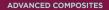
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Plant and Animal Based Composites

Edited by Kaushik Kumar and J. Paulo Davim

DE GRUYTER

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Preface

The editors are pleased to present the book *Plant and Animal Based Composites* under the book series **Advanced Composites**. The book title was chosen as it depicts the upcoming trends in composite materials for the next decade. This book is a compilation of different plant- and animal-based composites or in common words, *natural composites*. Emphasis is on the achievements, progress and recent developments and their applications.

The increasing demand for environmentally friendly materials and the need for cheaper fibers that increase the desirable mechanical properties force us to search for natural products. Natural fibers/fillers are largely divided into two categories depending on their origin: plant based and animal based. The fibers, in case of plantbased composites, can be extracted from the various parts of the plant, that is, bark/stem, leaves, fruits and so on, whereas, in the case of animal-based ones, the same can be from the scale (fish), skin (animals), feather (birds, chicken, etc.), eggshell, shell (mollusks, sea urchins, crustaceans, turtles and tortoises, armadillos, etc.), animal waste and so on.

One of the major disadvantages of the usage of these fibers is moisture absorption. Most of the nature-based fibers or fillers are hydrophilic in nature owing to the presence of functional groups such as hydroxyl in their structure and also being porous. Hence, they absorb a considerable amount of moisture from the surrounding environment. The high moisture absorption leads to a number of problems when used as reinforcement materials such as:

- (1) Fibers and fillers swell due to the absorption of moisture and shrink when moisture is removed due to the dry atmosphere and elevated temperatures. This frequent swelling-shrinking phenomena leads to the formation of cracks and hence leads to reduced mechanical performance and durability of the composites.
- (2) High absorption of alkaline solution present within the matrix leads to the degradation of fibers with time. These result in deterioration of the properties of the composites.
- (3) Moisture absorption of fibers and fillers leads to breakage of hydrogen bonds between the fiber and matrix and, therefore, weakens the fiber/matrix interface and, consequently, it deteriorates the mechanical strength.

Therefore, a number of fiber surface treatment methods are required to be undertaken to reduce the moisture absorption of fibers for applying them in composites.

At present, *Plant and Animal Based Composite* is a major discipline and many researchers and scholars are working in these areas. This book provides insight for all researchers, academicians, postgraduate or senior undergraduate students working in the area. The chapters in the book have been provided by researchers

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and academicians working in the field and have gained considerable success in the field.

The chapters in the book have been categorized into **three parts**, namely, **Part I: State of Art; Part II: Plant-Based Composites; and Part III: Animal-Based Composites.**

Part I contains Chapters 1–3, whereas Part II has Chapters 4–7 and Part III with Chapter 8.

Part I starts with **Chapter 1**, which provides the readers an insight into recent developments in plant and animal fiber-reinforced composite. The chapter reviews the recent reports about the properties, fabrication processes, applications and advancements in the field of composites fabricated with different biodegradable plant and animal fiber-reinforced composites. Various types of matrix and reinforcements that are available to be used with biopolymers for best maintainable green composites have also been addressed. These natural composites can be created at a low cost, have practically identical mechanical properties with synthetic composites and maintain a balance between ecology, technology and the economy.

Chapter 2 elaborately deals with the advances in animal/plant–plastic composites. This chapter aims at exploring the preparation, characterization and applications of plant/animal fiber-reinforced polymer composites. The chapter starts with a thorough overview of the various forms of plant/animal fiber-reinforced polymer composites followed by examples of such composites and their promising future in terms of research and development and their application in domestic and engineering products. Considering forest conservation and the efficient use of agricultural as well as other renewable resources, such as solar, wind and tidal energy, the use of renewable materials, such as plant/animal fiber-reinforced polymeric composites, is slowly becoming a key design requirement in the design and development of parts for a wide range of industrial products. An extensive research into such composites can lead to an even greener and healthier environment, to some extent.

Again, in **Chapter 3**, the last chapter of this part, a detailed review of the ductile attributes of natural fiber composites has been put forward. Normal fiber-strengthened composites are a developing territory in the polymer discipline. Hence, this chapter presents a detailed effort on the characteristics of natural fiber composites with an exceptional testimonial to the types of fibers, fabrication method and ductile attributes. This is a very pertinent problem as these natural fiber composites are biodegradable and noncoarse, and they exhibit the explicit attributes that are practically identical to those of regular fiber composites. Due to their low cost, equally great mechanical attributes, they have gained a lot of ground and the ductile characters are essentially impacted by the hook-up attachment among the matrix and the fibers.

Chapter 4, the next chapter of the book and the first chapter of **Part II**, provides the reader with an exclusive study of plant-based fibers and resins in composites. In the present era, the use of sustainable crude materials in the composite

industry is turning out to be progressively well known in view of the natural aftercare and the necessity to replace fossil resources. Plant oils with triglyceride spines can be artificially altered and used to integrate resin from inexhaustible assets. Plant oils contain a dominance of triglyceride, the glycerol esters of unsaturated fats. Five important types of unsaturated fats of the unsaturated fat chain are obtained as one of the outputs from the hydrolysis process of triglyceride with 16 to 18 carbons with 0 to 3 times the securities. The five unsaturated fats are oleic, linolenic, palmitic, linoleic and stearic acids. The percentage of specific unsaturated fat varies in different plant oils and also in similar plant oils. The variation depends on the plant species, weather and plant growth states. This chapter puts emphasis on plant-based fibers, resins and fillers derived from different plants.

In **Chapter 5**, an orthopedic application of plant-based composites has been described. Orthotic calipers have been fabricated and tested with plant-based composites. The main objective of the chapter was to use bark-based and fruit-based reinforcement with epoxy as the matrix to create the braces for orthotic calipers. The void content and mechanical behavior, including tensile and flexural test, were performed and then compared with the currently used materials. After comparison, it is observed that the specific strength and stiffness of bark-based reinforced composite are better than that of the presently used aluminum alloy.

In **Chapter 6**, the next chapter of the book, the content experimentally illustrates the mechanical potential of the polypropylene, jute and coir fiber-based composites. Three types of composite specimens – polypropylene, polypropylene reinforced with jute and polypropylene reinforced with jute and coir composites –, are prepared using the injection molding method. The experimental results show that the incorporation of jute and coir fibers into the polypropylene matrix resulted in improvement of the tensile, flexural and impact properties of the composites.

Chapter 7, the last chapter of the section, provides the mechanical properties of a very common plant-based composite, *rice straw fibre reinforced with epoxy*. The chapter discusses the preparation of different types of rice straw fiber with epoxy composite: rice straw fiber has been used without any surface treatment and treated with hot water and NaOH with different volume fractions. The fibers are used in aligned and cross-conditions with different layers. Mechanical properties as per the standard have been analyzed and compared.

The only chapter of **Part III** and the last chapter of the book, that is, **Chapter 8** presents the thermomechanical properties of forcespun polycaprolactone (PCL) fibers infused with fish scale-based hydroxyapatite (HA). This chapter highlights the usage of animal-based reinforcement. In one word, this chapter deals with a completely biodegradable composite, as both the matrix and the reinforcement are biodegradable. HA is a biomaterial with excellent characteristics that is suitable for biomedical applications. In this chapter, HA is synthesized from carpa fish scales by the calcination method and nanomilled for particle size reduction, and these particles were characterized using X-ray diffraction, scanning electron microscopy

and transmission electron microscopy. A novel forcespinning technique was used to fabricate microfibers from PCL infused with synthesized HA. Thermomechanical properties of the PCL/HA fiber mats were investigated using differential scanning calorimetry, dynamic mechanical analysis and tensile tests. The analyses suggest that infusing the HA in moderate quantities will enhance the thermomechanical properties of the composite fibers.

First and foremost, we would like to thank God. It was God's blessing that provided us the strength to believe in passion, hard work and pursue dreams. We thank our families for having patience with us for taking yet another challenge that decreased the amount of time we could spend with them. They were our inspiration and motivation. We would like to thank our parents and grandparents for allowing us to follow our ambitions. We would like to thank all the contributing authors as they are the pillars of this structure. We would also like to thank them for their belief in us. We would like to thank all of our colleagues and friends in different parts of the world for sharing their ideas in shaping our thoughts. We will be satisfied with our efforts when professionals connected with all fields related to lightweight materials get benefitted.

We owe a huge thanks to all of our technical reviewers, editorial advisory board members, book development editor and the team of **Walter de Gruyter GmbH** for their availability to work on this huge project. All of their efforts helped to make this book complete and we could not have done it without them.

Last, but definitely not least, we would like to thank all individuals who have taken the time out and helped us during the process of editing this book, without whose support and encouragement we would have probably given up the project.

> Kaushik Kumar J. Paulo Davim

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Nutenki Shravan Kumar, Tanya Buddi, Anitha Akkireddy and K. V. Durga Rajesh **Recent developments in plant and animal fiber-reinforced composite**

Abstract: Natural fiber composites are replacing conventional materials because of their eco-friendly and biodegradable nature. They are a renewable source and are preferred over artificial composites. A composite is a material that is a combination of two or more components or materials that together form a new one with higher strength. Natural fiber composites are materials fabricated by using either any one or both of the matrix and reinforcement, from natural sources that are biodegradable. In this chapter, the recent reports about the properties, fabrication process, applications and advancements in the field of composites have been discussed. Various types of matrices and reinforcements that are utilized with biopolymers for best maintainable green composites have also been discussed. These natural composites that have practically identical mechanical properties as synthetic composites maintain a balance between ecology, technology and economy which can be created at a low cost. This helps replace nonbiodegradable composites and are beneficial to the consumer.

Keywords: Natural fiber composites, biodegradable, matrix, reinforcement, green composites, nonbiodegradable composites

1 Introduction

Composite materials consist of two or more than two material phases combined together to form a new material that has superior properties than its individual constituents. The material constituents are combined at a macroscopic level and are not soluble in each other. The main difference between alloys and composites is that in composites, material constituents are not soluble in each other and each individual constituent retains its properties, but in alloys, material constituents are

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soluble in each other and form a new material that has properties different from those of their constituents. Composites are divided into polymer matrix, ceramic matrix and metal matrix.

Technically, the most significant composite materials are accessible as disorganize fiber. The quality of the fiber-fortified composite materials depends on the firmness based on weight. Quality is explicitly characterized by the proportion of solidity to thickness. The length of fiber impacts the mechanical properties of the material. Long persistent filaments are not difficult to process and arrange, but short strands can not be completely controlled to situate appropriately. The filaments utilized in business are, for the most part, of different types of carbon, glass, Kevlar and graphite. Each of these filaments is fused into a network of either stopped or nonstop length. The lattice materials may be elastic or a plastic polymer, ceramic or metal. Cover of required size is obtained by stacking a number of slight sand fiber lattice layers and merging together to achieve the necessary thickness. The direction of fiber in each layer is controlled for obtaining a wide scope of mechanical and physical properties of overlaid composite [1, 2]. The board classification of fibers is shown in Fig. 1.

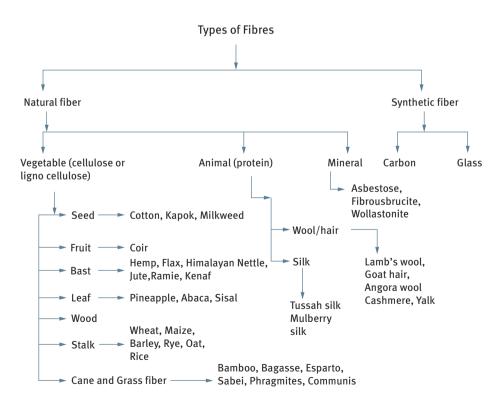


Fig. 1: Types of fibers.

1.1 Green composites

Green composites refer to a combination of biopolymers and natural fibers. They have numerous points of interest compared to polymer composites, the state of the composite is well decomposed, such as biodegradability [3] and ease in fabrication. They have favorable mechanical properties as a result of which they find their place in various segments, for example, electronic items packaging, inside vehicle parts and so on. Here, different blends of common fibers and biopolymers and their assembling procedures are discussed. Expanded bowing and elasticity can be acquired when PLA network is strengthened by kenaf fiber, and youthful modulus is accomplished by adequate increment, when the fiber content is increased by half [4]. At the point when PLA and bamboo fiber are blended, it brings about expanded twisting quality [5]. Along these lines, these biopolymer and regular fiber composites blend have great potential, and they will supplant nonbiodegradable composites because of their favorable mechanical properties.

1.2 Natural fiber

Fiber is utilized as reinforcement in composites to shoulder the heap moved equally through the lattice and it provides firmness and strength to the polymer [6, 7]. Fibers are obtained either naturally or artificially. Fibers obtained naturally are classified into three categories: mineral fiber, plant fiber and animal fiber.

- Animal fibers are fibers which can be acquired from creatures for instance, sheep wool, mammal hair, silk and goat hair [8].
- Silk fiber is, likewise, a creature fiber which has very high rigidity among all regular natural fibers [9, 10].
- Mineral strands are normally occurring or marginally adjusted fibers that are obtained from minerals. Asbestos and earthenware are usually known as mineral filaments. Mineral fibers are, likewise, apt for working in high temperature conditions [11, 12]. These are also used as an troublesome material.
- Plant fiber is a rich fiber among regular fibers. Himalayan nettle, hemp, sisal, kenaf, jute, abaca, flax, ramie, etc. are common plant fibers. Plant fibers are also called cellulosic fibers and they have high rigidity [13].
- Banana fiber is composed mainly of carbohydrates and proteins that make it a high strength fiber; however, it needs initial treatment [14].

Fiber selection is very critical for the development of green composites, and is essential to process great mechanical properties like firmness and elasticity. Different properties such as warm security, failure elongation, matrix and fibers attachment, over lasting and dynamic conduct, rate of moisture absorption and low manufacturing cost are significant in the choice of fiber. Characteristic fibers have their own significance in chemical properties when chosen for e fabrication, and it forms the basis for prediction of the final characteristics of fabricated fiber composite [15]. The chemical constituent of natural fiber mostly affects mechanical, physical, tribological and thermal properties. Tensile strength of a few natural fibers is shown in Table 1.

S. no.	Fiber	Tensile strength (MPa)
1	Hemp	690
2	Flax	345-1,500
3	Sisal	468–700
4	Ramie	400-938
5	Kenaf	295–1,191
6	Jute	393-800
7	Spider silk	1,300–2,000
8	Coir	131-220
9	Cotton	287-800
10	Bamboo	140-230
11	Wool	50-315

Table 1: Tensile strength of natural fibers.

The merits of natural fibers are that they are nontoxic, compostable, nonabrasive and lightweight. They possess insulating property, are an inexhaustible and interminable stockpile of crude materials, are liberated from wellbeing hazards (no skin disturbances), and display adequate explicit quality, excellent sturdiness and warm properties. They are an income source for the rustic/horticulture network. The demerits are: less similarity with hydrophobic polymer framework, degradation of fiber due to storage over a long time period, moisture absorptivity, propensity to frame total during handling, relatively low warm security and less protection from dampness and hygroscopicity.

2 Classification of natural fiber composites

2.1 Plant fiber

Fibers acquired from the plants are called plant fibers. They are available everywhere in the plant – from roots to leaves, stems and in the soil. These fibers are collected from the plant part for business or potential neighborhood use. Presently, we discover increasingly more about helpful impacts of fiber in natural products, vegetables and grains and they are known as dietary strands [16, 17]. Filaments are delicate or hard strands, surface strands or endogenous fibers. Normal fibers need to be prepared to make them appropriate for various purposes. Various types of fibers obtained from plants are presented here.

2.1.1 Bamboo fiber-reinforced polymer

Bamboo is one of the freely available resources in forests as well as an agricultural crop and can be used in polymer composites. It is one of the natural resources which can grow in less time. But it has not been utilized to the full extent possible. Traditionally, it has been used in different tools due to its high strength-to-weight ratio. This property is because of the arrangement of filaments in longitudinal direction. In practice, the bamboo-based composite needs to be manufactured, notwithstanding the extraction of bamboo fiber from bamboo trees in a controlled manner. Naturally, bamboo fiber has good mechanical properties; however, it is weak in nature as compared to other natural fibers because of the extra lignin content covering the bamboo fibers. Now, bamboo is thought of as a significant fiber available from plants and moreover, it shows great potential in polymer composite industries. Its auxiliary variety, fiber extraction and mechanical properties, thermal properties and synthetic alteration have made it adaptable to be used in composite industry [18–23].

2.1.2 Fiber extraction

The bamboo fiber is obtained from bamboo trees; it is separated into two types of fiber using diverse procedure stream and strategy – natural unique bamboo fiber and bamboo mash fiber (in particular, bamboo thick fiber or recovered cellulose bamboo fiber). Unique bamboo fiber is legitimately obtained from characteristic bamboo with no synthetically added substance, using physical and mechanical strategy. In order to distinguish it from bamboo mash fiber (bamboo thick fiber), we call it "unique" or "unadulterated" normal bamboo fiber. In any case, bamboo mash (thick) fiber has a place with recovered cellulose fiber as synthetic fiber. There are two kinds of handling to acquire bamboo filaments – mechanical handling and synthetic preparing. The two procedures at first incorporate parting of bamboo strips, followed by mechanical preparing or synthetic handling, contingent on the further utilization of bamboo filaments [22, 23]. The microscopic structure of the bamboo fiber is shown in Fig. 2. The properties of bamboo fiber as given in Table 2.



Fig. 2: Extracted photograph of bamboo fiber using microscope [23].

S. no.	Property	Value		
1	Density	1.18 g/cm ³		
2	Soften point 57 °			
3	Water absorption	Water absorption 2%		
4	Tensile strength	0.0103 KPa		
5	Modulus elasticity 0.4145 K			

Table 2: Properties of bamboo fiber.

2.1.3 Banana fiber-reinforced polymer

Banana fiber is a cellulosic fiber obtained from the pseudo stem of banana plant. This fiber generally exhibits good mechanical properties. Banana fiber exhibits great explicit quality properties similar to those of conventional material and glass fiber. It has lower thickness than glass filaments [24]. The pseudo stem is a barrel-shaped, grouped conglomeration of leaf stalk bases. Banana fiber squander is a result of development and has not been appropriately used. Proper use of such filaments would generate adequate interest which will be reflected in reduction of costs. Banana strands have profound qualities and offer incredible possibilities: they are light in weight, stretch little, are imperviousness to fire, have good dampness ingestion and are biodegradable. Banana fiber has perceived use in clothes and home decorations [14, 25].

Banana fiber is of exceptional interest in making high quality paper. It is also is used in making items like channel paper, paper packs, welcome cards, light stands, pen stands, enlivening papers, rope, mats, composite material, and so on Banana fiber is used in making currency notes in Germany and had a preliminary run in India, too. Polypropylene strengthened with banana fiber is used by vehicle organizations in making under-floor security boards for high-end vehicles like Mercedes. Banana fiber is also used in painstaking work such as home decor. The composite material of banana fiber is used in structural sheets as they are impervious to fire. Studies have shown that paper made out of this fiber has a life of more than 100 years, as it is the most grounded of the long strands as compared to other discovered characteristic filaments, which would collapse 2,000 times [26].

S.M. Sapuan et al. [2] examines the mechanical properties of woven banana fiber-fortified epoxy composites. The examinations of elastic and bending tests are done using regular fiber with composite materials. From the outcomes, it has been discovered that the most extreme estimation of worry in an x-axis is 14.14 N/mm²; meanwhile, the most extreme estimation of worry in y-axis is 3.398 N/mm². As for modulus of elasticity, the estimates of 976 MN/m² in x-axis and 863 MN/m² in y-axis have been computed. With respect to the instance of three-point bending (flexural), the maximum load applied is 0.03625 KN.

Samrat Mukhopadyay et al. [24] concentrated on the different diameters of banana fibers. Hundreds of strands are picked at random from the assortment of banana strands. The distance across the strands varies from 8 cm to 32 cm. The class interim of 2.9 cm shows that the standard variation has diminished with an expansion of distance across the strands implying that courser filaments are more normal in nature. Most of the strands fall in the distance across range of 17 to 19 cm. Consequently, such strands are picked for malleable testing. Aftereffects of elastic testing showed that strain rates assumed a significant part in the nature of the pressure strain bends, the quality of the strands and the idea of disappointment. The Mechanical and physical properties of banana fibers are tabulated in Table 3.

S. no.	Property	Value
1	Diameter	0.080-0.25 mm
2	Length	100–500 cm
3	Moisture content	60%
4	Tensile strength	0.529–0.914 KPa
5	Sp. tensile strength	0.392–0.677 KPa
6	Strain at failure	1–3%
7	Density	950-750 kg/m ³
8	Modulus elasticity	0.027–0.032 KPa
9	Sp. Young's modulus	0.020–0.024 KPa

Table 3: Mechanical and physical properties of banana fiber.

2.1.4 Jute fiber

Jute fiber is a type of fiber acquired from jute plants containing three principle classifications of substance mixes in particular: cellulose of (58–63%), hemi cellulose (20–24%) and some other little amounts of constituents like fats, gelatin, fluid concentrate, etc. Jute fiber is made of little units of cellulose solidified by hemicellulose and lignin [27–29]. The low cellulose content, coarseness, solidness, low extensibility, low hold execution and other properties genuinely confine the turning capacity of crude jute fiber. Progressions of wet compound handling groupings are expected to improve the turn capacity of jute. The characteristics of the fiber and yarn (as shown in Fig. 3), for the most part, rely upon the degumming impact. From the perspective of wood substitution, jute composites could be a perfect arrangement [30]. With regularly draining timberland holds, a composite dependent on inexhaustible assets is now ready to enter the market.



Fig. 3: Jute fiber.

Indigenous wood supply for pressed wood industry having been halted for all intents and purposes, and with expanding landed expense of imported compressed wood facade, the jute composite sheets generally offer excellent incentive for the clients with no trade-off in properties [31]. Jute-coir sheets have found better application over pressed wood sheets in railroad mentors for sleeper compartment backing, for building insides, entryways and windows as also in transportation division as support for seat and backrest. Run-of-the-mill jute composites sheets do not demonstrate well on the ground because of their dampness assimilation and screw holding quality. Point by point assessment of the jute-coir board tests has been completed for their applications in compartment support and segments in railroad mentors; the outcomes fit in with the railroads' prerequisites. The use of jute fiber tangles in blend with polymer films possibly offers a fast and direct strategy for gathering composites through movie marking, warming and press combination [31–35]. As seen in Figs. 4 and 5, with an increase in fiber proportion, there is reduction in compressive strength and split tensile strength. Mechanical and physical properties of jute fibers are given in Table 4.

S. no.	Property	Value
1	Cellulose	64.4%
2	Hemi cellulose	12%
3	Lignin	11.1%
4	Moisture content	1.1%
5	Density	146 × 10 ³ kg/m ³
6	Fiber length	8–60 cm
7	Diameter	50–250 cm
8	Tensile strength	0.4–0.8 KPa
9	Modulus of elasticity	0.010-0.030 MPa
10	Elongation at break	1.8%

Table 4: Mechanical and physical properties of jute fibers.

Fiber proportion v/s compressive strength

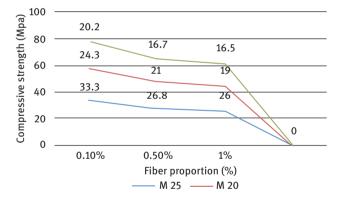


Fig. 4: Fiber proportion vs. compressive strength.

2.1.5 Sugarcane bagasse

Composite materials are also called course of action materials or curtailed to composites. These materials are developed using at least two constituent materials with fundamentally unique physical or manufactured properties [36]. When we combine these materials, a material with qualities that are different from those of the separate parts is produced. Composite materials that contain solid burden conveying material are known as support, and those imbedded in more vulnerable materials

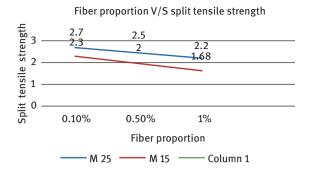


Fig. 5: Fiber proportion vs. split tensile strength.

are called grid. Fortification gives firmness and quality, assisting with supporting basic burden. Bagasse is the bio result of sugarcane fiber that is left after the juice has been separated from the sugarcane. Bagasse strands are common fiber items and they biodegrade in 3–9 weeks [37]. Bagasse filaments are good fibers like banana strands. They comprise water, filaments and limited quantities of solvent solids. The percent composition of each of these parts may change as per the development, assortment, reaping and the proficiency of the crushing plant. For every 10,000 kg of sugarcanes squashed, a sugar-manufacturing plant creates almost 3,000 kg of wet bagasse.

