3D weaving or fabric-wide weaving and pinning. However, these methods are not economical, inefficient and have design-specific limitations, which can be solved by creating new material processing techniques.

➤The durability of JFRCs is a major unpredictable factor. Thus, new techniques or methods should be developed to investigate and evaluate their durability and biodegradability properties.

➤At the laboratory level, existing pre- and post-treatment JFRCs are appropriate, but they are not very suitable for commercial products. The length of these procedures needs to be reduced for use in large-scale manufacture.

➤Extensive research is needed to overcome impediments such as water intake and moisture absorption for long-term stability in outdoor applications, specifically in extreme weather conditions.

➤A composite comprised of three entities is vulnerable to reinforcement, matrix and interface failure. The failure of one may initiate failure in the other and the process occurring can be identified by the load needed to activate individual fibres. Composite failure should usually be controlled by the mechanism triggered by stress. Focus on important major aspects such as fibre treatment and resin/matrix bonding is imperative for location of possible fields for the application of JFRCs.

➤JFRCs are used in various sectors such as automobiles, aerospace, and electronics. However, more research has to be carried out for their application in the biomedical field as scaffolds. The further exploration of this topic of research and the industrial use of JFRCs will open a new pathway.

9. Summary and concluding remarks

The desire for more environmentally friendly materials in the modern world has caused researchers to focus on natural cellulosic fibers, which have successfully replaced synthetic fibers in a variety of diverse uses. The main aim of this review was to get the attention of materials researchers to explore the

potentiality of jute fibres as an alternative to replace synthetic reinforcements in composites. Jute fibres show exceptional properties and their ecological considerations are attractive features for stimulation of the urge to use jute as a reinforcement. This review is the collective source of preceding experimental effort and meticulous view of jute fibres from their processing and properties to their product level for various applications. Their processing methods have been well recognized with adequate analysis of several key properties. According to this review, they can be summarized as follows:

>Jute fibres obtained by the retting process have higher strength compared to other processes. Hence, the retting process is recommended for extracting the fibres from plants.

>Jute fibre has been chemically treated with various reagents for a reduction in cellulose hydrophilicity. These treatments have been found to strengthen the adhesion between the fibres themselves, as well as the fibres and matrix.

>Surface treatment can help in the removal of the hemicellulose, lignin, and pectin that cover the outer surface of fibre cell walls, leading to the breakdown of the cell structure and the separation of the fibers into smaller filaments.

>Composites with jute fibre reinforcement can be produced using several methods. Among them, many researchers have used hand lay-up, compression moulding, vacuum bag moulding, resin transfer infusion and pultrusion processes.

>Correct selection and monitoring of process parameters, for example, working temperature, load, time, relative and humidity, are necessary pre-requisites for the successful manufacture of JFRCs.

>The fibre surface decomposition, resin flow pattern, void formation, cross-linking of fibres, and wetting conditions are the major parameters that have an effect on the properties of the prepared composites.

>Substantial improvements in the properties of surface-treated JFRCs relative to untreated JFRCs are viable.

>The mechanical strengths of JFRCs increase initially with the addition of the fibres, and then decrease when the fibre content exceeds 50%.

>Tensile and flexural strength and their moduli impact the energy absorbed and the hardness of JFRCs can be improved with an increase in fibre content.

>The addition of jute fibres to polymer matrices increases the wear characteristics of the composites by a reduction of 3.5–45% in the friction coefficient.

>DMA tests confirm the modification in the fibre/matrix interphase by the addition of compatibilizers and chemical treatment of the fibres.

>Breakdown of the jute fibres under stress and voids between the fibre and matrix was shown by electron microscopy observation, suggesting the probability of enhancement of the reliability fibre/matrix interface.

>LCA results show the use of resin as the most influential factor on environmental impact. The comparison of the environmental cautiousness in JFRCs showed that they are better than SFRCs.

>FEA results show the occurrence of high correlation between experimental and theoretical results.

>The conclusion from the machining of JFRCs is that responses such as thrust force, torque, and surface roughness are highly affected by the speed of cutting, depth of cut, feed rate, geometry of the tool and material.

Abbreviations

AE	Acoustic emission
AFM	Atomic force microscopy
ATR	Attenuated total reflectance
DSC	Differential scanning calorimetry
DMA	Dynamic mechanical analysis
DMTA	Dynamic mechanical thermal analysis
FEA	Finite element analysis
FTIR	Fourier transform infrared spectroscopy
HDT	Heat deflection temperature
He	Helium
IFSS	Interfacial shear strength
ILSS	Interlaminar shear strength
JFRCs	Jute fibre-reinforced composites
LCA	Life cycle assessment
MAgPP	Maleic anhydride grafted polypropylene
NFRCs	Natural fibre-reinforced composites
N ₂	Nitrogen
O ₂	Oxygen
PFRCs	Plant fibre-reinforced composites
POM	Polarized optical microscopy
PLA	Poly(lactic acid)
PP	Polypropylene
KMnO ₄	Potassium permanganate
SEM	Scanning electron microscopy
NaOH	Sodium hydroxide
TGA	Thermogravimetric analysis
TEM	Transmission electron microscopy
UTM	Universal testing machine
XPS	X-ray photoelectron spectroscopy

Conflicts of interest

There are no conflicts to declare.

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