Bagasse is, for the most part, utilized as a crude material that is consumed in the sugarcane plant heaters. Bagasse has low caloric force, which makes the process low in effectiveness. Ceramics and plastics have been the prevailing and emerging materials. The volume and quantities of use of composites have developed consistently, infiltrating and penetrating new markets consistently [38, 39]. Current composites find their use from normal things to modern applications. While composites have just exhibited their incentive as weight-saving materials, the current test is to make them functional.

E.F Cerqueira et al. [36] assessed the impact of chemical adjustment on the mechanical properties of sugarcane bagasse fiber or polypropylene composites in light of the fact that the common fibers show some disadvantages – for example, the inconsistency between fibers in polymer frameworks, the inclination to shape totals during handling and the poor protection from dampness reduce the use of regular strands as fortification in polymers. To overcome this issue, a few strategies for surface alteration are used to improve strands and polymer frameworks similarity, similar to the compound treatment discussed in his paper in which composite is pretreated with 10% sulfuric corrosive arrangement, followed by delignification with 1% sodium hydroxide arrangement. The Mechanical and physical properties of sugarcane bagasse are tabulated in Table 5.

S. no.	Property	Value
1	Cellulose	45-55%
2	Hemi cellulose	20-25%
3	Lignin	18-24%
4	Pectin	0.6-0.8%
5	Ash	1-4%
6	Moisture content	49%
8	Fiber length	8–28 cm
9	Diameter	100–340 cm
10	Tensile strength	0.180–0.290 KPa
11	Extractives	1.5-9%

Table 5: Mechanical and physical properties of sugarcane bagasse.

2.1.6 Coconut fiber

Experts, the world over, have recognized the future requirement for development materials that are light, solid, easy to utilize, economical, but more earth-feasible [40]. One of the recommendations in the vanguard has been the sourcing, advancement and utilization of nonordinary nearby development materials including the possibility of using some horticultural squanders as development materials. Characteristic strengthening materials can be acquired with little to no effort using nearby labor and innovation [41–45]. Use of regular filaments as a type of solid improvement is specifically helpful in less evolved districts where ordinary development materials are not promptly accessible or are prohibitively costly [46–48].

Coconut fiber is separated from the external shell of a coconut. The normal, logical name and plant group of coconut fiber are coir, *Cocos nucifera* and Arecaceae (palm), respectively Coconut development is seen in the tropical areas of East Africa and Asia. Coconut strands have favorable properties: for example, they are impervious to growths and spoil, give incredible protection against temperature and sound, are not effectively burnable, are fire resistant and are unaffected by dampness and moisture. They are intense and strong, flexible, spring back to shape considerably after steady use, are absolutely static free and simple to clean [49–54]. T. Balarami Reddy [46] explains that the mechanical properties, viz., tensile strength of the green coconut fiber-reinforced HDPE composite material is extraordinarily affected by fiber length as well as fiber volume fraction; and the rigidity of the composite material decreases with increment in the fiber length [55, 56]. The Mechanical and physical properties of coconut fiber are shown in Table 6.

S. no.	Property	Value
1	Cellulose	33.61%
2	Hemi cellulose	8.50%
3	Lignin	36.51%
4	Pent sans	29.27%
5	Ash	0.61%
6	Total water soluble	26%
7	Modulus of elasticity	856.849 MPa
8	Load at break	1082.634 N
9	Tensile strain at break	0.0423 mm
10	Extension at break	4.117 mm

Table 6: Mechanical and physical properties of coconut fiber.

3 Animal fiber

Animal fibers are one of the most widely utilized natural fibers, after green fibers. For the most part, they contain proteins and can be potential fortifications in polymers. Examples of this fiber include fleece fiber acquired from sheep, goats, lamas, bunnies, musk bulls, and so on [57]. Thus, silk, quills and hair are acquired from different sources. In this segment, a few significant creature fibers are discussed.

3.1 Human hair

Hair is a protein fiber that develops from follicles found in the dermis or skin. It is one of the characterizing qualities of well evolved creatures. Human body, aside from the territories of brilliant skin, is shrouded in follicles which produce thick, terminal and fine villous hair [58]. Most regular enthusiasm for hair is centered around hair development, hair types and care; however hair is also a significant biomaterial essentially made of protein, prominently keratin. Regarding crude components, normally, hair is made of 50% carbon, 20% oxygen, 17% nitrogen, 6.3% hydrogen, and 5% sulfur. The part underneath the skin is called the hair follicle, and when it is pulled out from the skin then it is known as the bulb [59, 60].

Human hair shows acceptable strain; hence it can be used as a fiber-fortifying material. Hair giber is nondegradable and available in plenty, at an exceptionally modest cost. Human hair is treated as waste material in many parts of the globe and is a typical constituent found in metropolitan waste streams, causing many environmental. Additionally, the high rigidity, extraordinary substance arrangement, warm protection and so on make the hair fiber amenable for use as fortifying material [61–64]. Sreevani et al. [65] describe how as the percentage of hair fiber and time period increases, there is an increase in the compressive strength as shown in Fig. 6. Mechanical and physical properties of human hair are tabulated in Table 7.

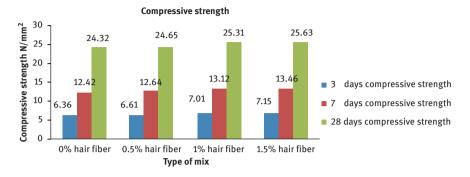


Fig. 6: Variation in compressive strength of beams using hair fiber with time period.

S. no.	Property	Value		
1	Fiber length	40 mm		
2	Diameter	60–100 mm		
3	Modulus of plasticity	3.5 GPa		
4	Linear density	13.20 kg/m		
5	Yield strength	74,340 GPa		
6	Breaking strength	119 MPa		
7	Strain at breaking	29%		
8	Tensile strength 384,7			

Table 7: Mechanical and physical properties of human hair.

3.2 Silk fiber

Silk is delicate, shiny, smooth, solid and tougher than any regular or fake fiber. The mechanical and business uses of silk promoted silkworm production throughout the world, particularly in developing nations [66]. Silkworms produce silk in order to fabricate covers that will insure them from natural dangers and predator assaults during their transformation. Therefore, casings of these silkworms should meet the desired requirements of good fiber. Studies have shown that cases ought to be considered as a permeable composite that comprises sericin network and fibroin fiber support. A few properties of these "permeable composites" are researched in a few studies [67, 68]. Arachnid silk shows a decent blend of high rigidity, solidness and adaptability. The

far-reaching mechanical properties of arachnid dragline silk are better than those of aramid fiber, one of the most grounded artificial fiber materials [69].

Silk filaments spun by a few types of arthropods have existed naturally for many years. The biological elements of the silk filaments are firmly identified with their properties. According to Kuna Cao and Yong Liu, the structure of the silk proteins will affect the properties of the fiber, and to acquire recovered silk strands of good quality, complete reproduction of the turning procedure of insects and silkworms is required [69, 70]. The level of shell for eri-cocoons is about 14% for all the spots, while crude silk proportion of mulberry is 13–14%. The mean estimation of fiber fineness of eri silk is 3.09 dtex from Bahir Dar, 2.91 dtex from Awassa and 3.11 dtex from Awash Melkassa, while fineness of mulberry silk is 2.1 dtex from Awassa and 2.4 dtex from Inundated Melkassa. Over 60% of the cases studied are acceptable [66]. Mechanical and physical properties of silk fibers has been tabulated in Table 8. Comparison between various natural silks are given in Table 9.

S. no.	Property	Value
1	Diameter	1–15 Lm
2	Mass density	1,250-1,350 kg/cm ³
3	Moisture absorption	5-35%
4	Tensile stiffness	5–25 GPa
5	Tensile strength	0.2–1.8 GPa
6	Sp. tensile stiffness	4–20 GPa/g cm ³
7	Sp. tensile strength	0.1–1.5 GPa/g cm ³
8	Tensile failure strain	15-60%
9	Toughness	0.025–0.250 kJ m ³
10	Sp. toughness	0.020-0.185 kJ m ³ /g cm ³

Table 8: Mechanical and physical properties of silk fibers.

Table 9: Comparison between various natural silks.

Fiber	Stiffness (N/mm²)	Strength (N/mm ²)	Elongation (%)	Toughness (kJ∙m³)
B. mori reeled silk.	15,000	700	29	0.150
B. mori cocoon silk	7,000	600	19	0.070
A. Diadematus silk (flagelliform)	3,000	500	275	0.150
A. Diadematus silk (dragline)	10,000	1,100	26	0.180

3.3 Bone ash particulate composites

The impact of particulate bone increments on the mechanical properties and tribological conduct of bone-fortified polyethylene composite is assessed to evaluate the chance of using it as another material for designing applications. Dairy animals' bone particles strengthened with reused low thickness polyethylene is obtained by replacing the bovine bone particles of 5–25 wt% with 5 wt% interim utilizing pressure technique. Wear of the polymers is led using nail circle machine by fluctuating pace, time and burden. Factorial structure test is performed according to standard 1.8 symmetrical exhibits, so as to study the plan parameters. Speed, burden and time together influence the dry sliding wear on the polymers.

Better outcomes are obtained at high fiber content weight%. Of all properties researched, 8 wt% gave better outcomes in bone debris and bone particulate strengthened composites. It shows the best pliability, and furthermore, has good flexural and hardness values [71, 72]. The linear conveyance of bone particles in the microstructure of dairy animals' bone-fortified RLDPE polymer is the main consideration during expansion and development in quality. Decreased porosity is seen with expanding measure of fortification. Impact of factors – that is, load and sliding velocity – are progressively articulated on the wear of the polymer instead of time. Factorial structure of the trial can be effectively used to portray the wear conduct of the examples and the created direct condition models can be used to forecast the wear pace of the material under the chosen trial conditions [73–75]. The Elemental composition of bone ash is given in Table 10.

Metal	Fe	Ca	Au	Sr	Sn
Composition (%)	2.60	23.32	Nil	Nil	Nil

Table 10: Elemental composition of bone ash.

The mechanical properties of epoxy polymers strengthened with particles of bone are listed in the table – the relationship between the wear volume loss and chosen mechanical properties, for example, extreme elasticity, lengthening and hardness have been accounted for in single pass investigations of composites [73]. Mechanical properties of bone ash are tabulated in Table 11.

The SEM highlight of worn surface of 0 wt%, 3 wt%, 6 wt% and 9 wt% polymer at a heap of 0.03 KN and 8×10^3 m sliding separation of steady speed at 1.884 m/s is shown in Figures 7 and 8 and includes framework break, stripping of network, fiber-lattice de-holding, and shear deformation of the fortification with the assistance of SEM micrograph [73, 74, 76, 77]

	Pure epoxy	Epoxy with 3 wt% bone particles	Epoxy with 6 wt% bone particles	Epoxy with 9 wt% bone particles
Tensile strength (KPa)	0.38	0.38	0.39	0.39
Hardness	81.5	82.5	83.5	85.5
Extension (cm)	32	32.6	32.9	33.2

Table 11: Mechanical properties of bone ash.

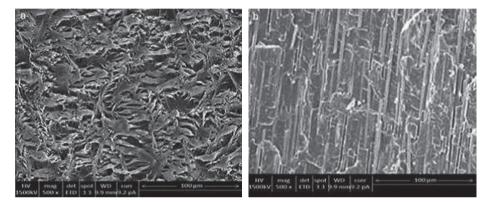


Fig. 7: (a) 3 wt% composite (b) 6 wt% composite.

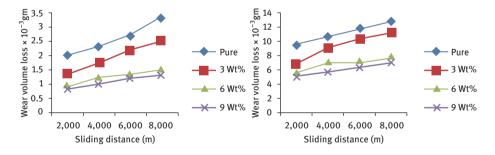


Fig. 8: Loss of volume due to wear at a sliding distance of 10N and 30N.

4 Conclusion

Among the different categories of reinforcements, fibers are stronger and stiffer materials than any other type, which is the key explanation for the overwhelming attraction of fibrous reinforcements. Different forms of fibers used in composites have different properties and thus influence the composite properties in various ways. Most reinforcing fibers have far greater mechanical properties than those of un-reinforced resin systems. Consequently, the mechanical properties of fiber/resin composite are dominated by the fiber's contribution to the composite.

In the present times, natural composites can be created at a moderate expense with equivalent mechanical properties of synthetic fibers. These are biodegradable composites, so it can keep up a balance between environment, economy and innovation; this can assist with supplanting the oil-based composites, and as a result, bring about shopper benefits. Alternate natural fibers may be considered to get the best choice for the required conditions.

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Harrison Shagwira, Thomas O. Mbuya and F. M. Mwema Advances in animal/plant-plastic composites: preparation, characterization and applications

Abstract: This book chapter aims at exploring the preparation, characterization and applications of plant/animal fiber-reinforced polymer composites. The book addresses several key research work that have been happening as well as some of the drawbacks that affect the application of these composites in real-life applications. Each chapter begins with an overview of the various forms of plant/animal fiber-reinforced polymer composites, followed by examples of composites made up of plant/animal fibers and their promising future in terms of research and development and application in domestic and engineering products. Today, man has realized that if the environment is not preserved, the over-consumption of naturally existing resources and a drastic reduction in the amount of fresh air generated in the world would endanger him. Forest conservation and the efficient use of agricultural as well as other renewable resources such as solar, wind and tidal energy have already become critical issues globally. With this kind of concern, the utilization of renewable materials such as plant/animal fiber-reinforced polymeric composites is slowly becoming a key design requirement for the design and development of parts for a wide range of industrial products. An extensive research into such composites can, to an extent, lead to an even greener and healthier environment.

Keywords: natural fiber, composite, matrix, reinforcement, polymer

1 Introduction

Composites materials are usually produced when two or more materials are combined, where the main material acts as the matrix phase (ceramic, metal or polymer), while the second material forms the reinforcing phase (particles, fibers or sheets). Generally, the composite materials are identified by the type of reinforcing material, such as cement-matrix composites, polymer composites, metal-matrix composites, etc. [1]. Many of the commercially produced composites are largely the polymer-matrix composites, where polymeric resin forms the matrix with various additive materials being used as the reinforcing materials.

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Polymer materials categorized as thermoplastics such as polyether ether ketone (PEEK), polypropylene (PP), polyvinyl chloride (PVC), polyethylene (PE), epoxy, polyolefin, polystyrene (PS), etc. as well as thermosets such as phenol–formaldehyde (PF) resin, polyester and so on can be reinforced by the use of various types of fibers such as plant or animal fibers. Discussing the cement-matrix composites, concrete/ cement is reinforced with materials such as steel to enhance the mechanical properties and is used in buildings and construction applications. Metal-matrix composites are made up of a metal as the material to be reinforced with other additives such as organic compounds, ceramic or metal.

With increase in population, naturally existing materials are increasingly used as substitutes to artificially made materials. This has resulted in attention to the use of plant/animal fibers in reinforcing and strengthening of composites. Plant/animal fibers have a number of important advantages when compared to synthetic fibers. Today, different kinds of naturally occurring materials are being explored for their use as fibers in composite materials; these include fibers like barley, jute straw, paper mulberry, hemp, wheat, coir, water hyacinth, wood, oats, flax, rye, rice husk, kenaf, pennywort, sugarcane, bamboo canes, pineapple leaf fiber, grass, reeds, ramie, papyrus, kapok, banana fiber, sisal and oil palm. Natural fibers, based on their sources, are classified into three categories: Plants based, mineral based and animals based. A composite containing mineral fiber is basically made up of asbestos fibers, which occur naturally but its use poses some health risks because it can lead to cancer in humans [2].

Fibers from animals are protein-based, for example, silk or wool, while fibers from plants are usually ligno-cellulose-based, which contain cellulose, hemicellulose and lignin. Due to their capability of being a substitute for synthetic fiber composites such as carbon or glass fiber composites, plant/animal fiber-reinforced composites have increasingly attracted much research attention. The benefits of the use of natural fiber composites are their renewability, ease of availability, low cost, high specific strength, low weight and rigidity. Replacement of synthetic polymers by the use of biodegradable polymers is anticipated in the near future, for applications where a short product life is more desirable. In particular, applications where long life is not necessary, natural fiber-reinforced composites are found to be ideal substitutes for synthetic-reinforced fiber composites and much research is still in progress. In such composite materials, a natural fiber (e.g. sisal, hemp, wood fiber and kenaf fiber) is blended with a polymeric material (e.g. PVC, polypropylene (PP) and polyethylene (PE)).

In recent years, natural fiber composites (NFCs) made up of both thermosets and thermoplastics have undergone incredible growth in the automotive industry because of renewability of natural fibers, environmental friendliness, good noise reduction capability and better fuel efficiency (due to weight reduction of components). Such composite materials have become very effective for commercial semistructural and structural applications. For instance, use of natural fiber-reinforced Polypropylene (PP) for interior parts, for example, door trim panels, and use of natural fiber-reinforced polyester resin in exterior parts such as transmission covers and engines have found great application in the automotive industry. The benefits of using NFC-based thermoplastics composites over NFC-based thermosets composites include improved design flexibility since they are appropriate not only for recycling but also for ease of extrusion and injection molding processing.

2 Classifications of plant/animal-plastic composites

There are several distinct groups of plant/animal fibers classified based on the source of the reinforcing fiber. The groups are displayed in Fig. 1. Plant fibers are essential natural fiber types, which are typically mainstreamed from the roots, stem, leaves or fruits of various plants, and basically contain lignin, hemicellulose, cellulose and pectin. Animal fibers mainly consist of protein, which include fur, hair, wool, silk and feathers. Mineral fibers are found in soil and rocks and contain minerals, essentially in silicates.

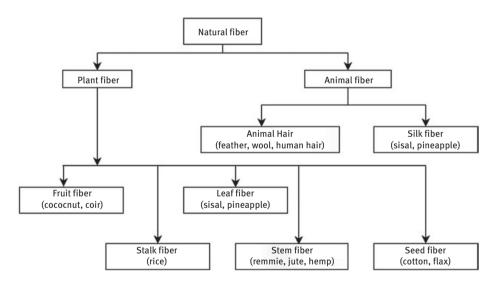


Fig. 1: Classifications of animal fibers and plant fibers.

2.1 Animal fibers-reinforced composites

Fibers of animals typically contain proteins. Examples of these include silk, fur, feathers and human hair. Wool contains many characteristics that distinguish it from fur or hair. It is usually squeezed, is elastic and is in staples. The source of animal fiber is mostly hairy mammals such as horse hair, alpaca hair, goat hair (mohair, cashmere), sheep wool, etc. Wool fiber commonly finds application in textile industries.

2.1.1 Silk fiber-reinforced composites

Silk is a fiber containing natural protein that can be crafted into textile products in several ways. The commonly known silk type is derived from the cocoons of the mulberry silkworm larvae. The glittering nature of silk material is caused by the structures which are triangular prism-like, which allows the silk material to refract.

2.1.2 Human hair fiber-reinforced composites

The human body contains follicles beneath the skin that produce human hair. Hair is a very thin stripe biomaterial which originates from follicles. Most of the human population is only concerned with the growth of hair, hair styles and treatment of hair, but hair is also an essential biomaterial primarily made up of protein, especially keratin with around 95% concentration. Hair is an interesting material since it is dynamic, non-homogenous and can be well integrated into a polymer.

2.1.3 Feathers fiber-reinforced composites

Feathers are one of the most intricate, integumental structures present in vertebrates that are composed of small follicles containing keratin proteins. Epidermis is an exterior layer. Feathers are a distinctive external cover on birds, representing one of the epidermal growths on animals. They are one of the most complicated integumentary structures present in vertebrates.

2.2 Plant fibers-reinforced composites

2.2.1 Stem fiber/Bast fiber-reinforced composites

Jute fiber: This plant fiber is usually collected from the cord of the stem. The wrapping material of objects such as bags, the carpet covering, the binding and yarns are some of the many uses of this fiber.

Stalk fiber: These fibers are obtained from plant stems. Wheat stalks, grass, rice, barley and bamboo are some of the examples.

2.2.2 Leaf fiber-reinforced composites

Sisal: Sisal plant is usually categorized as part of the Agavaceae family of the plants kingdom. The appearance of the plant is like that of big pineapples. The soft tissue of the sisal plant is manually extracted or machined-out of the fibers. It is used primarily for manufacturing carpets, mattresses and as a reinforcing material.

2.2.3 Seed fiber-reinforced composites

Flax: Flax fiber is among the strongest of the natural fibers. Flax has an excellent heat conduction property. In sharp folds, however, continuous creasing tends to tear the fiber at the very same place. Ropes, canvas, linen and sacks are some of the products produced from flax.

Ramie fiber-reinforced composites: Ramie is a costly and long-lasting fabric. In terms of fabrics, the ramie plant can be easily grown. This is commonly used in the creation of wallpaper, furniture covers and so on.

Hemp fiber-reinforced composites: The good strength and long length of hemp fibers have enabled the use of hemp fibers in the production of textiles, paper, sails and ropes. Hemp is an easy-to-grow plant which grows well in a slightly cool climatic condition. Hemicellulose, cellulose, pectin and lignin are the primary basic constituents of hemp fibers.

Cotton fiber-reinforced composites: Cotton is the leading material used as a fabric in the textile industry. It is obtained usually by picking, a process typically done manually. Cotton is primarily used in the production of carpets, blankets and clothes, unlike the other natural fibers.

2.2.4 Fruit fibers-reinforced composites

Coir fiber-reinforced composites: Coconut fibers are typically extracted from the coconut palm fruit husk. These cocoa fibers are hard, lightweight and resistant to heat and salty water.

3 Processing of animal/plant-plastic composites

Composites of natural fibers are produced in many ways. The majority of the techniques typically used for the production of glass fiber composites are appropriate for the manufacture of natural fiber composites. Nevertheless, the well-known composite production method is hand spray. It is one of the simplest and most common composite fiber production methods. The mold is gel-coated and waxed and then heated in a hot oven. During the processing, resin, which is catalyzed and contains the chopped fiber, is scattered into the mold cavity using a sprayer, where there is a build up of a secondary spray-up layer. The resin impregnates the middle of the laminates, resulting in the composite. Continuous fiber string materials and fiber layers are placed in the mold. Each layer is filled with a catalytic resin and the lightweight laminate is produced by applying the appropriate pressure.

Another widely used method is the Resin Transfer Molding (RTM). It offers a superior quality surface finish of the composite and it is capable of producing ideal forms of surfaces with relatively low energy. The equipment used usually coats the mold pieces and then continuously lays or cuts the strand materials and seals the compartment. Vacuum pressure and/or injection pressure moves the resin into the mold. The curing temperature depends on the resin injection process.

Compression molding is a form of molding process that is usually carried out in very small cycle times at a low cost. The sheet molding compound (SMC) is a method that is used to produce a surface which separates the resin paste between two plates. In the paste, the fiber is the main material and a separate film carrier is formed between the layers of resin. When it is packed, it shuts, tightens and pressurizes. Using the SMC technique, the pressure experienced when molding is between 500-1,200 psi. After curing, the mold compartment is opened and sheets are removed manually and ready to be used in an injection system. The injection molding process using the automated bulk molding compound (BMC) of thermosets has rapidly dominated the typical thermoplastic applications in the manufacture of electrical and automobile parts, domestic equipment and engine parts. A 15–20% chopped fiber-thermoset resin blend in BMC is a low-profile recipe (almost zero shrinkage). The injection molding technique is a simple, low pressure, voluminous and closed operation. The speeds of injection are usually 1 to 5 s, with a production capacity of approximately 2,000 small parts in an hour. A screw or a ram in the cavity moves the hot viscous material through the hot barrel of the machine to a heated mold. Heat and power reduction are managed by carefully preventing accumulation. After cure and injection, only a limited finishing is required for parts such as high volume automatic devices for manufacturing pressure vessels, pipes, tanks, tubes and shafts, and other cylindrical shapes using fine filament winding. Dry fibers are pulled out of the racks and wrapped around a mandrel through a resin trap. Pultrusion can be described as a continuous automated shut-off procedure that is economical in the production of continuous, broad volume crosssectional sections. Pultruded pattern profiles are used in common forms such as channels, lines, columns, pillars, bars and sheets.

As a mixture, wool fibers are mixed with polyester fibers and woven into several yarns as fabrics, usually for clothing. Fibers made up of wool, which are then knitted into fabric and used to make cloths, are typically woven into multilayered fibers as threads. The composite matrix is made of polyester resin comprising 1% methyl-ethyl ketone peroxide hardener. Sheets of the composite material are made from wool skeletons in a laboratory. Samples separately coated with mixture of the resin layers are then put in a mold of rectangular shape. The mold's top part is then screened and a hydraulic pressure of 1.2×10^6 N/m² is applied over exactly one day. The applied pressure is subsequently decreased to around 0.6 MPa. The sample plate is then removed and the remaining rigid resin is recomposed. The composite obtained is more likely to be soiled. For sample sheets of 40, 30, 20 wool, mass and a polyester resin device, this technique was repeated. Different types of silk fiber, feather fiber and human hair fiber composites can be produced by compression molding and/or hand laying method that have already been established in the production of plant fiber composites.

4 State of the art: ongoing research in animal/ plant-plastic composites

Over the last two decades, there has been increasing interest in the research of plant/animal fiber-reinforced composites. This has been motivated by the urge to (i) lower prices, (ii) lower weight and (iii) market use of green materials. There were less important technical requirements, so the use was confined to non-structural components for a number of years. This is because of the traditional demerits of plant/animal fiber-reinforced composites: poor impact strength and moisture degradation. Nevertheless, recent work has shown that substantial changes to these properties can be made, leading to more comprehensive applications.

Pratheep et al. [3], produced plant–plastic composites by mixing plastic with the coir pith powder, maize cob powder and wood for environmentally friendly applications. The composite consisted mostly of wastes derived from corn powder, coir pith and wood powder. Residues obtained from agricultural produce were used in their study. The composite reinforcement materials were the maize cob powder, hybridized coir pith and hybridized wood powder. The powder materials were bound to one structure using the resin from PVC and PPA hardener. The compression molding technique was used to produce the PVC matrix composite which needs to undergo a curing phase of at least 8 hours. The plastic and the powder were combined in varying concentrations and the produced composites had different properties at different powder concentrations. The level of powder loading was kept at 20%. Considering the mechanical tests conducted, addition of maize cob, as compared to the same amount of added wood powder into the polymer matrix, increased the flexural and tensile strength. Moreover, the produced wood-plastic composite [WPC] had a greater impact strength than other tested samples containing maize cob and coir pith powder content.

Ramesh, Sadashivappa and Sharanaprabhu [4] used several wood species at different proportions to produce chemically treated WPC using the hot compression molding process. After fully mixing phenolic formaldehyde and wastes from wood in a blender with rotators and PF-based WPCs have been tested in compliance with ASTM specifications for physical and mechanical properties. The experimental findings demonstrate the technological viability of WPCs based on phenol formaldehyde. Composites prepared chemically, however, have stronger characteristics than untreated wood plastics.

Ramesh et al. [4], fabricated a WPC by hot compression molding technique. The composite was chemically treated with distinct species of wood at various ratios [v/v]. The WPC sample having a thickness of 6 mm was made after mixing phenol formaldehyde and wood waste thoroughly into a rotational blender and then hot pressed. Wood–PF composites were then tested according to ASTM standards in consideration of their physical and mechanical properties, as per mixing proportions. The test results indicated that wood–PF composite is technically feasible. The composites chemical treatment, however, improved its properties as compared to the WPCs that were untreated. These composite samples exhibited a decrease in their water absorption rate, swelling of thickness and moisture content as the concentration of the PF matrix increased. The swelling in thickness and the rate of water absorption of WPC decreased as the temperature of immersion increased. It was also observed that the WPC's hardness and strength are improved when they are chemically treated. However, by incorporating additives such as coupling agents, the physical properties and mechanical properties of the WPC can be enhanced.

Vedrtnam, Kumar, and Chaturvedi [5], used wood dust from the specified Indian trees and incorporate it into polypropylene matrix to produce WPCs. The mechanical tests, morphological characterizations and wear of WPCs were done. It was observed that the WPC's impact strength, flexural rigidity, tensile strength, wear hardness, etc. depends on the type of wood dust. High tensile strengths were observed in when the composite was reinforced using wood dust of Babool followed by Sheesham with the least strength exhibited in mango wood dust. However, mango WPC had the highest impact strength with high wear resistance at lower wood concentrations. Additionally, high hardness value was recorded for babool WPC. It was observed that the increased concentration of the wood dust reduces wear resistance in samples of WPC. The biodegradation test revealed that water degradation is greater than earth degradation. The exposure to ultraviolent radiation adversely affects the matrix of WPCs, resulting in deteriorated mechanical properties.

Suffo, La Mata, and Molina [6], prepared agro-composites by mixing lowdensity polyethylene [LDPE], among the most commonly produced plastic, with an organic residue called "carbocal" found in the processing industry of sugar beet. The composite synthesis done was a straightforward and did not involve the use of binding agents, adhesive polymers or grafting. The tests conducted indicated that the composite had improved mechanical strength as compared to a pure polymer and demonstrated the role of the waste reinforcement in the polymer matrix.

Jiang et al. [7], prepared a biodegradable composite by using biodegradable poly (butylene succinate) (PBS) polymer matrix incorporated with bamboo charcoal (BC), ZnO and Si₃N₄. The findings indicated that the composite's mechanical properties were affected by different rates of filling. The better mechanical properties of the prepared composite material were achieved when 40% the bamboo powder was added. Generally, it was apparent from mechanical and chemical composition and also from thermal properties that the ideal conditions to produce decaying WPC were 150 °C for 10 minutes, whereas BC, Si₃N₄ and TiO₂ were 5%, 3% and 3% of the poly [butylene succinate]'s weight, whereas PBS and bamboo powder were 60% and 40%, respectively. The most remarkable thing was that the material comprises fewer organic volatiles, meaning that it was a good, green material and less toxic.

In the production of WPC, [8] assessed implementation of technological performance to wastes from industries. The main materials used were Cedrela odorata L. sawdust, high-density polyethylene (HDPE) and polyethylene terephthalate (PET) thermoplastics and calcium carbonate. Direct extrusion method was used while varying the concentrations of raw material and then conducting tests to evaluate their physical-mechanical properties. This study showed a linear relationship between the additive's concentration and the physical properties of the composite, with the exception of their density. In comparison, this influence of the additive concentrations resulted to an inversely proportional relationship to the board's mechanical properties. Statistical analyzes indicate how polymer type affect the boards' physical and mechanical properties by more than 70%, especially when used in more than of 50% concentrations. The concentrations of wood [sawdust] of more than 50% tend to reduce board resistance in terms of their physical properties, leading to a low mechanical strength.

Gibier et al. [9], developed WPCs by incorporating tropical hardwood and softwood using a radiation-curing resin that contained chlorine or phosphorus groups as fire retardant. The heterogeneous impregnation was determined by microscopic analysis and was shown to be influenced by the density, sample orientation and the wood species used. Unmodified wood and resin-impregnated woods were investigated and their physical properties [water content hardness, conductivity, stiffness,] correlated with their densities. Cone calorimeter was used to analyze the flame retardancy. While the addition of the resin was unfavorable for fire retardancy, it was possible to decrease flammability by adding chlorine or phosphorus groups. In contrast to chlorine-containing resin, phosphorus-containing resins restrict smoke emission. Finally, the wood plastics composites that were denser exhibited better mechanical properties with no flammability increase as compared to unmodified wood-plastics composites.

Wang et al. [10], produced biochar fibers by pyrolyzing corrugated cardboard at various temperatures (350 °C, 400 °C, 450 °C). In order to prepare WPCs, the biochar and corrugated cardboard control fibers were then mixed with highdensity polyethylene (HDPE) and malleated polyethylene (MAPE). The influence of various pyrolysis biochar temperatures on mechanical, rate of water absorption, viscoelastic and thermal properties, rheological behavior, performance of bio-durability and weatherability of WPC were assessed. The melting of the corrugated cardboard composite indicated higher modulus and viscosity as compared to biochar composite, thus implying greater melt strength. Biochar composites exhibited improved tensile strength (4%) and tensile modulus (30%) as compared to corrugated cardboard composites. The biochar composite had higher tan δ and adhesion factor as compared to corrugated cardboard composite, indicating a substantial interfacial interaction between HDPE and biochar fibers. There was no significant change in melting temperatures (T_m). For biochar composites, the HDPE degree of crystallinity decreased when compared to corrugated cardboard composites, whereas in comparison with corrugated cardboard composites, the biochar composite's thermal properties increased. High rate of water absorption (3.9%) was reported in the corrugated cardboard composite with swell thickness [3.8%] seen after 70 days.

WPC was prepared by reinforcing polypropylene (PP) [11] with animal fibers (hair). Compression molding technique was used to prepare the composite materials. The amount of horse hair was varied by setting the fiber [hair] length 0%, 10% to 20% and 30% respectively. Experimental findings indicated that the flexural strength, tensile strength and impact strength increased up to 20% horse hair content and further increase in horse hair content adversely affected the mechanical properties but the rate of water absorption continued to increase.

Similarly, [12] studied the mechanical properties of hair fiber reinforced composites. The reinforced composites demonstrated that the mechanical performance of composites is affected by the loadings and the ratios of the fibers incorporated into the HDPE polymer. A comparison was drawn on the influence of untreated and treated fiber on animal polymer composites. The tensile strength was observed to increase as the fiber concentrations increased up to an optimal concentration of 15% wt. ratio and started to decrease with further increase in fiber concentrations. High tensile strength at the optimal value was probably attributed to the fact that there was heavy bonding between polymer matrix and the hair fiber, hence there was a good load distribution between polymer matrix increased further, tensile properties diminished due to poor distribution of fiber in the polymer matrix, poor polymer-fiber interaction and increased voids in the composite material.

5 Applications of animal/plant-plastic composites

5.1 Sound insulation components

Wood–plastic composites have had some fantastic applications as sound insulation components [13] found that a significant technical index for assessing the building wall physical property is the sound insulation capacity. The findings indicated that when compared to that built by pinewood as wall studs, the wall which was designed by WPC as wall studs showed a difference of less than ± 3 dB in sound insulation. The external wall panel material was greatly influenced by the property of the wall's sound insulation. The insulation was significantly higher than the one of the plastic hanging board of polyvinyl chloride (PVC).

5.2 Automotive manufacturing

Composites reinforced with wood are very attractive and reliable eco-friendly materials for long-term use. They do not use any harmful substance. Plant fibers are typically appropriate for reinforcing polymers because of their high strength and rigidity, renewability, low CO_2 emission, low cost, biodegradability and low density when compared to other fibrous materials. The rapidly growing example of polymer additives are plant fibers used as fillers and polymers reinforcements. Even though automotive manufacturers wish to recycle or biodegrade all components, green composites based on biodegradability of polymers and plant fibers still seem to be a potential way to go. In technical terms, such bio-based composites improve acoustic and mechanical properties, increase fuel efficiency, reduce weight, increase health of passenger, reduce costs of production, are shatterproof under sudden change in temperature and enhance the biodegradability of interior parts of automobiles [14]. Examples of vehicle interior parts made up of natural fiber-reinforced composite include front and rear door linens, parcel shelves, boot linens, door-trim panels, seat backs and truck linens [15].

5.3 Civil engineering applications

In addition to the automotive industry, animal/plant–plastic composites have found great application in the building and construction industries. Animal/plant–plastic composites are mostly used in producing non-load carrying interior parts in civil engineering due to their susceptibility to environmental attacks [16]. Green buildings are designed to be environmentally friendly and effective and convenient for living and working. Bio-composites are known to be amongst the most significant materials commonly used in green materials. In construction, bio-composites can be classified

as structural bio-composites and non-structural bio-composites. The examples of structural bio-composites are the roofs and bridges, while examples of non-structural bio-composites are door frames, composites panels, exterior construction and windows [17].

5.4 Fluid container

Filament wounds from spinning natural fibers can be used to produce low cost filament winding equipment and reusable containers to store and convey liquids (soysauce, beer, water, fish and wine). The vessel is "foldable once it is empty" when natural rubber is used as resin. Natural fibers have thermal properties which keep the stored or transported products cool. Their lightweight nature, eco friendliness and reduction in shipping and production costs are the benefits over steel or plastic vessels.

5.5 Small boats

Small boats can be made with vacuum techniques. Such boats are more resistant to corrosion as compared to metallic boats and are easy to maneuver both on and offshore because of their low weight. They are also longer lasting than boats made up of bamboo, wood or steel.

6 Conclusion

Composites made up of plant/animal fibers have attracted much research interest due to their cost-effectiveness, low density, recyclability, and environmental friendliness. Plant/animal fibers-reinforced composites are among the most promising materials that can be used by technicians, engineers, industrialists and producers because of their potentially great application in various technical fields such as in packaging, building and construction, railways, automobiles, safety engineering and so on.

Plant/animal fibers can boost the mechanical and physical properties of polymers and cement matrices, when used as reinforcing agents. Plant fiber is a widely used class of natural fiber in manufacturing as compared to animal and asbestos fibers. This is because of its environmental friendliness, renewability, consistency and recyclability. While asbestos natural fibers have desirable mechanical, thermal and sound insulation properties that can enhance their use in engineering applications, due to their carcinogenic nature, a large number of developed and developing countries have banned their use because it causes diseases. There is a need to create alternatives to conventional asbestos-reinforced composite material. The use of animal fibers as a reinforcing agent in composite preparation is an emerging field with much research attention. Animal fibers can be obtained as waste from animals and can be used to produce composites that can be used in certain technical applications. Composites in this area are restricted by fiber deficiency, but feather science is still evolving. Actually, the composite products that can be produced can result in turning waste into revenue. A wide variety of plant fibers are available in nature. They can be enhanced genetically and modified in different ways resulting in a wide variation in physical, mechanical and chemical properties of the produced composite. Specific plant fibers polymer-reinforced composites have been developed and used in different applications using various polymeric matrices. Composite reinforced with natural fiber is valuable because it is bio-derived, biodegradable and sustainable. It is, therefore, necessary to produce bio-derived/biodegradable composite materials to address the potential challenges of safeguarding non-renewables and to comply with the Kyoto Protocol for sustainable environmental and protection of life.

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S. Sathees Kumar and B. Sridhar Babu A review of ductile attributes of natural fiber composites

Abstract: Normal fiber-strengthened composites are a developing territory in the polymer discipline. These natural fiber composites are biodegradable and noncoarse. They exhibit explicit attributes that are practically identical to those of regular fiber composites. These include their low rate, equally great mechanical attributes, extreme explicit quality, non-rasping, eco-friendly and ecological attributes. The ductile characters of natural fiber-reinforced polymers are essentially impacted by the hook-up attachment between the matrix and the fibers. This report presents the detailed effort on characteristics of natural fiber composites with exceptional testimonials to the types of fibers, fabrication method and ductile attributes.

Keywords: natural fibers, fabrication, ductile attributes, types of fibers, mechanical properties, sisal, cotton, volume fraction

1 Introduction

In recent years, we have seen that polymers have supplanted huge numbers of traditional materials for different purposes. This is conceivable in light of the points of interest polymers offer across regularly used composites. The most important points of interest in using composites are their simplicity in handling and profitability. In a large number of these functions, the attributes of polymers are changed by using fillers and filaments to suit the extreme quality requirements. Fiber-strengthened polymers offer points of interest that are superior to those of customary materials when explicit attributes are considered. These composites are discovering uses in different domains from machines to rockets.

Characteristic fibers created a lot of interest among researchers and technologists on account of the favorable circumstances that these filaments offer over ordinary fortification materials [1–4]. These characteristic filaments are minimal effort strands with explicit attributes. Recyclability or ecological applications of common fiber composite items after a helpful life makes them more significant and encourage car makers to use characteristic strands. On the off chance that biodegradable strands were picked to substitute a significant number of composite applications, one may lessen the extraordinary troubles of discarding these items [5].

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Lucintel's [6] work explained that the commercial market for regular fiber composites would grow to 540 million dollars. Requests from car organizations for materials with clamor decrease capacities have increased similar to the way lessening weight has increased eco-friendliness [7]. This is because regular fiber composites have brilliant sound-engrossing abilities, do not break easily and have more proficient vitality compared to glass materials; hence, interest for normal fiber composites has expanded in the commercial market [8]

Use of common strands in composites made of plastic materials has grown by 20% in vehicles and by half or more in select civil engineering areas [9]. Natural fiber-based car parts, for example, different boards and shoes of brakes have found appeal in car businesses since they have reduced the heaviness of elements by over 10% and have additionally reduced the expense by as much as 5% [10, 11].

In any case, certain disadvantages, for example, mismatch with the polymer substance, inclination to shape totals through preparing and poor opposition to wetness significantly decrease the performance of characteristic filaments used in polymers. In this report, we shall study different parts of characteristic fiberstrengthened composites and refer a portion of the fundamental matters of such composites that are being developed.

2 Types of natural fibers

Based on the sources, the natural fibers are majorly classified into three types: seed hair, leaf fibers and bast fibers. Figure 1 depicts the classification of fibers.

2.1 Leaf fibers

- i) **Sisal**: Sisal plant comes under the agave family (Agavaceae). It looks like a giant pineapple. It is used in making mats, floor coverings and numerous other fortification materials.
- ii) **Cotton**: Cotton is the most significant fiber used in the material business. Cotton is commonly picked by hand. Like other normal filaments, cotton is, for the most part, used in the manufacturing of garments, covers and carpets.
- iii) Flax: Flax fiber is one of the most stable among common fibers. Flax has great warmth directing attributes. Be that as it may, consistent wrinkling in a similar spot in sharp overlap will, in general, break the fiber.

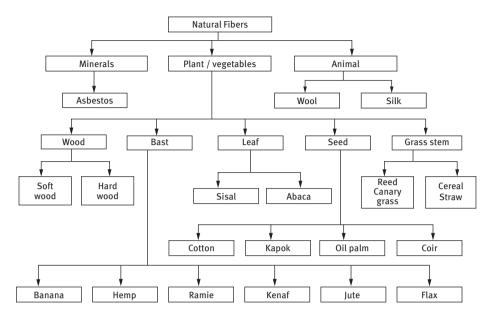


Fig. 1: Classification of natural fibers.

2.2 Fruit fibers

i) **Coir:** Generally coconut fibers are obtained from the husk of the coconut palm. These coconut strands are solid and light and can effectively withstand the salt water.

2.3 Bast fiber/stem fiber

- i) **Jute fiber:** Generally, jute filaments are extricated from the strip of the stem. It is used as bundling material (sacks), carpet backing, ropes and yarns and in numerous other ways. Table 1 shows the classification and uses of plant fibers, and Table 2 illustrates the composition of chemicals for various fibers.
- ii) **Stalk fiber:** These strands are really separated from the stalks of the plant for instance, rice, grain, straws of wheat, bamboo and grass.

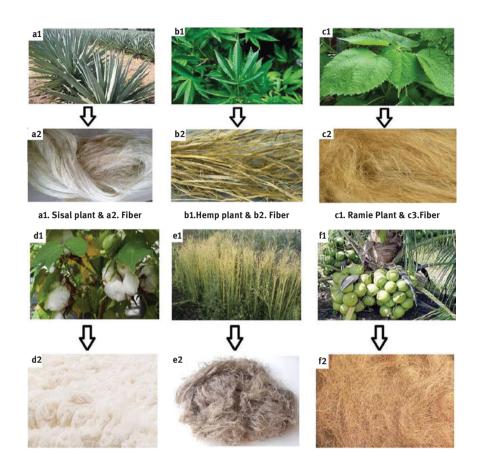
Fig. 2 depicts the plants (a1, b1, c1, d1, e1, f1 and g1) and fibers (a2, b2, c1, d2, e2, f2 and g2).

Common name	Fiber type	Scientific name	Family	Uses	
Flax	Bast (stem)	Linum usitatissimum	Linaceae	Linen fabrics, seed oil	
Ramie	Bast (stem)	Boehmeria nivea	Urticaceae	Textiles, paper, cordage	
Hemp	Bast (stem)	Cannabis sativa Cannabaceae		Cordage, nets, paper	
Kenaf	Bast (stem)	Hibiscus cannabinus	Malvaceae	Paper, cordage, bagging	
Urena	Bast (stem)	Urena lobata	Malvaceae	Paper, bagging, cordage	
Abacá	Hard (leaf)	Musa textilis	Musaceae	Marine cordage, paper, mats	
Coir	Fruit fiber	Cocos nucifera	Aracaceae	Rugs, mats, brushes	
Ramie	Bast (stem)	Boehmeria nivea	Urticaceae	Textiles	
Jute	Bast (stem)	Corchorus	Tiliaceae	Cordage, burlap bagging	
Kapok	Fruit trichome	Ceiba pentandra	Bombacaceae	Upholstery padding,	
Sisal	Hard (leaf)	Agave sisalana	Agavaceae	Bagging, coarse fabrics	
Sun hemp	Bast (stem)	Crotalaria juncea	Fabaceae	Fire hoses, sandals	

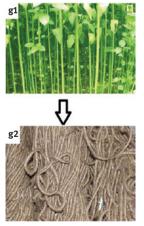
 Table 1: Classification and uses of plant fibers.

Table 2: Composition of chemical for various fibers.

Fiber type	Hemicellulose (wt%)	Cellulose (wt%)	Lignin (wt%)	Pectin (wt%)	Moisture content (wt%)	References
Abaca	-	56-63	12-13	1	5-10	[12]
Banana	10	63-64	5	-	10-12	[12, 13]
Cotton	5.7	85-90	-	0-1	7.85-8.5	[13]
Coir	0.15-0.25	32-43	40-45	3-4	8	[13]
Flax	18.6-20.6	71	2.2	2.3	8-12	[12, 14]
Hemp	17.9-22.4	70-74	3.7-5.7	0.9	6.2–12	[13, 15]
Jute	13.6-20.4	61.1-71.5	12-13	0.2	12.5–13.7	[13, 15]
Kenaf	21.5	45-57	8-13	3-5	-	[15]
PALF	-	70-82	5-12.7	-	11.8	[16]
Ramie	13.1-16.7	68.6-76.2	0.6-0.7	1.9	7.5–17	[15]
Sisal	10-14	66-78	10-14	10	10-22	[15]



d1.Cotton plant & d2.Fiber



g1.jute plant & g2.Fiber

Fig. 2: Plants and fibers.

e1.Flax plant & e2.fiber



3 Methods of fabrication of natural fiber composites

Manufactured composites are finished by specific techniques, and these are explained below. These cycles are redone for the reasonable materials that are presence dealt with. Determination of the suitable resin and polymer proportions assume significant importance in it. The accompanying strategies are broadly used by various specialists.

3.1 Hand lay-up

This fabrication process is the least difficult process handling natural fiber. In this process, first, an agent is applied on the outside to avoid adhering of polymer to the exterior. To achieve greater and nice outer surface finish completion of the item, thin plastic layers are utilized at the topmost end and base of the shape. Filaments in slashed structure are set at the external of shape as a fortification. At that point, a blend of thermosetting pitch and an appropriate hardener are decanted on the outside of the tangle previously positioned in the shape.

Polymers are consistently distributed with the assistance of contact. Another layer of stronghold is then situated on the polymer outside, and a roller is used to empty air similarly as wealth from the current structure. This is done frequently for each layer of support and matrix untill the necessary stiffness is accomplished. In this method, epoxy resin is used by various analysts [17–21], and polyester pitch is used by researchers [22–25] for the creation of hybrid composites.

3.2 Compression molding

Compression molding is a fastened molding progression with extreme weight usage. In this technique, both coordinated metal molds are used to create a composite item. The bottom plate is fixed, while the top plate is mobile in pressure decay. Fortification and lattice are done in the metallic shape, and the entire gathering is maintained in the middle of the pressure decay [26]. The necessary measure of warmth and weight depends on geometry and composite size. Support and grid are used between the decay overlays, owing to the use of weight and warmth. In the wake of composite restoration, the shape is unbolted and the composite item is brought out for additional preparation. This technique is reasonable for thermo sets similar to thermoplasts-built strands (manufactured usually) composites. Strands-strengthened thermosets composite was set up by various scientists [27–32] and thermoplasts-built fiber composite was set up by Narendar et al. [33].

3.3 Injection molding

This methodology is mostly used in the advancement of plastic parts with extraordinary accuracy in dimensions. The things, for instance, house items, toys, car parts, furniture, bundling things, apparatuses and clinical evacuation needles are conveyed by the infusion forming process. Infusion shaping is the technique of encircling an item by convincing fluid plastics under tension into a structure where it is cooled and solidified along these lines, conveyed by invoking the two parts of the form [34]. This system is sensible for thermosets similar to thermoplasts-based fibers (manufactured normally). The thermoplasts-based fiber composite is fabricated by [34–36] using this technique.

3.4 Pultrusion

The pultrusion process is a predictable technique for amassing of things that have a consistent cross fragment, for instance, bar stock, basic shapes, projecting posts, channels, bars and pipes. In this system, constant wanderings of fortification are impregnated with tar, bypassing them through a tar shower, and a little later, traverse a steel bite the dust. The steel bite the dust fortifies the soaked support, puts the condition of the material and controls the fiber extent. The fiber is warmed to practically fix the gum [36]. This technique was used by [37] to make half-and-half composites.

4 Mechanical attributes

Elasticity and modulus of rigidity of fibers increase by expanding cellulose [37]. The small scale fibril point determines the fiber's stiffness. These fibers are more tensile if the smaller scale fibril has a winding direction to the fiber pivot. On the off chance that the smaller scale fibrils are printed corresponding to the fiber pivot, the strands will be unbending, solid, and will have peak elasticity. The attributes of natural fiber composites depend upon various factors, like fibers volume proportion, viewpoint proportion, fiber framework bond, stress move at the interlink and, furthermore, direction. The regular fiber composites comprise experimental investigation of mechanical attributes with respect to the fiber content and the utilization of outside connection agents [38–42]. Further angles incorporate the expectation of modulus for both stage frameworks and correlation with test data [43]. Lattice and fiber attributes are significant in enhancing the mechanical characteristics of the natural fiber composites.

Unbending nature is more responsive to the framework attributes; however, the modulus is dependent on the properties of the fiber. To increase the solid interface and low pressure focus, fiber direction is required; however fiber obsession and high fiber point of view extent select elastic modulus.

4.1 Tensile attributes

Ductile attributes of common fiber composites are, for the most part, influenced by the interlink bond between the resin and fibers [44]. Substantial and substance alteration of the fiber and tar increase the ductile attributes of composites. The ductile attributes of common fiber-based materials are profoundly reliant on the strands volume division in the grid sap. Although various researchers have indicated unpredictable patterns for pliable attributes of natural composites with respect to the volume fraction, generally, it is evident that by an expansion in the fiber volume part under an ideal worth, the load is conveyed to more filaments, and the grid can convey the applied burden after strands crack. It can refer to the extreme elasticity for the composite. The opposite and unpredictable patterns for the pliable attributes can be a direct result of numerous components, including contrariness amongst the fibers and medium, fiber deprivation, and improper manufacturing processes [45].

With further increments in the fiber volume part after the ideal sum, weak break happens in the filaments, and the framework cannot bolster the extra burden from the fibers. Under these conditions, the low elasticity in the long run prompts disappointment of the entire composite [46]. Sathees Kumar [47] developed hybrid composites by using three dissimilar natural fibers (sisal, jute and sorghum bicolor)-reinforced polyester composites. In this experiment, the specimens were fabricated through the hand lay-up method. The configuration of hybrid composites is illustrated in Table 3. Figure 3 shows the ductile test specimen.

Designation of	Natural fiber composition (wt%)				
sample	Jute fiber	Sisal fiber	Sorghum fiber		
A	45	50	4.5		
В	50	40	10.5		
С	35	50	15		
D	40	40	19.5		
E	25	50	25		
F	30	40	29.5		

Table 3: Composition of hybrid composites [47].



Fig. 3: Tensile test specimen [47].

From Fig. 4, it can be seen that sample D achieved the maximum ductile strength. As sisal and jute fibers are equally shared, the maximum weight % (40 and 40) and sorghum fiber composition are very less (20%). This result exhibits the individual characteristics of fibers. The maximum contents of individual fiber have a few unique characters. Figure 5 demonstrates the prolongation of crossover composites and the stretching character of composites expanded ahead to the degree of 14.4 mm (D).

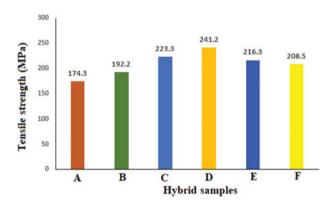


Fig. 4: Tensile strength of composites [47].

The tensile feature of composites expanded slowly from A, B, C and D samples. Unexpectedly it has diminished in E and F samples. Expanding the sorghum fiber substance might decrease the yielding property and expand the weakness of the samples.

Generally, higher fiber content is required to achieve short fiber-fortified polymer composites (SFRP). It is frequently seen that the fiber content in the polymeric substance promotes the composite quality and ductile modulus [48]. The impact of fiber content on the ductile attributes of natural fiber strengthened composites is of exact attention and importance for some investigators [49]. Sathees Kumar [50] studied the effect of mechanical attributes on three dissimilar natural fibers. In this

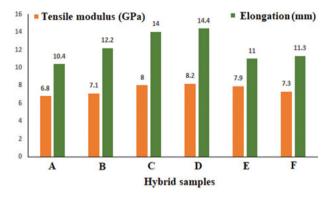


Fig. 5: Tensile modulus and elongation of composites [47].

study, different combinations of compositions were used to prepare the samples shown in Table 4.

Samples names	Natural fiber composition (wt%)					
	Sorghum fiber	Sisal fiber	Coir fiber			
A	5	90	5			
В	20	80	10			
С	25	70	15			
D	30	60	20			
E	35	50	25			

Table 4: Composition of natural fiber composites [50].

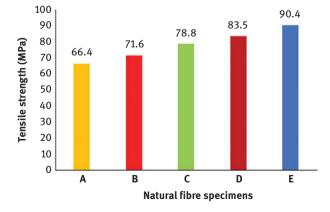


Fig. 6: Tensile strength of natural fiber composites [50].

In this study, sisal is the base fiber and the remaining two fibers are fillers. Half of the weight %(50) of sisal and the remaining two halves of each fiber's %(25 and 25) attained the higher tensile values (Fig. 6). Due to proper dispersion of base fiber and equal sharing of high fillers, better ductile property is attained [51–55].

5 Conclusions

- 1. In the most recent decade, the utilization of regular strands has been essentially expanded for industrial and automotive applications.
- 2. Natural fiber composites, of late, have had an incredible recharged enthusiasm for an assortment of reasons: for example, diminished cost, simplicity of creation, lower thickness and weight, and an expanded mindfulness regarding the matter of reusing, and the effect of materials on the earth have likewise assumed a significant role in the increased reception for common fiber composites.
- 3. Natural fiber-based composites are mostly utilized in a grouping of inside and outside bits of vehicles, bundling-in ventures and development fields.
- 4. Current research on fiber composites will add to a more prominent and intriguing take-up of this normal fiber-based composite grid by an industry that everexpanding number of things into the business, later on.

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Part II: Plant-based composite

S. S. Godara, Vinay Swami and R. S. Rana Study of plant-based fibers and resins in composites

Abstract: The use of sustainable raw materials in the composite industry is turning out to be progressively well known in view of their natural aftercare and the necessity to replace fossil resources. Plant oils with triglyceride spines can be artificially altered and used to integrate resin from inexhaustible assets. Plant oils dominatingly comprise of triglyceride, the glycerol esters of unsaturated fats. Five important types of unsaturated fats of the unsaturated fat chain are obtained as one of the outputs from the hydrolysis process of triglyceride, consisting of 16 to 18 carbons with 0 to 3 times the securities. These five unsaturated fats are; oleic, linolenic, palmitic, linoleic and stearic acids. Specific unsaturated fat varies across different plant oils. Even within a similar plant oil, the unsaturated fat depends on the plant species, weather and plant growth states. This paper emphasizes on plant-based fibers, resins and fillers derived from different plants.

Keywords: plant oil, epoxidized plant oil, fiber-reinforced composites, resins, natural fibers

1 Introduction

Plant oils are a common asset in the manufacture of bio-based polymers due to their properties such as low cost, tremendous amount and potential biodegradability [1–3]. The overall production of plant oil was over 150 million tonnes in 2012. The industrial application of plant oils is generally based on the synthetic alteration of carboxyl along with unsaturated carbon bunches present in unsaturated fats [4, 5]. Iodine value (I.V.) is one of the most significant properties influencing the physiochemical properties of unsaturated fat. The level of unsaturation is also estimated by the iodine esteem (I.V.). Plant oils can be categorized into three types based on Iodine value: drying oil, semi-drying oils and non-drying oils. Oils with Iodine value more than 130 fall under the category of Drying Oil, an example of which is Linseed Oil., Plant oils with Iodine value between 100 and 130 fall under

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the category of Semi Drying Oil, an example of which is Soybean Oil. Plant oils with Iodine value less than 100 fall under the category of Non Drying Oil, an example of which is Palm Oil [6]. Plant oils with higher iodine value are attractive for composite application, which implies increasingly useful cohesion that encourage the more exceptionally cross-linked structures for better, warm and mechanical qualities. These plant oil-based normal filament composites can be additionally used in nanocomposites, which have broad potential applications [7].

Regular plant oils, for example, Linseed Oil, Peanut Oil, Palm Oil, Soybean Oil, Sunflower Oil and Olive Oil are considered due to their unsaturated fats arrangement, double bonds and iodine value. Palm oil has the largest oil creation volume capability. Soybean Oil, Rapeseed Oil and Sunflower Oil follow Palm oil. Soybean is largest grown plant in Brazil and is second in the U.S. Plant oils are essentially used in food applications. Around 20% is used as plastic feedstock, surfactants, biofuels, coatings, paints, ointments and so on. The inner twofold obligations of plant oils are wealthy in electrons and accordingly are powerless to cationic polymerization. Palmitic and stearic have a place with soaked unsaturated fats. Oleic, linoleic and linoleic acids have a place with unsaturated fats. The properties and fatty acid compositions are displayed in Table 1.

Plant oil	Saturated		Unsaturated			Double bonds	Iodine value
	Palmitic	Stearic	Oleic	Linoleic	Linoleic		
Linseed	5	4	22	17	52	6.6	168-204
Olive	14	3	71	10	1	2.8	75-94
Palm	44	4	39	10	-	1.8	44-58
Peanut	11	2	48	32	-	3.4	80-106
Soybean	11	4	23	53	8	4.6	117–143
Sunflower	6	4	42	47	1	4.7	110-143

Table 1: Typical properties and fatty acid compositions of common plant oils [8-12].

In Table 1, different plant oils are categorized based on different parameters like fatty acids composition, double bonds and Iodine value. Composite stuffs are produced using these resins to manufacture a variety of enduring and firm material. Fats are categorized into two types – saturated and unsaturated. Palmitic and stearic acids fall in category of saturated fatty acids. Acids such as linoleic, Oleic and linoleic acids fall under category of unsaturated fatty acids. These plant oils contain triacylglycerol containing a range of fatty acids. Fatty acids and triacylglycerol composition define the physical, chemical and nutritional characteristics of these plant oils, whereas Iodine value is a degree of average unsaturation.

2 Composites from epoxidized plant oils

Epoxidized soybean oil and epoxidized linseed oil are now available as the main bio-inexhaustible epoxies. By the epoxidation of the double bonds of unsaturated fats using peracids, epoxidized plant oil can be manufactured. A lot of interest being centered on the advancement of plant oil-based composites because of their maintainable qualities, incredibly improved firmness, modulus and quality. A composite methodology incredibly extends the expected use of plant oil-based polymeric materials. There are a number of promising options in contrast to plant oilbased materials for transportation and development purposes. Ongoing advancement is more towards elite green composites and bio-based materials for special purpose and basic applications. Based on the type of reinforcement, plant oil form composites. At the macro level, fiber, whether it is natural or synthetic, can be chopped or continuous. The molecule size and surface area of the nanocomposites supports volume at the nanoscopic level.

Petroleum-based epoxy copolymerized with epoxidized plant oil enhance the characteristics of the resulting resin. Plant oil is affluent and a flexible material for bio-based polymers. Plant oil-based composites are appealing options in contrast to petrochemical-based polymers [13].

Thermosets and composites can be mixed with a mixture of epoxidized squander plant oils and diglycidyl ether of biphenyl-A. Sanitization can expel contaminations that result from warm corruption in the searing procedure [14].

2.1 Fiber-reinforced composites

The mechanical quality of fiber-reinforced composites is fundamentally reliant on the characteristics of the persistent phase reinforcement. The matrix phase supports and ties the reinforcement altogether and divides stress to reinforcement. While fabricating a composite for an auxiliary application, the polymer matrix must sufficiently be able to proficiently move stress over the reinforcement without causing breaks that are of adequately high crosslink thickness. Most unadulterated epoxidized plant oil polymers show lower crosslink thickness.

Studies have shown that some epoxidized plant oils were optimum-restricted in mixes, which is less than 30 wt%, in the light of the fact that the exclusively epoxidized plant oil segment could not give the mechanical and warm properties needed for fiber-reinforced composites. Unadulterated epoxidized plant oil or high epoxidized plant oil content (e.g., more than 50 wt%) forming polymer networks for superior composites purposes are uncommon and progressively appropriate for non-auxiliary purposes [15–18]. Glass filaments are the most widely used reinforced materials in epoxy composites on account of their accessibility, minimal effort and great attachment to matrix resin. Using the technique of hand lay-up, Espinoza-Perez et al. [19] fabricated fiber glass composite. Bis (4-aminocyclohexyl) methane restored epoxidized plant oilbusiness epoxy mix was used as the framework. The 30 wt% epoxidized plant oil mixed composite warm and mechanical execution was marginally lower than the composites without epoxidized plant oil, yet were equivalent to those of the anhydride restored ones.

Epoxidized methyl ester, epoxidized allyl soyate and epoxidized soybean oil of soybean oil were used in bio-composite assembling using pultrusion preparation, however, these epoxies were constrained to being a smaller part in mixes, for example, less than or equal to 30 wt% of the epoxy mix. More prominent mechanical properties were exhibited by Epoxidized allyl soyate composites than those of Epoxidized soybean oil or Epoxidized methyl ester of soybean oil because of a developed oxirane substance and a preferable reactivity. A further increase in Epoxidized allyl soyate substitution content, up to 50 wt%, was additionally endeavored. The pulling power of pultrusion fabricating decreased due to good lubrication provided by the slick biobased segment triggered by the immersed and inert part of Epoxidized allyl soyate.

Cellulosic filaments, for example, flax, hemp, and jute are additionally encouraging fortifications for polymer composites because of their accessibility, high explicit quality, minimal effort and their natural disposition. Polymer composite reinforced by plant oil cellulose is routinely referred to as "green" composite due to the fact that matrix resin and reinforcement are from bio-sustainable assets. In any case, these composites have lower mechanical quality than comparative composites fortified with glass strands.

Hemp fiber invigorated epoxidized linseed oil composites were produced by Boquillon [20] using a hot squeezing strategy. Results demonstrated that the limit modulus expanded. It was 17 MPa for smooth sap and 850 MPa for 65 volume percentage of fiber-based composite at a rubbery district.

The soaking of anhydride hardener on the surface of hemp fiber results in an off-stoichiometric reaction between the anhydride and the epoxy. Glass transition temperature (T_g) degradation was also found in flax fiber stronghold of epoxidized linseed oil composites by Fejos et al. [21], which was seen in an engineered reaction among the anhydride hardener and the hydroxyl group.

Liu et al. [22] produced flax-reinforced fiber using the weight framing strategy. Epoxidized soybean oil and 1, 1, 1-tris (p-hydroxyphenyl) ethane triglycidyl ether mixture, abbreviated as THPE-GE, were used in this operation. Extended fiber associations and dissipating issues result in increased flexibility and tractable modulus with higher fiber loading. The correct fiber content was around 10 weight percent. An elevated level of THPE-GE was required to obtain high thermal and mechanical quality composites in the mixture. Short fibers have mechanical properties than long fibers .

Jute fiber composites were produced by Manthey et al. [23] in which amine diminished epoxidized plant oil and diglycidyl ether of bisphenol A (DGEBA) mixtures played the cross section role. Epoxidized hemp oil composite demonstrated elevated mechanical quality than epoxidized soybean oil composites. Both composites showed lesser mechanical properties with extended epoxidized soybean oil stacking. Greater decline in quality occurred at more than 30 wt percent bio sap obsession.

2.2 Nanocomposites

Polymer nanocomposites have attracted excitement over the latest couple of years in the light of their capability to show exalted thermal quality, light weight, mechanical quality, and optical straightforwardness at decently low atom centers, for example at less than or equal to 5 weight percent. Nano clay, silica, silsesquioxane, carbon nano-tubes and alumina are all examples of nanocomposites. These composited have been used with epoxidized plant oil to form polymer nanocomposites. Among all organo improved montmorillonite, soil pamphlets are significantly beneficial stronghold fillers for polymer nanocomposites.

Nano clay platelets with large plane area, elevated perspective extent and strong interfacial coordinated efforts among polymer and nano clay show an activity in limitation of polymer chain adaptability underneath tension. Polymer properties can be liberally developed. Thus, the critical test experienced throughout the availability of polymer earth nanocomposites is true dissipation of mud inside a polymer lattice on a nano metric degree, to obtain structures that are stripped, interpolated or mixes.

It was declared by Wang and Schuman [24] that nanocomposite etymologies show thermal and mechanical qualities due to their earth obsession and dispersing methodology. Fast shear blending along with ultra-sonication diminished the platelet tactoids to a lesser degree. This resulted in better properties when compared to a shear mixing procedure alone. Compared to clean polymers, just 1 weight percent of mud sparsed by ultra-sonication elevated the nanocomposite unbending nature and modulus between 13% and 22%, independently. Flexible modulus could be extended up to 34% by 6 wt% earth with no reinforcement retribution. Moreover, the glass progress temperature (T_g) increased by 4 to 6 °C, depending upon the OMMT center.

Anhydride cure epoxidized soybean oil nanocomposites were studied by Tan et al. [25]. Using the technique of ultra-sonication, organo-modified nano clay, abbreviated as OMMT, ranging from 1 to 5 wt% center was dissipated into epoxidized soybean oil. The surface updater of organo-modified nano clay and imidazole co-catalyze the epoxy-anhydride reestablishing reaction. With a shed structure, the flexibility of the organo-modified nano clay nanocomposite was extended to 4 wt%.

malleable modulus. Glass change temperature (T_{o}) and warm quality of the epoxidized soybean oil were similarly extended in the wake of including OMMT. The split strength and break extension were reduced as a result of improved robustness and crosslink thickness. In a near anhydride reestablished epoxidized soybean oil-earth nanocomposite system, Miyagawa et al. [26, 27] declared nanocomposites of anhydride reestablished the blend of diglycidyl ether of bisphenol F, abbreviated as DGEBF, and epoxidized linseed oil. Earth nano platelets were perfectly stripped and homogeneously dissipated in the epoxy sort out after ultra-sonication dispersing. The resulting nanocomposites demonstrated elevated limit modulus than smooth polymers to adjust a diminished storing modulus achieved by the substitution of DGEBF by epoxidized linseed oil. The Izod influence quality didn't change with the inclusion of soil, while the glow distortional temperature and glass transition temperature (T_g) were less because of the plasticizing impact of organo-modified nanoclay updater. The nanocomposite was used as a network for carbon fiber braced polymer composite. The outcomes showed that bury laminar shear nature of the composite was enhanced in the wake of mixing 5 wt % intercalated soil. All the same, the stripped earth nano platelets were less effective in hindering the split spread.

Logically enormous improvements in quality have been seen in epoxidized plant oil-based nanocomposites with low glass change temperature (T_g). Nanocomposite inflexibility and modulus were extended by over 300 percent for 8% by weight. Organo-modified nano clay has been studied by Liu et al. [28] when the triethylene tetramine, abbreviated as TETA, was used as a diminishing administrator for epoxidized soybean oil. Organo-modified nano clay was dissipated in epoxidized soybean oil using the ultrasonication technique to shape an intercalated fabric. The glass progress temperature (T_g) of the ideal polymer was extended from 11.8 °C to 20.7 °C with 5% by weight mud. Elevated organo-modified nano clay centers show a decline in characteristics because of soil concentricity.

Shabeer et al. [29] coordinated nanocomposites using epoxidized allyl soyate and anhydride. Two types of dissipating procedures, ultra sonication and pneumatic, were carried out to strew the organo-modified nano clay into epoxidized allyl soyate. The nano clay was shed inside pitch as a result of the mud participation and reaction with anhydride. Tractable checking showed results that the organo-modified nano clay enhanced the moldable modulus and quality by 625% and 340%, respectively. These basic changes in quality were expostulated by a firm coordinated effort of epoxy along with soil platelets and the enhanced modulus of earth platelets compared well with that of the versatile polymer chains. In any case, the glass transition temperature (T_g) of the polymer below the normal temperature further reduced with extended earth loadings and is generally suitable for nonfundamental usage.

The antibacterial properties of vegetable oils can be generally improved by methods that combine very small amounts of metal oxide nanoparticles, for instance TiO_2 , Fe3O4, CuO or ZnO. The anti-microbial capacity of nanoparticles

compares with their associate characteristics such as size, shape and basically on the core interest. The combining of metal oxide nano particles with vegetable oilbased composites is a common way to misuse their outstanding antimicrobial properties [30].

3 Plant oil-based resins and fibers

Most recently, over the last couple of years there has been increased enthusiasm for using plant oil-based fiber, resin and filler in composites for development materials with minimal effort. The use of plant oil-based fiber, resin and filler have been more conventional than the use of traditional materials. The interest highlights the advantage of plant oil-based fiber, resin and filler due to the minimal effort required, low weight, high explicit modulus, sustainability, and biodegradability. Linseed, olive, palm, nut, soybean and sunflower are easily accessible in large amounts. All of these can be acquired legally from the normal asset. They are simple and furthermore and have the upsides of inexhaustibility, minimal effort, and simple accessibility.

3.1 Linseed

More than 78% by weight of green composites material are found from the material Linum usitatissimum. A blend of methyl nadic anhydride (MNA) and maleinized linseed oil (MLO) play the role of cross-linkers and the epoxidized linseed oil plays the role of resin. Using resin transfer molding (RTM), flax fibers can be used to obtain composite overlays. Flax strands can be changed using glycidyl-silane, maleic anhydride and amino-silane treatments to assemble a mid polymeric grid of lignocellulosic fibers. Thermal and mechanical properties can be verified by impact, versatile and flexural test, similar to dynamic mechanical assessments to understand the viscoelastic lead. The possible results of Linum usitatissimum in bio-composites don't restrict its usage as a help fiber. Oil acquired from seeds is routinely called linseed oil. It is a triglyceride and its synthesis depends upon such a plant and manufacturing circumstances. Usually, this oil is used as a spread in paint definitions and wood finish due to its drying limit [31]. Notwithstanding this, the elevated unsaturation measure (carbon-carbon twofold securities) present in the critical unsaturated fats, for instance, linolenic and linoleic unsaturated fats with 2 and 3 unsaturation, respectively, provides the opportunity of changing over this plant oil into thermosetting tars. To obtain this, polymerization is caused via reactions including these twofold bonds. Notwithstanding the quick polymerization (homo polymerization) that is achievable and really possible, for the most part, the used course is to change the twofold bonds into valuable get-togethers that can then be viably polymerized on account of reactivity. This is the epoxidation method which changes the twofold securities contained in particular unsaturated fats in oxirane rings through the reaction of oil with peroxo acids. In the last ten years, epoxidized linseed oil (ELO) is used as bio form thermoset polymer in various examinations.

Epoxidized linseed oil can be cross-associated with different assuaging masters, for instance dicarboxylic acids and cyclic anhydrides. Consider that base cyclic anhydrides, for example phthalic anhydride and maleic anhydride, are in solid state at normal temperature. They are used in cross-interfacing epoxidized plant oils. Cyclic anhydrides like DDSA, MHHPA, MTHPA and MNA are comprehensively used as cross-interfacing masters in epoxy pitches for high thermal execution [32, 33].

The use of maleinized linseed oils (MLO) and epoxidized linseed oils (ELO) improves the solidity of ordinary fiber composites. Both misleadingly adjusted plant oils beneficially improve the warm reliability of the ideal framework [34].

Epoxidized linseed oil included with poly (lactic destructive) – PLA composites along with hazelnut shell flour gives a plasticizing effect and change the inferior normal bendable characteristics. The use of epoxidized linseed oil in composites through an interesting strategy to change the low intrinsic delicacy of green composite provides a similar improved warm trustworthiness [35].

3.2 Olive

Agro crop developments fill in as a source material for bio-imperativeness and result in bio-based polymer composites. Wastes of biomass-based filler materials are dynamically used as help material for thermoplastic composite materials. A mixed model can be made by blending olive stone and polypropylene to form another thermoplastic polymer. There are mechanical engineering firms that have produced a homogeneous polymer compound by mixing olive stone as a trademark and biodegradable rough material. Materials, for example sheets, channels and chambers, have been clarified by removal and mixture progressions.

Olive oil extraction saves a great deal of solid form lignocellulosic waste, addressing up to 30–35 wt% of olive natural matter. Their engineered association usually comprises cellulose, lignin and hemi cellulose. Olive waste offers a generating strength as a stronghold filler material in various businesses. Flow olive oil plants use outward segment methods where the olive cake (waste) stores address 35% of the whole olive regular item. They are made from oil (9%), shell (22%), water (24%) and stone (45%). The referenced characteristics varied depending upon the strategy used for olive oil creation [36–38].

Olive waste can be obtained from the development in olive oil extraction. After the separation of olive oil, further progress is made out of skin, pound and stone. Progressively, the solid development can be used with no invention or dissolvable treatment. Olive pomace flotsam and jetsam (Prina trash), which is the remainder of the crush after the pressing of olives, to a plastic material. It is settled that an extension in prina flotsam and jetsam extent caused a development in both concealing change and the mechanical properties of the material [39].

3.3 Palm

The advancements in oil palm, Elaeis guineensis, are widespread at a larger fundamental level than it was a year ago as enthusiasm for vegetable oils increased. The female pack passes on around 2,500–3,000 regular items on 100–120 spikelet joined to a pednex front. Natural objects produce two standard things: palm oil by the outer middle layer and palm bit oil from the inner piece of nut [40]. Palm oil void normal item packages (OPEFBs) are expeditiously open in gigantic sums in palm oil-processing plants. It has been surveyed that number of void regular item packages (EFBs) that are open in Malaysia is consistently around 4.43 million. Presently, 65% is scorched and the pack flotsam and jetsam is reused as manure. In any case, incineration of group flotsam and jetsam isn't biologically recommended, considering the radiation of white smoke with some fly garbage.

Ridzuan et al. [41] uncovered that empty normal item packages (EFBs) are probably suitable for medium-thickness fiber boards (MDFs). Stopping the fiber to remove or flush out its excess oil improves the performance of the medium-thickness fiber board (MDF) manufacture. In addition, it eliminates contamination during the cementation of the sheets. Sodium hydroxide (NaOH) was considered more solid than water to empty the oil. Less fortunate fiber was found with an increased mass thickness and it reduces the mechanical and physical properties.

Palm oil fiber-reinforced polymers have triggered unimaginable enthusiasm amongst scholars and in industries. This is due to some extent the environmentally positive conditions of the customary fiber. Oil palm fiber can be rapidly made into a commercial material. It has been observed by experts that oil palm fiber can be used to help composites. Fiber stacking, fiber length, and fiber treatment impact the mechanical characteristics of composites. From a commercial outlook, the manufacture of palm oil fiber-fortified polymer composites and their subsequent usage is surprisingly alluring, as Malaysia the greatest palm oil-converting countries on the planet. These composites can be filled for artificial composites after some cure or hybridization. Palm oil fiber polymer composites can be made for diverse usage (helper, marine, present day) and custom materials. It is considered that with a comprehensive, accurate and reliable evaluation of fibers, palm oil fiber polymer composites are advantageous.

Palm shell is a solid waste provided by the oil palm industry. Oil palm shell treatment with cold or hot refined water and cold solvent base cure improves the portability and flexible nature of the resulting composite [42].

3.4 Peanut

Nut is one of the world's major consumable harvests. Its scientific name is Arachis hypogaea. It is often given fictitious names as groundnut, as they grow underground. Africa is the top nut producing area after the United States, China and India. Nuts provide large amounts of waste shells. Some industries have tried to explore forced or low-affinity application of these waste stuffs. Nutshells include lignin microfibrils, cellulose and hemicelluloses. In essence, the plans for filler substances are cellulose 35.7 wt%, hemicelluloses 18.7 wt%, lignin 30.2 wt%, protein 8.2 wt%, starch 2.5 wt% and flotsam and jetsam content 4.7 wt% [43]. The phrase, original filler in thermoplastic or trademark filler composites, will be used when anything is said to extend another usage pathway in the difference of agricultural wastes to ancillary devices in plastics organizations.

An assessment of nutshell-powder-strengthened polymer composite is resulting in an extended idea, owing to its remarkable properties and biological counsels. The usage of nutshell-powder-fortified composite can assist in providing reasons for living in both urban and nation domains, despite assisting in reducing waste, similarly as supplying for a predominant condition. Nutshell powder-braced polymer composites have high potential among other ordinary filler-reinforced composites. The most intriguing advancement of nutshell powder continuous polymer composite is its approachability and tilt to deal with a variety of polymer lattices and limitless deposition strategies that have never been previously associated with other trademark fillers. The use of summarized powder-fortified polymer composites, especially in packaging, nuclear family, vehicle and building application, has potential as an uncertainty composite material, which is qualified by its lightweight and simple properties.

Nutrients of polypyrrole, polyaniline, starch, chitosan aniline and chitosan pyrrole can be masterminded using nut waste and can be used for shading from valuable stone violet (CV) water media. Composites show extraordinary desorption properties, which reveals the reusability limit of composites [44].

3.5 Soybean

Soybean oil is manufactured effectively by removing triglyceride particles. Each triglyceride has three unsaturated fat chains incorporated into a glycerol space. Unsaturated fat chains have 0–6 twofold protections and change long from 16 to 22 carbon particles. To discuss an uncontrolled cross-linked thermoset, triglycerides must be incorrectly functional. The supported degree of activation is from 4 to 6 social occasions for each triglyceride. Epoxidized soybean oil is the result of oxidation of soybean oil with hydrogen peroxide and is either acidic or formic destructive. This is achieved by transforming twofold bonds into epoxy social events,

which is non-harmful and have a high reactivity. Triglycerides are first epoxidized and a short time later epoxidized soybean oil is reacted with acrylic, which is sufficiently financially open. The tar is then mixed with a co-monomer such as styrene and allowed to copolymerize through redox using a metal sponsor. Epoxidized soybean oil is largely used as a green plasticizer for polyvinyl chloride, while responsive epoxy bundles explain its unbelievable strength in both monomer amalgamation and polymer course of action.

The impact of epoxidized soybean oil on limit modulus, glass change temperature and mechanical characteristics on epoxy tar composite invigorated by jute surface can be analyzed. By increasing the epoxidized soybean oil content in composite, the glass change temperature increases [45].

Epoxidized soybean oil can react with meth acrylic destructive and, a short time later, by meth acrylic anhydride, resulting in meth acrylic anhydride rooted epoxidized soybean oil sap. Chicken crest can be used as fortifying administrators in polymer composites because of their enhanced unequivocal quality and modulus [46].

3.6 Sunflower

Sunflower acid oil is a consequence of a process of refining of sunflower oil. It is a huge wellspring of free unsaturated fats with a small ratio of glycerides to sterols. Sunflower oil is used to organize alkyd gum by alcoholicization-polyesterification technique in view of its accessibility and straightforwardness. Three assorted alkyd saps are included with sunflower oil using various amounts of phthalic and maleic anhydride. Specific physico-chemical characteristics of sunflower acid oil such as acid value, saponification regard, iodine regard, flighty issue and unsaturated fat creation can be developed. Supporting depiction of gums should be possible using Fourier transformation of infrared and proton nuclear appealing resonation spectroscopic methodologies. The drying characteristics of the alkyd gums were steadily enhanced with the development of maleic anhydride content. The film covering the alkyd saps can be verified by the soaking time, pencil hardness test, cross-cut bond test, shimmer estimation and manufactured resistance test. It would normally be assumed that sunflower oil has an anticipated usage as an unrefined stuff for the purpose of covering the surface. The measurement of the unsaturation unit and fragrant ring assumes an important function in the increase in the characteristics of the gum. The sap film adds to properties, for example, drying time, sparkle, hardness, grip and compound opposition and so forth which may be suitable as a foil for covering the surface and the composite. The sunflower acid oil-based alkyd pitch exhibition was viewed practically as a business tar similar to refined sunflower oil-based alkyd gums. The investigation found that the secondary product of sunflower acid oil, the refined oil, could have a potential use as well as a raw stuff for the covering business, like other vegetable oils.

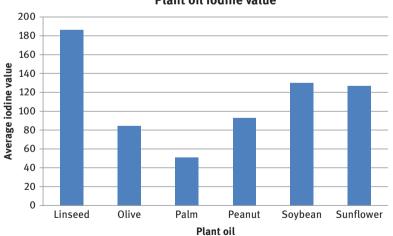
Sunflower oil can be used to obtain alkyd tar by alcoholysis-polyesterification strategy as a result of its openness and the negligible effort needed. Three types of alkyd gums can be blended from sunflower oil by using various ratios of phthalic and maleic anhydride [47].

Vegetable petroleum-processing plants use a mixture of unsaturated fats. The fats are treated with a chemical stock and a mineral resulting in sunflower acid oil. Sunflower acid oil includes free unsaturated fats with limited quantities of acyl glycerols, sogginess and sterols [48]. It has various key characteristics, for example, pleasant condition, negligible effort requirement and availability in large amounts.

Sunflower, as sunflower cake (SFC), is used in the manufacture of composites. The structure of various parts of the sunflower seed (husk, bit and furthermore the extracted protein) determine the plastic-like properties of that can be verified with thermo-gravimetric examination, differential checking calorimetry, and a powerful mechanical warm investigation contraption. In the end, this circuitous method of portrayal demonstrates that sunflower cake (SFC) can be considered as a characteristic composite. Sunflower cake has comparative rheological properties and other physiochemical properties that are ideal for forming or embellishment of plastic-handling hardware.

The sustainable sunflower stalks and wood particles can be used in various proportions in the creation of composite board. The subsequent boards can be used for general purposes such as furniture for indoor conditions [49].

Figure 1 shows a structured presentation of the diverse plant oils contrasted based on the Normal Iodine Incentive. Iodine value is one of the major properties influencing the physical and chemical properties of unsaturated fats. Iodine value



Plant oil iodine value

Fig. 1: Average iodine value of plant oils.

is a proportion of normal unsaturation. Keeping the Iodine values in mind, plant oils can be classified into three categories: Oils with iodine value more than 130 fall under category of drying oils, for example, linseed oil, Oils with iodine value between 100 and 130 fall under the category of semi-drying oils, for example, soybean oil and oils with iodine value below 100 fall under the category of non-drying oils, for example, palm oil. For thermosetting polymer applications, plant oils with a higher iodine value are attractive, meaning that they demonstrate increasingly practical coagulation that encourage an all-too-deep crosslink structure for improved thermal and mechanical strength.

4 Conclusion

Plant-based fibers, resins and fillers have been discussed in this paper. The use of plant-based fibers, resins and fillers are expanding in composites because of their ecological and financial advantages. These composites have fundamentally evolved over the years through nonstop research.

In this paper, all three components for the manufacture of composites, fibers, resins and fillers, have been studied. Oil palm fiber-reinforced composites studies are of "fibers", Peanut shell powder composite studies are of "fillers", and linseed, olive, soybean and sunflower have been studied for "resins".

Hence, it is also observed that there is a lot of scope in plant-based natural fiber composites due to their eco-friendly and bio-degradable properties and that they exhibit superior mechanical properties compared to traditional materials.

The following conclusions have been made on the basis of this study.

- Epoxidized linseed oil improves ductility and flexibility of composites. Also, they help in better fiber-resin interaction.
- In the manufacture of polymer composites with ideal olive shell flour content, one can use a solitary stage process in which olive shell flour, a coupling specialist, and a polymer are legitimately taken care of in the container. Composites with low filler stacking demonstrated great execution when compared with composites that have high filler content.
- Palm oil fiber-fortified polymers have triggered extraordinary enthusiasm among scholars and in industry. Palm oil fiber composites have been developed for many purposes such as industrial, structural and marine applications.
- Peanut shell powder acts as a filler in the fabrication of composites. It helps to reduce waste and there are countless manufacturing processes for this particular type of composite. It is of low cost and is light in weight.
- Soy oil resin-based composites have potential to manufacture large-sized materials for building construction. It has high storage modulus.
- Sunflower acid oil resin-based composites are a potential source of raw materials.

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Nisha Kumari and Kaushik Kumar Orthopedic application of plant-based composites: a case on orthotic calipers

Abstract: Natural fillers play a major role in industries and the global market due to their eco-friendly nature and biodegradability. Researchers/scientists have also drawn their attention toward utilization of polymer composite reinforced with natural fillers, which are obtained from nature: that is, plant based or animal based. The main objective of the authors' work is to use bark-based and fruit-based reinforcement with epoxy as matrix to create the braces of orthotic calipers. The mechanical behavior, density and void content, tensile and flexural test are recorded and then compared with the currently used material. After the comparison, it is observed that the specific strength and stiffness of bark-based reinforced composite is more than that of presently used aluminum alloy. The distribution of fillers in the matrix is also studied to check the microstructure of composites scanning electron microscope.

Keywords: polymer composites, natural filler, orthotic calipers, epoxy, wood, coir, microstructure, mechanical behavior, density, void content

1 Introduction

Increase in population has led to various problems including pollution, degradation, loss of flora and fauna, deterioration of environment by cutting down the forest and various disabilities, which may spread from virus and are infectious. The Environment Regulation Act has been passed to safeguard the environment and also emphasizes the use of natural resources. One disease which we are going to focus on is Poliomyelitis/Polio.

Poliomyelitis is a highly contagious disease that shows two different patterns. One is major illness, and the other is minor illness. Major illness affects the central nervous system, which, in the long run, may cause paralysis, whereas minor illness leads to nausea, vomiting and influenza-like illness. In about 0.4 percent of total cases, the infected person has problems related to the gait, movement or feeble joints and muscles [1]. Mostly, the cases affect the lower abdomen and get communicated from person to person via contaminated food, water and human feces. Polio began to

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affect millions, thousand years back in Europe and the United States, due to which researchers and scientists started working on the vaccine as well as aids/appliances that would help those with this kind of disability. Orthotic calipers were discovered to help the patients support their weak joints [2]. Orthotic calipers/braces gave adults and children affected by polio, hope to live their life on their own terms.. The functions of orthotic calipers include alignment of muscles, correcting the shape of the feeble legs, aiding in rehabilitation and decreasing the pain. The existing orthotic braces/calipers are shown in Fig. 1.



Fig. 1: orthotic braces/calipers.

In the early 1900s, patients were using calipers made of wood according to their shape and size. Then, plaster of Paris mold came into the market followed by Jaipur Foot by P. K Sethi, in the year 1969. Later, with the evolution of the industry, materials began to play a major role in manufacturing and production of orthotic calipers. Materials play a major role in designing of calipers as those that were earlier made had many disadvantages. As the years passed, researchers/scientists came up with new ideas and new materials. Patients required calipers that had good mechanical properties, that is, high tensile strength and stiffness and ease in fabrication as also those that were low cost, had low weight-to-volume

ratio and were environment friendly [3]. When iron was chosen for manufacture of calipers, it turned out to be heaviest, as patients were unable to bear the load of the iron and walk with it. Next, steel, and then, finally, aluminum became of the best materials for orthotic calipers. Aluminum still has a few cons, namely its weight and strength and most importantly the fact that it is manmade and not natural. Currently, researchers are trying their best to increase use of natural resources and get a product that exhibits a wide range of good properties that satisfy polio-infected patients. Due to the need and to make calipers that fit the child even when they grow up to become adults, special materials are required to fulfil the needs [4].

One solution lies in composite materials. Composite material is the need of the hour due to its wide range of applications and characteristics [5]. When compared to aluminum, composites are lighter. If we consider thermal expansion, then we get to see that thermal expansion of composites is almost negligible. They have high specific stiffness (stiffness to density ratio) due to which they get to play an important role in the field of aerospace and automobile industries [6]. When a product is made of composite, there is no need for any machining operation, as it provides an excellent surface finish with greater dimensional accuracy. Now-a-days, sensors are embedded in smart materials for service monitoring. With all the inherent advantages, composites are used in various fields such as marine, medicine, energy, sports, construction, transportation, etc. [7, 8].

Composite material or *composition material* can be defined as a material which is a combination of two different materials (having different physical or chemical properties), which includes matrix and reinforcement [9]. The final product obtained after mixing of the matrix and reinforcement results in a material that is said to be composite. The product will have better properties than those of matrix and reinforcement. Matrix helps in binding of the fibers, whereas reinforcement aids in enhancing the mechanical properties [10]. The fibers are reinforced in composites. Primarily, matrix materials include ceramic, polymers and metals. Polymers are nothing but plastics or resin and if fibers are reinforced in them, it is termed as composite material [11]. Some of the typical engineered composite materials are polymer matrix composite, metal matrix composite, ceramic matrix composite, composite wood such as plywood, reinforced concrete and other advanced composites.

Polymers can be categorized into plastics and elastomers. Plastics can be thermoplastic or thermoset [12]. Elastomers can be natural or synthetic. To ensure the safety of Mother Earth and Nature, it is mandatory to draw attention toward natural fibers that are readily available and are natural. After research and literature review it has been established that researchers are trying their best to eliminate synthetic fibers by using natural ones. One of the basic differences between natural and synthetic is that we get natural ones from plants and animals, whereas synthetic is manmade and is made of chemicals. Natural fibers are considered to be environment friendly and sustainable materials, when compared to synthetic ones [13]. Presently, the biggest concern globally is ecological and environment issues and the need to showcase a great variety of materials and polymeric-based composites [14]. Polymeric-based composites enhance the properties of composites if used with fillers that are natural. Natural fibers have shown great promise in industry as well as in global markets because they are easy to dispose after use, environment friendly, abundant and have high specific strength and modulus when compared to other fibers such as carbon fibers, glass, organic and ceramic. Glass fibers have properties similar to natural fibers, but are getting replaced due to potential and economic advantages such as less density, high specific strength, renewability and biodegradability [15].

According to the reports of "Discover Natural Fibers Initiative" and "Agricultural Organization of United Nations" that mainly work on the 14 categories of natural fibers and its statistics around the globe, by the end of year 2018, the world's natural fiber production was estimated to be around 32 million metric tons. Natural fibers include some plant-based fibers, namely, cotton, jute, hemp and flax. Animal-based fibers range from abaca to wool to silk. Natural fiber production has seen a growth from 28 to 35 million metric tons in past few decades. With the dynamic increase in production of natural fiber, the market is estimated to grow by more than 9–10%.

As discussed, natural fibers are obtained from different plant and animal resources. For purposes of widening, they are reinforced with polymer-based composites and were once used in low-scale applications such as making of mats, carpets, handbags and many more daily use products [16, 17]. From past literature, we know that the quality of plant-based natural fibers mainly depends on the age, locality, extraction and the process of extraction. There has been extensive research on natural fibers such as sisal, jute, coconut, pineapple, hemp and banana for industrial applications. As natural fibers are enriched with cellulose content, they possess good mechanical properties [18]. Natural fibers are drawn out from all parts of plant such as bast, leaf, fruit, bark and seed. Fibers that we get from bast are hemp, flax, kenaf, ramie, roselle and jute. From the leaf, abaca, pineapple and banana are obtained. Seed fibers include cotton and kapok, fruit fibers include coir, tamarind, sisal and crown; and from the bark fiber, we get rice and bamboo.

Bast fiber, which is most commonly used, refers to wood which is covered by a stem. Inside that stem, there are bundles that consist of fiber cells or ligaments, which have hemicellulose or cellulose. It is learnt that if there is high cellulose content in the stem, it will result in high tensile strength; and this is mostly seen in cotton, oil palm, wood and fruit bunch [19]. Cellulose with good strength and properties can be seen in vegetable and plant crops that are used by us daily. It has been reported that fibers (abaca, sisal, coir, jute, palm, etc.) are identified and characterized as suitable for practical applications due to their mechanical properties [20]. Bio fibers find application in industries, automotive applications and automotive due to high tensile strength [21]. Along with bio fibers various waste products are also being used as reinforcement for different polymeric-based composites. The

use of natural fibers with composite materials is increasing day-by-day, but a few considerations such as wear and frictional performance need to be taken care of, before according them importance [22].

Recently, a lot of research has been done on bark fiber and its use in the production of nanofibers [23, 24]. It is important to find the cellulose content in each part of the plant variant so as to know which one gives the best tensile strength. The composition of natural fibers that reinforce polymeric-based composites is of extreme importance for manufacturing a composite material that satisfies the functionality required by infected patients.

In the present work, the side sticks of orthotic calipers, which are made of aluminum, are made of fabricated epoxy-reinforced composite. Epoxy is reinforced with coir dust, which is a fruit-based filler and wood which is a bark-based filler. Mechanical behavior of the natural filler-reinforced composites including density and void content, hardness, tensile strength, flexural property, etc. is examined and compared with the existing material (aluminum). To study the dispersion of filler in the composite, microstructure of the composite has also been studied, using SEM.

2 Materials used

2.1 Epoxy

Resin can be of various types such as polyester, epoxy, polyamide and phenolic. Here, the type of resin is epoxy. Epoxy resins are available in the form of powder, liquids, paste, etc. [25] To prepare polymeric-based matrix, low solidifying curing epoxy resin (standard epoxy LY 556) that belongs to the "epoxide" family and hardener (HY 951) were taken in equal ratio, as stated by the manufacturer. Epoxy is used as a matrix because of its versatility and broad range of properties. It exhibits low shrinkage as well as excellent adhesion. It provides excellent adhesion to wide range of fillers, fibers and other substances [26]. Epoxies are reinforced with synthetic as well as natural fillers such as glass, aramid, carbon, jute, hemp, coir, flax, etc. due to their low impact strength and brittleness. It is widely used in aerospace and automobile applications.

2.2 Wood

Wood as filler is usually known as lignocellulosic elements that are picked from bark of the trees. The main components of wood filler are lignin, cellulose and hemicellulose. Cellulose in wood fillers help polymer-based composites gain mechanical strength [27]. It is used in making paper and composites. Wood provides low cost, low weight-to -volume ratio and higher strength and firmness, is easily available, and recyclable, shows ease in fabrication and is resistant to chemical, due to which it is used in furniture making, packaging and construction [28–30]. To make wood fillers strong, it is generally combined with thermoplastics and aluminum foil.

Wood dust is used as the reinforcing natural filler [31, 32]. It is obtained in abundance from saw mill wastes and is often used after segregation into correct sizes, using a sieve shaker.

2.3 Coconut coir

Coconut shell filler is obtained from the fruit and is an important natural filler found mainly in tropical countries. It is used to make paper, textiles, composites and ropes. Researchers/scientists use this material as filler due to its higher strength and modulus properties in creating automotive components. Coir filler provides high wear resistance, hardness and strength. Moreover, they are non-corrosive and non-hazardous [33] when compared to glass.

3 Conditioning of fillers

Being hygroscopic in nature, both the fillers should be dried efficaciously by conventional hot air dryers. Hot air dryers are not efficient in cutting down the moisture content. Wood filler requires low variation in dew-point (under 32 °C) and fixed drying temperature that ensure moisture content of 0.02% or lesser. As soon as the required conditions are reached, the composites showcase the desired optimal mechanical properties. Drying of wood fillers is done in a dehumidifying chamber (Fig. 2) at 80 °C for about 6 hours.

3.1 Coir filler

Coir fillers are extracted from the outermost layer of the pericarp of fruits and are kept in the dehumidifying chamber at 80 °C for about 24 hours [34], for complete drying. Proper drying of the fillers is essential to affirm the phenomena of adhesion across the filler surface for complete transfer of strength from matrix to filler, resulting in high strength. To further increase the adhesion, coconut coir fillers are soaked in 2% NaOH solution. After soaking, it is repeatedly washed with distilled water till the fillers become non- alkaline. This is tested with the aid of a litmus paper. The fillers are then dried again, in the oven, as per the parameters provided in Table 1.



Fig. 2: Dehumidifier for preheating of the fillers.

Table 1: Conditioning of natural fillers.

Material	Dried temperature	Time (in hours)
Wood	50 °C	24
Coir	80 °C	24

4 Composite fabrication

For the preparation of composites, epoxy is mixed with different natural fillers (wood and coir dust). The mixture obtained is used to prepare the specimen. As hand lay-up technique is used during mixing of the resin with hardener and fillers, a lot of air bubbles get trapped in, which results in void formation on curing. Hence, after mixing thoroughly and before curing, the mixing is done under vacuum in a vacuum chamber, where the mixture is kept in the upper chamber. Slowly, it is poured in the specimen mold kept in the lower chamber. All these activities are undertaken at -1 mmHg pressure, and the specimen is kept there for approximately 6 hours for complete curing. After completion of the curing process, the specimen is taken out of the mold and used for further experimental analysis. Table 2 shows the fillers' particle size and density of composite with epoxy (density 1.26 g/cc) as matrix mixed with 10% respective filler.

Material	Size	Density
Wood	70–80 µm	0.72 g/cc
Coir	70–80 µm	0.67 g/cc

Table 2: Size of particulate filler along withrespective composite density.

4.1 Experimental testing

4.1.1 Materials and composite fabrication

4.1.1.1 Matrix material

In this work, for making the matrix, epoxy resin (Standard Epoxy LY 556) which will be cured at room temperature and the corresponding hardener (HY 951) would be mixed in a manufacturer-defined ratio.

4.1.1.2 Filler material

Wood dust and coconut coir dust will be taken as the filler material.

4.1.1.3 Conditioning of the filler

Before the fabrication, it is important to dry natural fillers as they have tendency to absorb moisture from the air. Due to their nature, they are not completely dried by conventional hot air dryers, as hot air dryers are dependent on the surrounding air/ environment. They are not a good option for drying of natural fillers because they are inept in reducing water content. To get optimum mechanical performance from the fillers, consistent low dew-point dry air and temperature are required. The optimum dew-point dry air should be below 32 °C and the resultant moisture content should be 0.02% or less than that will also work. To attain this result, it is always recommended to dry wood fillers in a dehumidifier at a fixed temperature and time.

4.1.1.4 Composite fabrication

For the fabrication of composites, the fillers is toned to have a particular particle size and density, which is ensured by the sieve shaker; after cleaning and drying, they would be reinforced in epoxy resin. The mixture thus obtained (epoxy filled with natural filler) would be used to prepare the desired specimen. With the help of a vacuum casting chamber (Fig. 3), casting is performed so that air bubbles and imperfections can be avoided. There are two vacuum casting units – in one chamber resin and the corresponding hardener are placed, and in the upper chamber, mixing will be done with the chosen filler. The casting of the mixture will take place in the lower chamber



Fig. 3: Vacuum casting chamber.

in a rectangular glass mold coated with glass paper for easy removal of the mold. To ensure complete curing, at a specified temperature and time, the mold with composite would then be kept in a hot air oven. After the curing ends, the specimens (Fig. 4) are taken out from the mold and used for further procedure.

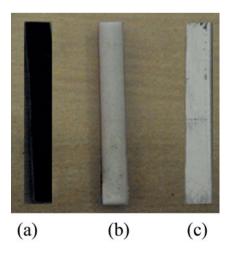


Fig. 4: Samples for testing (a) composite with coir; (b) composite with wood; (c) aluminum bar from the existing calipers.

4.1.1.5 Testing details

4.1.1.5.1 Void content

Density is ratio of mass of the substance per unit volume. It depends on the structure of the composite. Theoretical density is evaluated using weight additive principle, and actual density is determined by water immersion method with the aid of Mettler Toledo electronic balance (Fig. 5). The difference in both the densities provides the amount of impurity (here, air bubble or void) in the composite. Void content is one of the important defects studied in fabrication of fiber-reinforced composite. Investigations about void would show the void formation, characteristics and mechanical effects of the composites [35]. To estimate the quality of the composite, void percentage is important.



Fig. 5: Mettler Toledo electronic balance.

4.1.1.5.2 Tensile and flexural tests

Instron Universal Testing Machine (Instron Ltd., UK) (Fig. 6) has been used for both tensile and flexural test of the prepared samples. ASTM D 638 serves as the guideline for conducting the tensile tests, whereas ASTM D 790 provides the standard for flexural or 3-point bending test. For both the tests, samples of size 100 mm X 12 mm X 8 mm were taken. The ambient temperature was 24 °C, and relative humidity was maintained at 55%. For both the tests, crosshead speed or strain rate was kept constant at 1 mm/min.

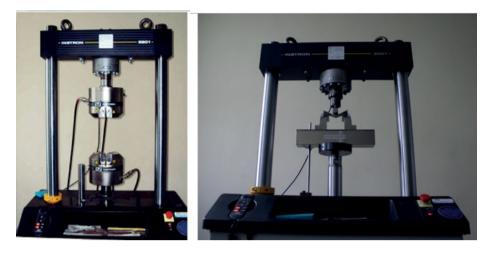


Fig. 6: Instron universal testing machine.

4.1.1.6 Hardness test

The resistance property of materials toward scratch, indent or deform is Hardness. Hardness test is categorized into three: Vickers hardness test, Brinell hardness test



Fig. 7: Vicker's hardness test setup.

and Rockwell Hardness test. Hardness test discloses the usage and dimensional ability of the composite for the orthotic calipers. In this test, indent plays a major role, as it varies according to the test that is performed. Indent can be a steel ball that has a fixed diameter, square pyramid or diamond cone. The extent of penetration of the indent indicates the extent of resistance to deformation, and it has great importance in product design. This, apart from giving stable dimensions, also indicates the strength and homogeneity in the structure.

In this study, microhardness of the prepared composite is evaluated using Vickers's Microhardness Tester. The instrument is a UHL microhardness tester (Model – VMHT MOT, Sl. No. 1002001, Technische Mikroskopie) (Fig. 7) with a Vickers diamond indenter. The dwell time which varies from 10–15 seconds and speed of indentation and applied load are kept constant.

4.1.1.7 Morphological study

The homogeneity of the prepared composite or the even distribution of the filler in matrix is studied through morphological study. If the characterization is known for a developed material, it becomes easy for the manufacturer to understand the reasons for variation of its properties, physical behavior and distribution of fillers into



Fig. 8: Scanning electron microscopy setup.

the matrix and performance during investigation. Hence, morphological techniques to study and characterize materials are in demand. Microstructure characterization of a material is useful in order to observe the microstructure in detail and is usually observed under a microscope. Generally, optical microscope, scanning electron Microscope images, etc. are used.

In this study, scanning electron microscopy (SEM) examinations are undertaken to observe the homogeneity of the filler distribution in the matrix for the prepared samples. The surface of composites is required to be covered with thin platinum coating, and the images are studied through a JEOL (model JSM 6390LV, Japan) microscope (Fig. 8).

4.1.2 Tribological test

The wear characteristics of any material are studied using the tribological tests. Here, a multi-tribotester (TR25 (Ducom, India) was considered (Fig. 9)), without lubrication. The testing conditions, that is, ambient temperature, relative humidity, etc. were as per the standard. The sample (size $20 \times 20 \times 8 \text{ mm}^3$) was kept firm in the sample holder of the instrument and was made to slide against the steel roller that acted as a counterface. A normal load was applied by placing dead weights on the loading pan on the top of the specimen, with the aid of a loading lever. The experimental data of coefficient of friction (COF) gets recorded on the computer that is linked to the testing apparatus. Specific wear rate is evaluated using the

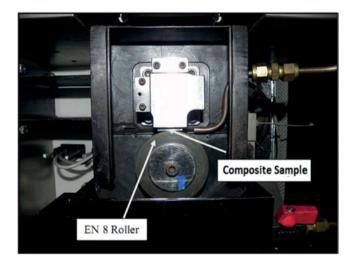


Fig. 9: Block-on-roller multi-tribotester setup.

weight loss of the composite for which samples are weighed before and after the experiments in a Mettle Toddler electronic balance.

5 Results and discussion

5.1 Void content

For polymer-based composites, the void content is calculated by taking the difference between the experimental and theoretical data. Table 3 shows that the experimental data is less than the theoretical data, which is an indication of the presence of void inside the fabricated composite. Voids typically showcase poor manufacturing of the material and mostly affect the mechanical properties and lifespan of the composites. For industrial and commercial applications, a void content of 1% is still viable. Voids present in composites is mostly due to impregnation, surface morphology, excessive quantity of resin being poured and curing parameters. Still, there is possibility of reducing void by combining vacuum bagging system and autoclave under constant parameters.

Composite	Density (g/cc)		Void fraction
	Experimental	Theoretical	(%)
Epoxy + wood dust	0.932	0.939	0.75
Epoxy + coconut coir dust	0.941	0.948	0.74

Table 3: Void content of composites.

5.2 Tensile and flexural tests

To carry out tensile test, the specimen is placed between the grips and then pulled until failure. It is carried out in accordance to ASTM D 138 with a maximum load of 1 kN, with gauge length of 0.5 cm and crosshead speed of 1 mm/ min. Data such as tensile strength and tensile modulus are recorded in the computer attached to the machine. Similarly, for flexural test/3-point bending test, ASTM D 790 standard is used, and the test is carried out by initiating load on the specimen, till its rupture. In flexural test, the gauge length of the specimen is taken as 0.5 cm with crosshead of 1 mm/min. with the help of 3-point bending flexural strength, and flexural modulus is recorded. The results of composite with coir dust have superior properties when compared with those of wood dust-reinforced composite as shown in Fig. 10.

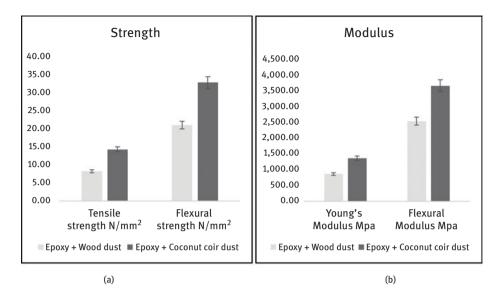


Fig. 10: Tensile and flexural properties: (a) strength; (b) modulus.

5.3 Hardness test

In this work, Vicker's hardness test setup is used to determine the microhardness value that ranges from 1–50 μ m. The indentation load is 100 gf, speed of the indentation is set as 50 μ m/s, and dwell time for holding the indent load is 10 seconds. With the help of a touch screen-based system, which is part of the tester, the microhardness tester is controlled. The hardness numbers are obtained by pressing the indent and producing indentation on the specimen. Three values are recorded and their average is taken into consideration. The hardness of coir-based sample is found to be better than the wood-based sample.

5.4 Tribological tests

Tribological test was undertaken in multi-tribotester. The specific wear rate and friction coefficient was calculated, and in tune with the other tests, it is seen that the coefficient of friction and specific wear rate are less in wood fillers when compared to coir.

5.5 Morphological study

SEM is one of the most common methods used for determining the morphology and microstructure of the matrix and the filler used in composites. Here, for scanning the

surface of the sample, a low energy electron beam is radiated, resulting in emission of photons and electrons. From the image (Fig. 11), dispersion of filler in the matrix uniformly can be seen clearly.

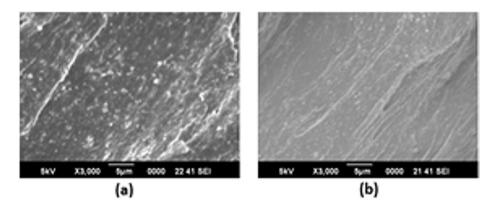


Fig. 11: Scanning electron microscopy images: (a) epoxy + wood dust composite; (b) epoxy + coconut coir dust composite.

5.6 Conclusion

Currently aluminum alloy is being used in calipers. Initially, steel was used, but the material changed to aluminum alloy due to its low self-weight. Though the processing of aluminum alloy is easier, its low elastic modulus is a major drawback together with the cost factor. The present paper used plant-based polymeric composite as an alternative. The experiment proved that the polymeric composite has a higher loadbearing capacity when compared to the aluminum one. Moreover, due to its characteristically low weight-to-volume ratio, the change of material would provide ease in mobility to the user. The tribological tests provide low friction and specific wear that would support longevity for the calipers. Out of the two reinforcements used, one from the bark of the tree and the other from the fruit, that from the fruit provided better results due to low lignin and high cellulose content that provides more bondage between the matrix and reinforcement, resulting in better strength and less wear.

5.7 Future scope

The future scope of the work can be divided into matrix, reinforcement materials, size, processing and testing.

5.7.1 Matrix

Other thermoset materials like polyester, vinyl ester, etc. can be used in place of epoxy. Thermoplastic materials like acetal, acrylonitrile butadiene styrene (ABS), high density polyethylene (HDPE), nylon (PA), polybutylene succinate (PS), polybutylene-terephthalate, polycarbonate, poly ether ketone, etc. can be used as a matrix.

5.7.2 Reinforcement materials

Other plant- and animal-based materials can be used as reinforcement. Materials from other parts of the plants like roots, leaves, etc. can be tested based on the lignin and cellulose percentage.

5.7.3 Size

The size of reinforcement materials can be changed from macro to nano level; moreover, instead of fillers, fibers as also combination of fibers and fillers (hybrid) can be experimented with.

5.7.4 Processing

In the present chapter, vacuum-assisted hand lay-up technique has been used. Other techniques like pressure casting, injection molding or compression molding can be employed for better mixing and better strength.

5.7.5 Testing

In this chapter, only mechanical and tribological testing have been included. Other tests like thermal degradation, water absorption, buckling, fatigue analysis, etc. are required to be conducted before clinical use.

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R. Uzwalkiran and B. Sridhar Babu Development and mechanical characterization of jute/polypropylene/coir composites

Abstract: The aim of this chapter is to study the mechanical potential of poly propylene, jute and coir fiber based composites by conducting experiments. Polypropylene, polypropylene reinforced with jute and polypropylene reinforced with jute and coir composites are three types of composite specimens that were developed using the injection molding method. Experimental results show that the incorporation of jute and coir fibers into the polypropylene matrix has resulted in an average improvement in the tensile, flexural and impact properties of the natural fiber composites.

Keywords: polypropylene, jute, coir, tensile strength, compressive strength, volume fraction, fabrication, composite, impact strength

1 Introduction

Reinforced plastics are mainly used in applications because they are inexpensive, are renewable, nontoxic, biodegradable and are replacements for carbon and glass fibers [1–5]. There are many advantages of natural fiber composites compared to glass and carbon fibers because of their low density, low weight, low energy consumption and good mechanical properties. Polypropylene (PP), polyethylene, and poly (vinyl chloride) are mainly used as matrices in natural fiber-based composites. For the last few years, researchers are studying the use and the mechanical and material properties of natural fiber-based composites by reinforcing fibers such as jute, coir, straw, rice husk, pineapple, banana, bamboo, etc. There is high demand for lightweight materials in the automotive industry for their good abatement property and their low weight for fuel efficiency [6–13]. Natural fibers offer good efficiency and demonstrate high shatter-resistant energy characteristics than other polymer based composites. In automobile applications, natural fiber based composites not only reduce the weight of the automobile parts but also lower the energy needed for manufacturing by 90% [14–18]. Eco-friendly composites can be developed to replace the carbon/ glass -based composites with different types of natural cellulose fiber-reinforced composites. But these

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natural fiber composites exhibit disadvantage such as low load-bearing capability compared to other polymer composites [19–25]. Change in the mechanical properties of natural fibers is another parameter that has to be considered when compared with the other composites [26, 27]. In this chapter, development of polypropylene, jute and coir based hybrid composites are discussed and the mechanical characterization studied by conducting tensile, compressive and impact tests.

2 Materials

The polypropylene crystals, jute, coir shown in Figs. 1, 2 & 3 were are used for preparing the specimens.



Fig. 1: Polypropylene crystals.







Fig. 3: Coir.

3 Fabrication process of the composite specimens

3.1 Preparation of the jute powder

- Jute shown in Fig. 4 was dried in sun for two days and microwaved to remove the moisture from it. The dried jute shown in Fig. 5.
- The dried jute is taken out and then converted into powder form as shown in Fig. 6 using a mixer.



Fig. 4: Jute with moisture.



Fig. 5: Dried jute.



Fig. 6: Powdered jute.

3.2 Preparation of coir powder

- Coir shown in Fig. 7 was dried in sun for two days and microwaved to make it free from moisture.
- The dried coir shown in Fig. 8 was separated and made into flakes. The dried coir was finally converted to powder and kept ready for further processes.
- After making the coir dry, it was crushed to form dried flakes of coir so as to obtain powdered coir shown in Fig. 9.



Fig. 7: Coir before drying.



Fig. 8: Coir after drying.



Fig. 9: Powdered coir.

3.3 Proportions and mixtures

Various compositions in percentage/ weight

- Specimen 1. Polypropylene: 100%
- Specimen 2. Polypropylene + jute: 75% + 25%
- Specimen 3. Polypropylene + coir: 80% + 20%
- Specimen 4. Polypropylene + jute + coir: 80% + 08% + 12%

The raw materials are thoroughly mixed in above proportions. The mixed proportions are shown in the following figures.

3.4 Preparation of specimen 1 (polypropylene 100%)

- The polypropylene granules are fed into the hopper of the injection molding machine and a temperature of 210–220 °C was maintained for the uniform liquidification of polypropylene granules.
- After the proper melting of granules, the liquid polypropylene is injected into the mold through the nozzle.
- Thereby, the liquid polypropylene acquires the shape of mold and is collected after cooling as shown in Fig. 10.
- Similarly, the liquid is injected into two further different molds and the specimens are collected.

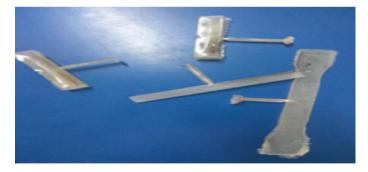


Fig. 10: Pure polypropylene specimen.

3.5 Preparation of specimen 2

- Proportion of specimen 2 75% PPE + 25% jute.
- In this preparation, 75 g of polypropylene and 25 g of powdered jute are taken and they are thoroughly mixed.

- This mixture is added to the injection molding machine through the hopper and a temperature of 210 °C is maintained.
- This results in the eventual melting of the mixture inside the bore and this melted mixture is collected as shown in Fig. 11.



Fig. 11: Melted mixture of polypropylene and jute.

 This mixture, when cooled, is taken and ground into granules by using a grinding machine.



Fig. 12: Ground mixture of jute and PPE.

- Again, this ground mixture is fed to the hopper with some amount of PPE and further, this is melted and injected into three different types of molds.
- These three specimens are collected after cooling as shown in Figs. 12 & 13.



Fig. 13: Jute and PPE specimens.

3.6 Preparation of specimen 3

- Proportion of specimen 2–80% PPE + 20% coir shown in Fig. 14.
- In this preparation, 80 g of polypropylene and 20 g of powdered coir are taken and thoroughly mixed.
- This mixture is added to the injection molding machine through the hopper and a temperature of 210 °C is maintained.
- This results in the eventual melting of the mixture inside the bore and this melted mixture is collected.



Fig. 14: Melted mixture of PPE and coir.

- This mixture, when cooled, is taken and ground into granules using a grinding machine.



Fig. 15: Ground granules of coir and PPE.

- Again, these grounded mixtures are fed to the hopper with some amount of PPE and this further is melted and injected into three different types of molds.
- These three specimens are collected after cooling shown in Figs. 15 & 16.



Fig. 16: Coir and PPE specimen.

3.7 Preparation of specimen 4

- Proportion of specimen 4-80% PPE + 08% jute + 12% coir.
- In this preparation, 80 g of polypropylene, 8 g of powdered jute and 12 g of coir are taken and thoroughly mixed.
- This mixture is added to the injection molding machine through the hopper and a temperature of 210 °C is maintained.
- This results in the eventual melting of the mixture shown in Fig. 17 inside the bore and this melted mixture is collected.



Fig. 17: Melted jute coir and PPE.

- This mixture, when cooled, is taken and ground into granules using a grinding machine.



Fig. 18: Ground form of PPE coir and jute.

- Again, the ground mixture is fed to the hopper with some amount of PPE, shown in Fig. 18 and this is further melted and injected into three different types of molds.
- These three specimens are collected after cooling shown in Fig. 19.



Fig. 19: Specimens of PPE, coir and jute.

4 Results and analysis

4.1 Evaluation of the tensile properties of specimens

Tensile properties of the specimens were measured with respect to ASTM D 3093/ D3039 M standards with composite specimen dimensions of $200 \times 24 \times 6$ mm using Al strips of 0.8 mm thickness to avoid gripping damage. A UTM (Model 55 R 4206, Japan) was used to determine the peak load and the ultimate tensile strength of the four specimens with a crosshead speed of 1 mm/min. Table 1 shows the peak load and tensile strength of the specimens which are prepared with polypropylene, jute and coir. The results showed that the peak load and ultimate tensile properties gradually increased from specimen 1 to specimen 4. That means specimen 4 has better tensile properties compared to the other specimens. The specimen 4 (polypropylene + jute + coir) is most suitable for industrial applications compared to other composites.

-			
S. no.	Specimen no.	Peak load	Ultimate tensile strength
1	Specimen 1	0.500 kN	11.014 N/mm ²
2	Specimen 2	1.150 kN	24.956 N/mm ²
3	Specimen 3	0.730 kN	17.810 N/mm ²
4	Specimen 4	1.000 kN	23.27 N/mm ²

Table 1: Peak load and tensile strength of the specimens.

4.2 Evaluation of compressive properties of specimens

Compressive strength properties of the 4 specimens were determined with respect to the ASTM D 3093/D3039 M standards. A UTM (Model 55 R 4206, Japan) was used to determine the peak load and the compressive strength of the specimens with a cross-head speed of 1 mm/min. Table 2 shows the peak load and compressive strength of

S. no.	Specimen no.	Peak load	Compressive strength
1	Specimen 1	0.230 KN	5.673 N/mm ²
2	Specimen 2	0.230 KN	5.673 N/mm ²
3	Specimen 3	0.290 KN	7.121 N/mm ²
4	Specimen 4	0.260 KN	6.363 N/mm ²

Table 2: Peak loads and compressive strength of the specimens.

the specimens which are prepared with polypropylene, jute and coir, The results showed that the peak load and ultimate tensile properties gradually increased from specimen 1 to specimen 4. This means that specimen 4 has better compressive properties compared to other specimens. Specimen 4 (polypropylene + jute + coir) is most suitable for industrial applications compared to other composites.

4.3 Evaluation of impact energy of specimens

Impact tests were conducted using charpy impact testing equipment to calculate the resistance of the composites to breakage flexural shocks caused by the pendulum-type hammers with one pendulum swing. The results of these impact tests show the energy absorbed per unit of specimen width. Impact tests were conducted on specimens with dimensions 120 mm × 11.7 mm × 2.6 mm with respect to ASTM D 6110–97. The Table 3 shows the impact energy of the specimens. From this table it can be observed that the resistance was constant for the all 4 types of specimens.

Table 3: Impact energy of the specimens.

S. no.	Specimen no.	Impact energy (Joules)
1	Specimen 1	2
2	Specimen 2	2
3	Specimen 3	2
4	Specimen 4	2

5 Conclusions

- 1. The aim of the chapter is to study the potential of coir, jute and poly propylene fiber-reinforced polymer composites and to investigate the mechanical behavior of the composites.
- 2. The review reports suggest that jute and coir fibers can be used as reinforcements in the polypropylene matrix.
- 3. The incorporation of jute and coir fibers into the polypropylene matrix has resulted in an average improvement in the tensile, compressive and impact properties of the composites.
- 4. The tensile, compressive and impact strengths of the pure polypropylene matrix are less than that of jute or coir fibers-reinforced polypropylene matrix.

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Chikesh Ranjan Mechanical behavior of plant-based composite: a case with rice straw

Abstract: The objective of the present chapter is to study plant-based composite. In many countries, burning of rice straw creates problems in terms of smoke. This smoke is very harmful to the environment. A lot of research work has been done on natural fiber plant-based composite. Rice straw fiber has improved tensile, impact and Young's modulus. In this chapter, we discuss about preparation of different types of rice straw fiber with epoxy composite. Rice straw fiber is used in untreated form, treated with hot water or with NaOH, with different volume fractions. The mechanical properties of all samples have been compared and analyzed. All testing was done according to ISO and ASTM standards.

Keywords: rice straw, epoxy, NaOH, tensile, impact

1 Introduction

Presently, researchers and industries are working on natural fiber-reinforced biodegradable composites. Composite materials are made by two or more materials that have different physical and chemical properties. The property of the composite material property depends on the property of fibers and resin used. At present, natural fibers are being used quite often in composite materials. Many researchers have worked on biodegradable plant-based composite materials. Figure 1 shows the document types of published articles related to plant-based composites. Major publications are available as articles and conference papers, that is, 69.8% and 17.5%, respectively. Books that have been published account for around 12.7%. Figure 2 shows the document types of published articles related to rice straw composite. Major publications are available as articles and conference papers, that is, 78.5% and 13.2%, respectively. Books that have been published account for around 8.3%.

Figure 3 shows the document by subject area of published articles on plantbased composite. Plant-based composite is used in the engineering field, that is about18.4%. In material science, chemical and environmental science, articles published account for around 15.9%, 12.4% and 12% respectively.

Figure 4 shows the document by subject area of published articles on rice straw composite. Rice straw-based composite is used in engineering field, that is about

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Documents by type

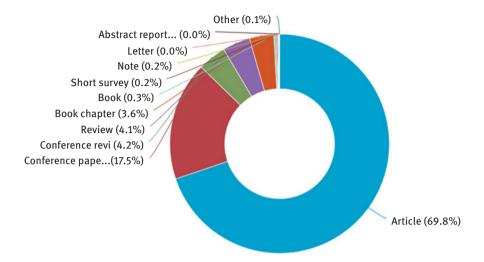


Fig. 1: Document types publishing the articles related to plant-based composite (Source: https:// www.scopus.com).

Documents by type

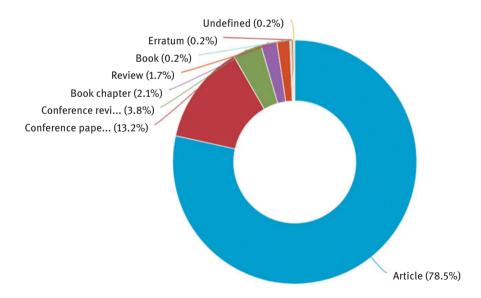
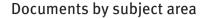


Fig. 2: Document types of published articles on rice straw composite (Source: https://www.scopus. com).



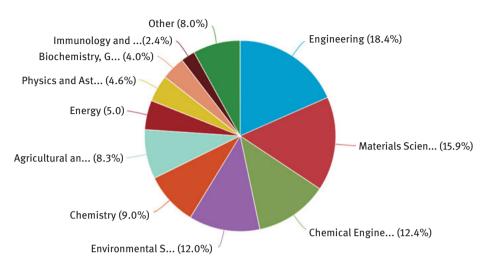


Fig. 3: Document by subject area of published articles on plant-based composite (Source: https://www.scopus.com).

Documents by subject area

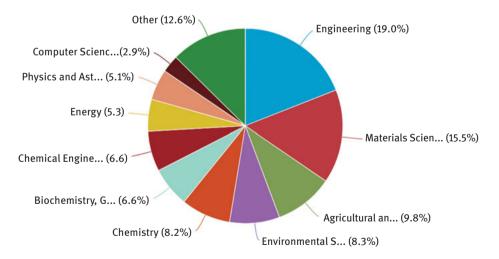


Fig. 4: Document by subject area of published articles on rice straw composite (Source: https:// www.scopus.com).

19.0%. In material science, agricultural and environmental science, articles published account for around 15.5%, 9.8% and 8.3%, respectively.

Figure 5 shows the document by year of published articles in different countries, on plant-based composite. Plant-based composite work has been done by people in China and around 159 articles have been published. In India, around 64 articles on plant-based composite have been published. Other countries like Egypt, the United States, Iran, South Korea and Thailand published 51, 29, 20 and 20 articles, respectively.



Fig. 5: Document by year of published articles in different countries on plant-based composite (Source: https://www.scopus.com).

Figure 6 shows the document by year of published articles in different countries on plant-based composite. Rice straw-based composite work has been done by people in China and around 666 articles have been published. In the United States, around 582 articles have been published on rice straw composite. In India, around 269 articles have been published on rice straw-based composite. Other countries like Germany, France, Spain, Italy and England published 202, 178, 137, 135 and 130 articles, respectively.

2 Composite parts

The main parts of composites are:

- a) Reinforcing material
- b) Matrix material

666 PEOPLES R CHINA	202 germany	130 England	121 BRAZIL	108 australia	96 so	3 IUTH KOREA	
	178	129					
582 USA	FRANCE	JAPAN	83 malaysia			51 RUSSIA	
	137 spain	126 CANADA					
			68 sweden	42 PORTUGAL	41	38 SOUTH AFRICA	
269 INDIA	135 ITALY IRAN	124			NETHE	RL AFRICA	
		IRAN	57 TURKEY	41 MEXICO	37 czech	I REPUBLIC	

Fig. 6: Document by year of published articles in different countries on rice straw composite (Source: https://www.scopus.com).

a) Reinforcing material

Fiber materials are used as reinforcing material to increase the strength of the composite. Natural fiber-based composites are biodegradable composite [1].

b) Matrix material

Matrix materials are a very important part of the composite material. They create proper bonding between the fiber and matrix.

3 Types of fiber

Fibers are classified, as shown in Fig. 7, into two categories – natural fiber and synthetic fiber. Natural fiber is further categorized into animal- and plant-based fiber. Fibers like silk, wool and hair are animal fibers. Plant fiber is further classified as leaf-, bast-, seed-, fruit-, wool-, stalk- and grass-based fiber [2]. Fibers like banana, abaca and sisal are leaf fibers. Fibers like flex, jute, okra and hemp are bast fibers. Fibers like cotton, kapok and cotton are seed fibers.

4 Types of composite materials

The types of composite materials are as follows:

- a) Fiber-reinforced composites
- b) Continuous or long fiber composite

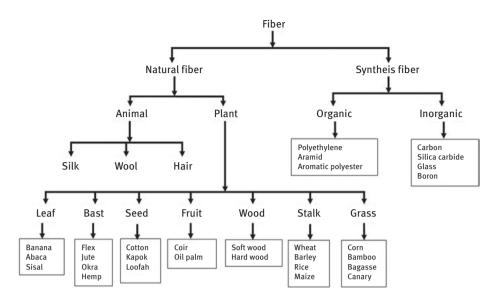


Fig. 7: Classification of fibers.

- c) Discontinuous or short fiber composite
- d) Laminate Composites
- e) Particulate Composite
- f) Flake composites

a) Fiber-reinforced composites

Fiber-reinforced composite made from fibers and resin. In industrial appliance, fiber-reinforced composites have very much used.

b) Continuous or long fiber composite

Continuous or long fiber composite is made from long fibers and resin. The long fibers are comparatively stronger than other fibers [3]. The long fibers are further classified into two:

- i) Unidirectional fiber composite
- ii) Bidirectional fiber composite

i) Unidirectional fiber composite

Unidirectional fiber composites have orientation of fibers in only one direction.

ii) Bidirectional fiber composite

Bidirectional fiber composites have orientation of fibers in two directions. The fibers are at right angles to each other or make some angle with each other.

c) Discontinuous or short fiber composite

Discontinuous or short fiber composite is made from small fibers and resin. The small fiber is mixed with resin properly to make very good composites [4, 5]. These composites further classified into two kinds:

- i) Biased or preferred oriented fiber composite
- ii) Random oriented fiber composite.

i) Biased or preferred oriented fiber composite

In based or preferred oriented fiber composite, the fibers are properly orientated.

ii) Random oriented fiber composite

In random oriented fiber composite, the fibers are randomly orientated.

d) Laminated composites

Laminated composites are made from layers of fibers one after another mixed with resin. Laminated composites are further classified as follows:

- i) Unidirectional
- ii) Angle-ply
- iii) Cross-ply
- iv) Symmetric laminates
- v) Hybrid laminates

e) Particulate composites

Particulate composites made from ceramics, metals and metals matrix materials [4, 6].

f) Flake composites

Flake composites are made of flake and resin [7, 8].

5 Chemical composition of natural fibers

The main chemical constituents of natural fibers are as follows:

- a) Cellulose
- b) Hemicellulose
- c) Lignin

a) Cellulose

Cellulose gives high mechanical strength to the fiber [9, 10]. If cellulose percent in fibers is high, fibers have good mechanical strength.

b) Hemicellulose

Hemicellulose is formed by bacteria and found in grains, fruit and plant steam fibers.

c) Lignin

Lignin creates a weak bond between the fibers and resin materials. Hence, chemical treatment of fibers is essential for fabrication of composite materials.

Rice straw is a natural fiber – plant-based stalk fiber. Table 1 [11] shows the chemical constituents of different types of rice straw.

Rice straw	Husk	Cellulose (%)	35-45
		Hemicellulose (%)	19–25
		Lignin (%)	20
		Residual ash (%)	14-17
	Whole straw	Cellulose (%)	41-57
		Hemicellulose (%)	33
		Lignin (%)	8-19
		Residual ash (%)	8-38
	Leaf	Cellulose (%)	37-41
		Hemicellulose (%)	22-25
		Lignin (%)	7-8
		Residual ash (%)	26-33
	Stem	Cellulose (%)	24-46
		Hemicellulose (%)	24–28
		Lignin (%)	4-6
		Residual ash (%)	8-16

Table 1: The ranges of the chemical constituents in rice straw.

6 Composites manufacturing processes

The composite manufacturing process is of two types:

- a) Open molding method
- b) Closed molding method

a) Open molding method

In open molding method, fibers and resin are mixed together manually [12, 13]. Open molding method is further classified as follows:

i) Hand Lay-up

- ii) Spray-up
- iii) Filament winding
- b) Closed molding method

In closed molding method, fibers and resin are mixed together in a closed chamber [14, 15]. The composite material is prepared with the mold.

Closed molding method is further classified as follows:

- i) Compression molding
- ii) Pultrusion
- iii) Vacuum bag molding
- iv) Vacuum infusion processing
- v) Resin transfer molding (RTM)

7 Experimental procedure

7.1 Preparation of test samples

The test samples are prepared with the help of following materials:

- a) Rice straw
- b) Epoxy resin
- c) Hardener
- d) Hot water
- e) NaOH solution

a) Rice straw

Rice straw is a plant-based natural fiber – biodegradable fiber. The fiber is collected from local area as shown in Fig. 8.



Fig. 8: Rice straw.

b) Epoxy resin

Epoxy resin R101 has been used for sample preparation.

c) Hardener Hardener H101 has been used for sample preparation.

d) Hot water Hot water was used for treatment of rice straw fiber.

e) NaOH solution NaOH solution was used for treatment of rice straw fiber.

After treatment of rice straw with hot water and 2% NaOH solution, the fiber is cleaned with fresh water. Cleaned fiber is then dried in sunlight [16, 17]. Treatment of fiber is essential for proper bonding between the fiber and resin. The samples with single-layer, double-layer, triple-layer biaxial untreated, treated with hot water and treated with NaOH solution rice straw fiber with weighted 10%, 20%, 30%,40% and 50% mixed with epoxy resin are as shown in Fig. 9.



Fig. 9: Sample preparation.

The sample has been prepared as per ASTMD3039 standard as shown in Fig. 10.





8 Mechanical testing

All mechanical tests – tensile, flexural test, flexural modulus, Young's modulus, impact strength and impact strength test were conducted on the machine. All samples of single, double and triple layers were tested.

9 Result and discussion

9.1 Tensile test

Figure 11 shows the result of the comparative study of tensile strength of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fraction [18]. The tensile strength is maximum at 30% fiber volume treated with 2% NaOH solution composite. When the fiber volume increases, a weak bond is created between the filler material and resin. This weak bonding gives less strength to the composite. When the fiber volume is between 31% and 50%, the tensile strength of the composite decreases.

9.2 Flexural test

Figure 12 shows the result of the comparative study of flexural strength of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different

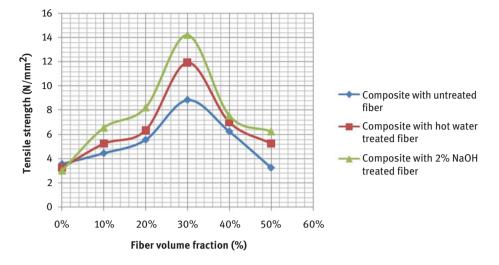


Fig. 11: Comparative study of tensile strength of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

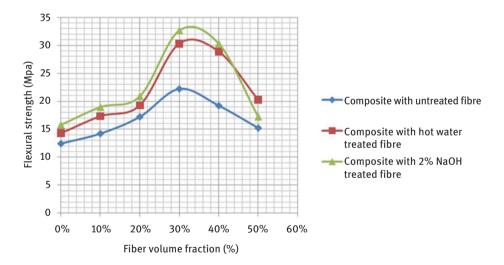


Fig. 12: Comparative study of flexural strength of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

fiber volume fractions. The flexural strength is maximum at 33% fiber volume treated with 2% NaOH solution composite. When the fiber volume increases, a weak bond is created between the filler material and resin. This weak bonding gives less flexural strength to the composite. When the fiber volume is between 34% and 50%, the flexural strength of the composite decreases.

9.3 Flexural modulus test

Figure 13 shows the result of the comparative study of flexural modulus of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The flexural modulus is maximum at 30% fiber volume treated with 2% NaOH solution composite. When the fiber volume increases, a weak bond is created between the filler material and resin. This weak bonding gives less flexural modulus to the composite. When the fiber volume is between 32% and 50%, the flexural strength of the composite decreases.

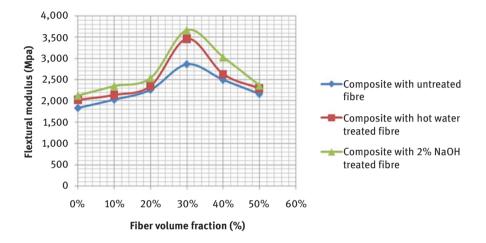


Fig. 13: Comparative study of flexural modulus of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

9.4 Young's modulus test

Figure 14 shows the result of the comparative study of Young's modulus of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The Young's modulus is maximum at 30% fiber volume treated with 2% NaOH solution composite. When the fiber volume increases, a weak bond is created between the filler material and resin. This weak bonding gives less Young's modulus value to the composite. When the fiber volume is between 32% and 50%, the Young's modulus of the composite decreases.

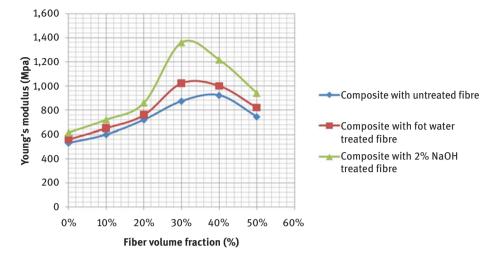


Fig. 14: Comparative study of Young's modulus of composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

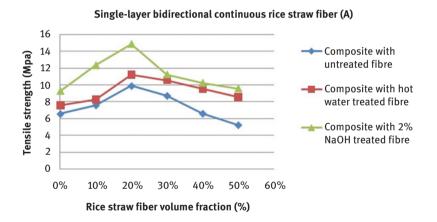


Fig. 15: Comparative study of tensile strength of single-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

9.5 Tensile strength

Figure 15 shows the result of the comparative study of tensile strength of singlelayer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The tensile strength is maximum at 20% fiber volume treated with 2% NaOH solution composite. When the fiber volume and layers of fiber increase, a weak bond is created between the filler material and resin. This weak bonding gives less strength to the composite. When the fiber volume is in between 21% and 50%, the tensile strength of the composite decreases.

Figure 16 shows the result of the comparative study of tensile strength of double-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The tensile strength is maximum at 20% fiber volume treated with 2% NaOH solution composite. When the fiber volume and layers of fiber increase, a weak bond is created between the filler material and resin. This weak bonding gives less strength to the composite. When the fiber volume is between 21% and 50%, the tensile strength of the composite decreases.

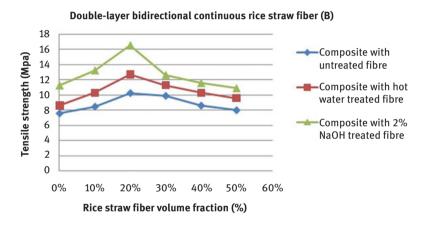


Fig. 16: Comparative study of tensile strength of double-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

Figure 17 shows the result of the comparative study of tensile strength of triplelayer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The tensile strength is maximum at 20% fiber volume treated with 2% NaOH solution composite. When the fiber volume and layers of fiber increase, a weak bond is created between the filler material and resin. This weak bonding gives less strength to the composite. When the fiber volume is in between 21% and 50%, the tensile strength of the composite decreases.

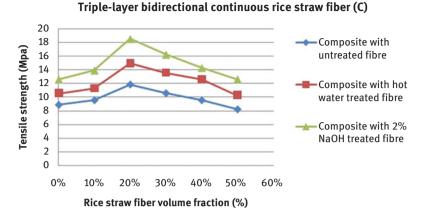


Fig. 17: Comparative study of tensile strength of triple-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

9.6 Impact strength test

Figure 18 shows the result of the comparative study of impact strength of singlelayer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The impact strength is maximum at 20% fiber volume treated with 2% NaOH solution composite. When the fiber volume and layers of fiber increase, a weak bond is created between the filler material and resin. This weak bonding gives less strength to

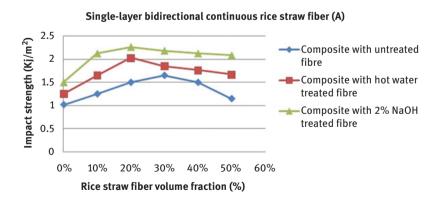
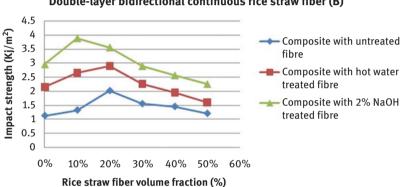


Fig. 18: Comparative study of impact strength of single-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

the composite. When the fiber volume is in between 21% and 50%, the impact strength of the composite decreases.

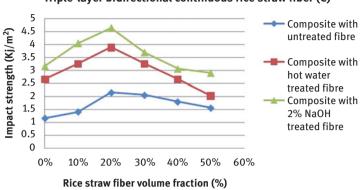
Figure 19 shows the result of the comparative study of impact strength of single-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The impact strength is maximum at 10% fiber volume treated with 2% NaOH solution composite. When the fiber volume and layers of fiber increase, a weak bond is created between the filler material and resin. This weak bonding gives less strength to the composite. When the fiber volume is in between 11% and 50%, the impact strength of the composite decreases.



Double-layer bidirectional continuous rice straw fiber (B)

Fig. 19: Comparative study of impact strength of double-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

Figure 20 shows the result of the comparative study of impact strength of triplelayer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions. The impact strength is maximum at 10% fiber volume treated with 2% NaOH solution composite. When the fiber volume and layers of fiber increase, a weak bond is created between the filler material and resin. This weak bonding gives less strength to the composite. When the fiber volume is in between 11% and 50%, the impact strength of the composite decreases. [19]



Triple-layer bidirectional continuous rice straw fiber (C)

Fig. 20: Comparative study of impact strength of triple-layer bidirectional continuous rice straw fiber composite with untreated fiber, hot water treated fiber and 2% NaOH treated with different fiber volume fractions.

10 Conclusion

The following conclusions can be drawn from test result:

- Rice straw fiber volume fraction 30% with epoxy resin treated with NaOH solution has more tensile strength than the other composites.
- Rice straw fiber volume fraction 30% with epoxy resin treated with NaOH solution has more flexural strength than the other composites.
- Rice straw fiber volume fraction 30% with epoxy resin treated with NaOH solution has more flexural modulus than the other composites.
- Young's modulus of rice straw fiber volume fraction 30% with epoxy resin treated with NaOH solution is more than the other composites.
- Rice straw fiber volume fraction 20% of single-layer bidirectional continuous rice straw fiber with epoxy resin treated with NaOH solution has more tensile strength than the other composites.
- Rice straw fiber volume fraction 20% of double-layer bidirectional continuous rice straw fiber with epoxy resin treated with NaOH solution has more tensile strength than the other composites.
- Rice straw fiber volume fraction 20% of triple-layer bidirectional continuous rice straw fiber with epoxy resin treated with NaOH solution has more tensile strength than the other composites.
- Rice straw fiber volume fraction 20% of single-layer bidirectional continuous rice straw fiber with epoxy resin treated with NaOH solution has more impact strength than the other composites.

- Rice straw fiber volume fraction 10% of double-layer bidirectional continuous rice straw fiber with epoxy resin treated with NaOH solution has more impact strength than the other composites.
- Rice straw fiber volume fraction 20% of triple-layer bidirectional continuous rice straw fiber with epoxy resin treated with NaOH solution has more impact strength than the other composites.

10.1 Future scope

The following are research recommendations:

- Work can be done on hybrid composites like rice husk and rice straw.
- Chopped rice straw with epoxy resin composite can be made.
- Rice straw can be treated with other chemicals and mixed with epoxy resin.
- Composites can be made with some other fiber volume fraction.
- Moisture, microscopic and thermal degradation tests can be done.
- Composite wear behavior can be tested.
- Composite corrosion behavior can be tested and compared with other composites.

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Part III: Animal-based composite

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Thermomechanical properties of forcespun polycaprolactone fibers infused with fish scale-based hydroxyapatite

Abstract: Biomaterials synthesized from natural resources have attracted plentitude of attention over the past few decades. Hydroxyapatite (HA) is one such biomaterial with excellent characteristics suitable for biomedical applications. In this study, HA is synthesized from carpa fish scales by calcination method. The HA particles were nanomilled for particle size reduction and these particles were characterized using X-ray diffraction, scanning electron microscopy and transmission electron microscopy. A novel forcespinning technique was used to fabricate microfibers from polycaprolactone (PCL) infused with synthesized HA. Thermomechanical properties of the PCL/HA fiber mats were investigated using differential scanning calorimetry, dynamic mechanical analysis and tensile tests. The analyses suggest that infusing HA in moderate quantities enhances the thermomechanical properties of composite fibers.

Keywords: bio-based hydroxyapatite, fish scales, polycaprolactone, scaffolds, forcespinning, mechanical properties

1 Introduction

Waste materials from animal sources can serve as valuable resources for the extraction and production of hydroxyapatite (HA)-based biocomposites [1]. In the past few years, the development of biocomposites from sustainable resources has become the point of interest for various researchers. This interest is fueled by the demand to develop more sustainable alternatives to traditional materials. HA is an important biomaterial that can be synthesized from various animal sources such as seashells, eggshells and fish scales and used within biocomposites. Massive amount of waste shells from biogenic sources are being disposed of after use, which directly leads to land and air pollution. China alone produces around 10 million tons of waste sea

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shells annually that are discarded in landfills [2]. France generates huge amounts (160,000 tons) of shells from shellfish and 45,000 tons from fisheries annually [3]. Disposed shells are detrimental as the shell waste results in contamination of land and air due to release of intensive odors during the decay process along with disintegration of heavy metals from waste [4]. In Africa, tons of waste shells have been disposed of and left to pile on shores. Such piles of waste shells become an impending source for disease-carrying organisms, which leads to health hazards [2, 4].

One of the substantial challenges of the fishing industry is the skyrocketing consumption. Globally, more than 27% of fish captured is unused. About 18–30 million tons of undesirable fish waste is being disposed of [5, 6]. The enormous amounts of fish by-product waste create detrimental effect on the environment. Fish scales contribute to approximately 8% of the fish waste [6]. Treatment of discarded fish scales to obtain HA serve as a sustainable route to reuse these waste materials [7]. Waste produced from animal sources, that is, trash converted into valuable biomaterials [1, 8–10] would serve as a great engineering and scientific breakthrough that can help create a more economically and environmentally stable planet.

1.1 Overview of hydroxyapatite

The Greek word "apatos," which means "to deceive," is the root word for "apatites," as, in the past, the term "apatite" is used to distinguish the mineral apatites that are frequently confused with priceless gemstones such as topaz [11]. Apatites are commonly expressed as $M_{10}(XO_4)_6(Z)_2$, where M^{2+} corresponds to a metal and XO_4^{3-} and Z^{-} are anions. HA, also known as hydroxylapatite, is a calcium phosphate mineral, where metal M^{2+} becomes Ca^{2+} , XO_4^{3-} and Z^- is PO_4^{3-} and OH^- (hydroxyl group), and is usually represented as [Ca₁₀(PO₄)₆(OH)₂] [12, 13]. HA is one of the major constituents of the minerals of the calcified tissues which makes up to 60–70% mass of the total bone and 98% of the mass of the enamel [14]. In general, the stoichiometric HA has Ca/P molar ratio of 1.67 and has a chemical composition similar to the mineral phase of the bone [1, 14]. Additionally, due to biocompatible, osteoconductive and bioactive nature, synthetic HA has been extensively used in biomedical applications, especially in orthopedic and dental applications for repairs and regenerations [13–15]. However, the molar ratio of hydroxyl-rich and carbonate-deficient mineral form of apatite found in mammals is less than 1.67 [16]. This is caused by the presence of various impurity ions in HA lattice such as Na⁺, Zn²⁺, CO₃²⁻, F⁻, Si²⁺, Mg²⁺ and K^+ [17]. Among these impurities, the carbonate ion (CO₃²⁻) is the most abundantly found impurity with 3–8 wt% [18, 19]. These trace elements have proved to be effective in accelerating the bone formation process [1, 17]. These ions, if tailored and regulated effectively as therapeutic ions, can enhance the biological properties of HA, making them a resourceful platform for various biomedical applications [14]. In view of these advantages, synthesis of HA from natural sources has gained lot of interest in the past few decades. Natural HA can be synthesized from various biological sources such as animal sources, shell sources and plant and algae sources. Animal sources include horse, bovine, cow, camel, fish bones and fish scales. Shell sources include clam, eggshell, seashell and oysters.. In this chapter, HA from animal-based sources was discussed.

1.2 Animal sources of hydroxyapatite

Among the sources available for HA, animal sources including marine and mammal sources became major source of HA. HA that is derived from these sources is nonstoichiometric and bioactive, and contains trace impurities that help in bone regeneration [12]. A summary of the sources and the processes used to synthesize natural HA is shown in Fig. 1. HA is commonly extracted from the bovine bone (cow, buffalo, etc.) in mammals [20–24]. The bones of other mammals such as camel [25], horse [26], deer [27], goat [28, 29], sheep [30], crocodile [31], chicken [32, 33] and pig [34] are also used to extract HA [1]. The bones acquired from these animal sources are carefully selected and cleaned to remove the visible impurities. The collected bones are simmered and rinsed in any suitable solution to remove blood, tissues and other impurities. The bones thus obtained are subjected to chemical treatment to remove fats, proteins and other organic material. The bones are cut, crushed into tinier pieces and are subjected

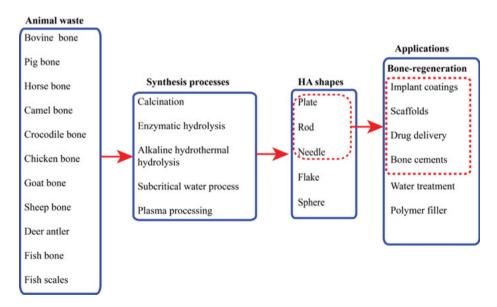


Fig. 1: Schematic representation of synthesizing HA from animal sources for various applications.

to calcination or alkaline treatment to obtain HA [12]. The size of the particles is further reduced by other mechanical methods such as ball milling. The hydroxyapatite thus synthesized might have different microscopic forms and properties, depending on the bone type, calcination temperature and synthesis procedure. Some of the microscopic forms include plate-like structures, rods, needles and spheres. In addition to the mammal bones, crustacean exoskeletons [35, 36], fish bones [37–39] and scales [33, 40] are used to synthesize HA. The biowaste obtained from these marine sources is cleaned, dried and then subjected to hydrothermal treatments to synthesize HA.

1.3 Synthesis process of hydroxyapatite

From natural sources, HA can be obtained by various synthetic techniques that are economical, effective and environmentally preferable. The process parameters are controlled carefully to synthesize HA from animal sources such that phase purity, properties, efficiency and the size are attained with desirable characteristics [17]. In general, bones from animal sources are subjected to pretreatment of the bone which includes boiling and washing with solvents to remove impurities prior the extraction of HA [1]. Some of the prominent synthetic methods are as follows:

- Heat treatment: Heat treatment or calcination method is the most prevalent process for extracting HA. This is a simple thermal decomposition method where the bones or the fish scales are calcinated in a furnace under different ambient conditions at different temperatures [17, 23]. The temperatures may vary from 600 to 1,400 °C [1]. This process removes the organic matter and leaves the HA residue. The calcination temperature influences the crystallinity of the HA and also the other calcium phosphate phases. Hence, a suitable temperature is to be selected for the calcination process such that HA with the desired characteristics can be attained while getting rid of the pathogens [41].
- Enzymatic hydrolysis: HA from fish scales is isolated in an enzymatic hydrolysis process. The enzymatic hydrolysis process has a greater extraction rate in a short time without racemization effect. In this process, enzymes such as protease N and flavourzymes are used to hydrolyze the organic component with optimal temperatures and pH conditions [42, 43]. Once the organic component and the collagen degrades, the resulting hydrolysates are treated with boiling water to deactivate the enzymes by treating the hydrolysates with boiling water [1]. The resulting HA is collected by centrifuging the mixture. Although this process is environmentally friendly, the cost involved is high.
- Alkaline hydrothermal hydrolysis: This is one of the best approaches to obtain nanostructured arrangement of HA from natural sources. In this method, the bones or scales are mixed with chemicals in aqueous solution at required temperatures and pressures in an autoclave or a pressure vessel [23, 44].

- Subcritical water processing: Water is treated at subcritical conditions to extract HA. Usually, temperatures above 100 °C and less than 375 °C (critical temperature) under desired pressures are employed to obtain HA [23].
- Plasma processing: This technique employs plasma arc treatment to melt the bones of animal sources such that the organic matter is completely removed, leaving the residue of HA. The power level, flow rate and melting time are to be controlled to obtain the desirable characteristics of HA [45].

In addition to these processes, various methods like vibromilling, ball milling, microwave heating and ultrasonication are used to reduce the size of the synthesized HA particles [1, 9, 10, 12, 44].

1.4 Biomedical applications of fish-based HA composites

HA that is synthesized from fish is widely used for adsorption [6, 37, 46] and biomedical [47] applications. Specifically, HA is significantly used in bone tissue engineering applications by producing effectual scaffolds, which promotes cell adhesion and proliferation. Polyethylene glycol–HA-based scaffolds developed from solvent casting have shown desirable mechanical performance with interconnected porous structures that are suitable for bone regeneration [48]. The PMMA-HA scaffolds were able to meet the physiological requirements for bone generation. In addition to this, the in vitro studies of these scaffolds confirmed that apatite, similar to bone, was formed on the scaffold surface [49]. Peptide-based hybrid scaffolds for periodontal applications were developed from salmon and red snapper fish scale-based HA that showed promising properties [50]. The cell proliferation along with calcium deposition improved when 3D-printed polycaprolactone (PCL) scaffolds were coated with extracts from fish bone [51]. Fish bone powder-based polyvinylidene fluoride/HA/ β -Tricalcium phosphate composites that were developed have shown elated bioactivity when immersed in a simulated body fluid for 7 days [52]. Fish scale-based mineral-ion-loaded HA composite scaffolds have shown improved cell adhesion and alkaline phosphate activity suitable for tissue engineering applications [53]. Nanofibrous fish scale-reinforced PHBV composite scaffolds were fabricated from wet electrospinning and freeze drying technique. These composite scaffolds have shown elevated biological and mechanical properties [54]. Similarly, fish scale-infused chitosan biocomposites developed from freeze drying have also shown enhanced cell proliferation [55]. The corrosion performance and biomineralization of magnesiumbased implants improved when infused with fish scale-derived HA [56, 57].

Despite the advancements in the use of HA synthesized from fish sources, demand persists for feasible techniques to develop fish scale-based HA composites. Research efforts are yet anticipated to explore thermal, mechanical and biological properties of HA-based composites that make them viable for biomedical applications. In this chapter, we present the thermal and mechanical properties of fish scale-based HA-infused forcespun PCL fibers that are suitable for biomedical applications. Forcespinning technique, which uses centrifugal force, has been successfully used to generate various polymer fibers [58–60]. PCL has become a noteworthy source for biomedical applications due to its remarkable mechanical and biodegradable properties [61, 62]. On that account, understanding the influence of HA particles on thermomechanical properties of PCL composite fibers can highly contribute to the field of biomedical applications.

2 Materials and methods

2.1 Materials

In order to develop forcespun polymer composite nanofibers, powdered PCL (molecular weight of 50kDa) was considered. This was purchased from Polysciences, Inc. (Warrington, PA, USA). Chloroform (ACS reagent \geq 99.8%) was used as a solvent and this was obtained from Sigma-Aldrich (St. Louis, MO, USA).

Sundried raw fish scales belonging to the carpa family were acquired from Nizona Inc., India. These raw scales have 20–30% moisture content. The fish scales were calcinated at 800 °C for 3 h in a furnace. The obtained calcined scales were crushed using mortar and were nanomilled for 2 h at 2,000 rpm. The nanomilling process was performed using MTI compact nanoagitator bead mill, which consists of 0.3 mm zirconia balls that are used for milling. Distilled water is used as a solvent for the nanomilling process. The nanomilled powder was then collected and centrifuged to separate the HA nanoparticles. The collected HA particles were dried and labeled for use.

2.2 Forcespinning of fibers

PCL (16 wt%) was dissolved in chloroform to obtain PCL solution. The PCL solution was then mixed using a magnetic stirrer for 3 h at 170 rpm. To prevent the evaporation of solvent, the vials were sealed during the mixing process. The HA nanoparticles were then infused into the PCL solution by 1, 2 and 3 wt%. The solution mixture was then mixed in a Thinky mixer (ARE-250), which is a planetary noncontact mixer, for 7 min at 1,900 rpm.

The nonwoven fibrous mats were obtained from the polymer solution mixture using forcespinning Cyclone L-1000M apparatus from Fiberio. Using a pipette, a 2 mL of the PCL/HA solution was infused into the needle-based spinneret with 24 ga × 1/2" stainless steel regular bevel needles. The spinneret was allowed to spin at 7,000 rpm for 10 min. The fibers were collected in a collector with equally spaced

vertical plates positioned at 115 mm from the needle (190 mm from the center of the spinneret) as shown in Fig. 2(a). The collected fibers (Fig. 2(b)) were stored under desiccation prior to the characterization.

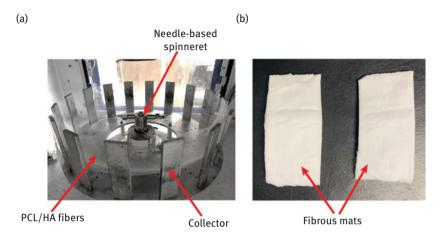


Fig. 2: (a) PCL/HA fiber web and (b) forcespun fiber mats collected from the web.

2.3 Characterization of nano-HA and PCL/HA fibers

2.3.1 X-ray diffraction

X-ray diffraction (XRD) studies were performed to analyze the raw and calcined fish scales. A Rigaku diffractometer (DMAX2100) that is equipped with Cu-K α radiation was used for the analysis. This was operated at a scan rate of 1°/min, with a step size of 0.02°, varying from 3° to 80° Braggs angle of diffraction at 40 kV, 30 mA and 1.2 kW.

2.3.2 Scanning electron microscopy

The morphology of the synthesized HA nanoparticles and PCL/HA fibers was analyzed using JEOL JSM-7,200 F field-emission scanning electron microscope (FESEM, JEOL USA, Peabody, MA) at 2 kV. Energy-dispersive X-ray analyzer (EX-37001) facilitated with scanning electron microscopy (SEM) was used to perform energy-dispersive spectroscopy (EDS) analysis of the synthesized HA nanoparticles. The samples were subjected to sputter coating using gold/palladium (Au/Pd) for 3 min at 10 mA using hummer sputter coater. 134 — Deepa Kodali et al.

2.3.3 Transmission electron microscopy

The particle size, shape, distribution and crystallinity of the nanoparticles were analyzed by using a JOEL 2010 transmission electron microscope. Nanoparticles were first dispersed in ethanol, then dispensed on Cu grid and air dried. This copper grid was further used for transmission electron microscopic (TEM) analysis at an operating voltage of 200 kV.

2.3.4 Differential scanning calorimetry

A TA-Q series 2000 differential scanning calorimeter was used to analyze the thermal properties of the fibers. The samples weighing approximately 10–12 mg were sealed in hermetic pans and were tested against an empty reference pan. The samples were heated at a rate of 5 °C/min from –80 °C to 80 °C. Subsequently, they were cooled to –80 °C and again heated up to 80 °C to obtain a heat–cool–heat cycle.

2.3.5 Tensile testing

The mechanical properties of the nonwoven fibrous mats with 5 mm width and 20 mm length following ASTM D882-10 standard [63] were investigated using uniaxial tensile tests. The test window frame with fiber mat is shown in Fig. 3(a). The thickness was measured with a micrometer (Mitutoyo 293-340-30 digital micrometer) having

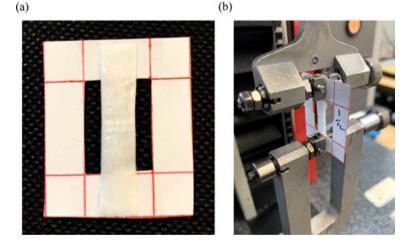


Fig. 3: (a) Tensile test sample template with fibrous mat and (b) tensile test setup with the template.

0.001 mm resolution. To obtain statistically reliable results, the average thickness of the fibrous mats was obtained by taking measurements at 10 different places.

A Zwick/Roell Z2.5 universal mechanical testing machine was used to perform the tensile test. The test window frame was loaded between the grips and the frame portion was cut as shown in Fig. 3(b). The tensile tests were carried out with a constant crosshead speed of 5 mm/min under displacement control mode with a preload of 0.01 N and a 20 N load cell. The tensile properties, including tensile modulus, elongation at break, tensile strength and elongation at maximum stress, were evaluated from tensile tests.

2.3.6 Dynamic mechanical analysis

The fibrous mats with sizes of 40 mm × 5 mm were considered for dynamic mechanical analysis tests. The thickness of the mats was measured similar to that of tensile tests. A TA-Q series 800 dynamic mechanical analyzer was used to understand the viscoelastic behavior of the fibrous mats. The tensile mode under multi frequency strain mode (frequency sweep method) with a static preload of 0.01 N was considered to perform the test. The specimen was held isothermally at 30 °C, oscillating at a constant strain of 1% over a frequency range of 1–10 Hz.

3 Results

3.1 Characterization of HA nanoparticles

The mineral phase of the fish scales generally consists of calcium-deficient HA [64]. The calcination process results in obtaining calcium phosphate and its mineral forms. Figure 4(a) illustrates XRD studies of raw fish scales that contain all the other organic matter such as fats, proteins and collagen. Hence, the peaks regarding the HA are not evident. The calcined fish scales (as shown in Fig. 4(b)) show evident peaks that closely match with standard HA (JCPDS-Pdf # 98-000-0251) and corresponding polycrystalline hexagonal lattice cells. The calcined fish scales were nanomilled for 1 h to reduce the particle size. The HA peaks were more evident in Fig. 4(c) for nanomilled calcined scales. In addition, the peaks became broader suggesting the particle size reduction. The increase in the intensity suggests that the crystal growth was promoted along the *c*-axis [65].

The nanomilled particles are, in general, irregular in size and shape. SEM and TEM micrographs were observed to analyze the size, shape and distribution of the nanomilled particles as shown in Fig. 5. The SEM micrographs show small chunks of HA particles (Fig. 5(a)). The shape of the HA nanoparticles was slightly irregular

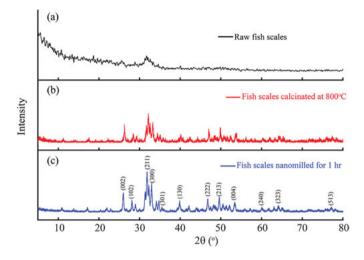


Fig. 4: X-ray diffraction patterns of (a) raw carpa fish scales, (b) fish scales calcined at 800 °C for 3 h and (c) calcined fish scales after nanomilling.

and these particles were agglomerated (Fig. 5(b) and (e)). The size of the particles was reduced to a nanorange, which was evident from both the SEM and TEM micrographs (Fig. 5(c) and (f)). The size of the synthesized HA particles was within the range of 50–80 nm. The crystallinity and distribution of HA particles were analyzed

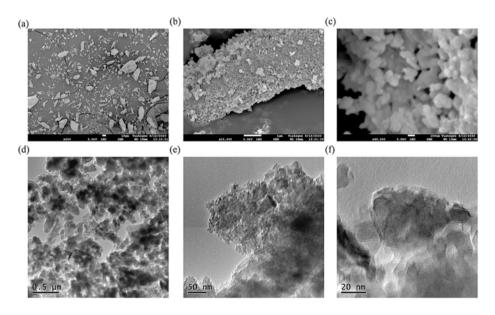


Fig. 5: (a,b,c) SEM micrographs of the nanomilled HA particles at various magnifications and (d,e,f) TEM micrographs of nanomilled HA particles at various magnification factors.

from TEM micrographs (Fig. 5(d), (e) and (f)). The synthesized HA has plate-like structures as shown in Fig. 5(f).

The EDS spectra of the nanomilled HA particles were shown in Fig. 6. From the EDS analysis, the Ca/P ratio was determined to be 1.31, which is less than the molar ratio of stoichiometric HA (1.67). This suggests that the HA that is obtained from the carpa fish scales is calcium deficient. The plate-like structures of the synthesized HA particles together with their Ca/P ratio make them suitable for biomedical applications.

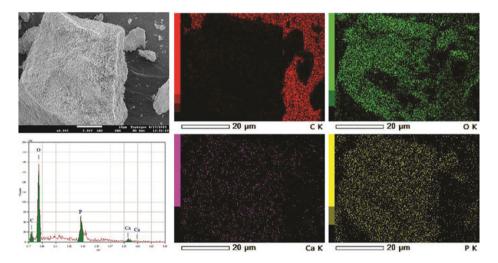


Fig. 6: EDS spectra of the nanomilled HA particles with carbon, oxygen, calcium and phosphorus distributions.

3.2 Characterization of PCL fibers

3.2.1 Morphology

The SEM micrographs of the forcespun PCL/HA fibers obtained at 7,000 rpm are shown in Fig. 7. The neat PCL fibers with no HA are shown in Fig. 7(a). Homogenous fibers were obtained forming a three-dimensional mesh with random orientation. The diameter of the fibers was estimated using Gaussian distribution (see Table 1). With the infusion of HA, the fiber diameter increased. The fibers with 1 wt% of HA were uniform and the diameter of the fibers increased compared to that of the neat fibers. The fibers were still uniform with 2 wt% of HA and there is slight increment in the diameter compared to that of 1 wt%. The fibers with 1 and 2 wt% of HA suggest an effective dispersion of nanoparticles in the polymer solution. However, as the infused amount of HA increases to 3 wt%, the fibers became nonuniform. Bulged structures

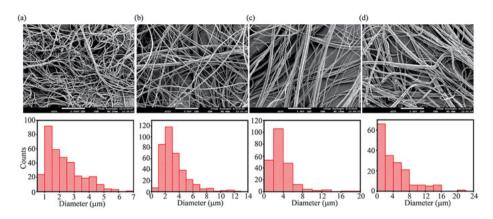


Fig. 7: SEM micrographs of PCL/HA fibers with corresponding histograms showing fiber diameter distribution for concentration of 16 wt% at rotational speed of 7,000 rpm with (a) PCL neat, (b) 1 wt % HA, (c) 2 wt% HA and (d) 3 wt% HA.

Table 1: Mean fiber diameter of the forcespun

 PCL/HA fibers for various weight percentages.

HA wt%	Mean diameter (µm)		
PCL neat	1.65 ± 0.174		
(0 wt%)			
1 wt%	2.52 ± 0.098		
2 wt%	2.95 ± 0.048		
3 wt%	4.12 ± 3.42		

were observed in the middle of the fibers due to the agglomeration of the particles. There is a sharp increase in the average fiber diameter for 3 wt% due to the nonuniformity of the fibers.

3.3 Thermal analysis

The thermal behavior and properties of PCL/HA fibers were analyzed from thermographs obtained from the differential scanning calorimetry (DSC) analysis as shown in Fig. 8. The glass transition temperature (T_g) is estimated from the primary heating curve. The melting temperature (T_m), heating enthalpy (ΔH_m) and crystallinity were evaluated from the succeeding heating cycle of the DSC endotherms and the results are tabulated in Table 2. The glass transition temperature increased significantly with infusion of HA nanoparticles (Fig. 8(a)). As the amount of filler increased, T_g also increased suggesting that the filler materials successfully restricted

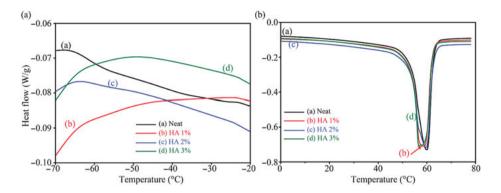


Fig. 8: (a) DSC thermographs showing glass transition temperature and (b) melting endotherms for PCL fibers with various HA weight percentages.

HA wt%	Glass transition temperature <i>T</i> g (°C)	Heating		% Crystallinity (χ _c)
		7 _m (°C)	ΔH _m (J/g)	
PCL neat(0 wt%)	-59.71	59.81	53.22	38.15
1 wt%	-54.88	58.35	65.09	46.66
2 wt%	-48.31	59.5	88.83	63.68
3 wt%	-35.4	56.72	63.35	45.41

Table 2: DSC analysis for the PCL fibers obtained at various rotational speeds.

the mobility of the polymer chains, thus increasing the stiffness of the chains. As ceramics are thermally stable, infusing them as fillers into the polymer has improved the thermal behavior of composite fibers.

The peak melting points of fibers do not show any significant shift with the weight percentage of HA nanoparticles (Fig. 8(b)). The melting point slightly decreased compared to that of neat PCL. The melting enthalpy increased for the HA-infused fibers compared to that of neat fibers. However, as the amount of HA reaches 3 wt%, the enthalpy decreases.

The crystallinity was calculated from the following equation:

$$\chi_{\rm c} = \frac{\Delta H_{\rm m}}{\Delta H_{\rm f}} \times 100$$

where χ_c is crystallinity and ΔH_f is the fusion enthalpy of 100% crystalline sample (139.5 J/g for PCL). The crystallinity of the fibers is shown in Table 2. The crystallinity of fibers increased with the addition of HA nanoparticles. The PCL/HA fibers with 3 wt% of HA exhibited a downtrend in crystallinity. The reduction in crystallinity

might be due to the development of intermolecular hydrogen bonds between PCL and HA nanoparticles, which might have impeded the chain flexibility [66, 67].

3.4 Mechanical analysis

The mechanical properties of the PCL/HA fibers were analyzed by tensile and dynamic mechanical analysis. An average of four specimens for each weight percentage was considered for tensile test, and the results of which are shown in Fig. 9(a). The summary of these results are tabulated in Table 3. The tensile modulus of the fiber mats increased with the infusion of HA compared to that of neat fibers. This complies with the finding that the mechanical properties of PCL composites can be enhanced with the blending of composites [68]. The Young's modulus varied from 8.87 to 11.17 MPa for PCL/HA fibers, which approximates with the findings of Heo et al. [51]. The Young's modulus for 2 wt% HA was observed to be around 2.5 times the Young's modulus of neat fibers. The tensile strength and elongation at break also increased with the increase in the amount of HA particles and are in accordance with the results presented in the previous studies [69, 70]. However, for 3 wt % of HA, the tensile strength is slightly less than that of neat fibers. A similar trend

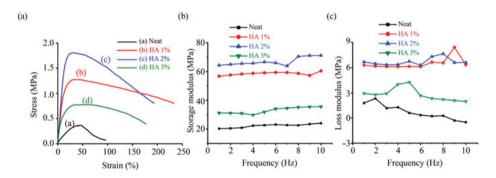


Fig. 9: (a) Averaged stress-strain curves, (b) storage modulus and (c) loss modulus of PCL/HA fiber mats for various weight percentages of HA.

Table 3: Data summary of the tensile test analysis for forcespun PCL/HA fibers for various weight percentages.

HA (wt%)	Young's modulus (MPa)	Tensile strength (MPa)	Strain at break (%)
PCL neat	4.7 ± 0.81	1.07 ± 0.19	214
1 wt%	9.43 ± 1.48	1.31 ± 0.21	382
2 wt%	11.17 ± 1.83	1.82 ± 0.37	257
3 wt%	8.87 ± 0.95	0.83 ± 0.15	208

was observed for the elongation at break. The enhanced mechanical properties of the PCL/HA fibers can be attributed to an increase in the glass transition temperature and the crystallinity of the fibers. This suggests that temporary intermolecular bonds might have been formed when the nanoparticles were under tensile loading such that they orient and align themselves within the polymer chains, improving the strength of the fibers [71]. It was observed that crystallinity decreases for 3 wt% of HA compared to that of 2 wt%, as shown in Table 2. Due to the decrease in crystallinity with higher amounts of HA, the mechanical properties were also observed to be deteriorated. This is due to the increase in HA particle aggregation that restricts the mobility of HA particles [72]. As such, stress concentration increases at PCL/HA interface and the energy dissipation ability of HA decreases [71]. However, some studies have reported a downtrend of mechanical properties with the addition of HA particles [73, 74]. The disparity in the results might be due to the difference in particle size and dispersion.

The viscoelastic nature of the PCL/HA fiber mats were analyzed using dynamic mechanical analyzer, the results of which are shown in Fig. 9(b) and (c) within the specified frequency range of 1–10 Hz. Both the storage and loss moduli increased when compared with the neat PCL fiber mats. However, similar to the tensile test results, the viscoelastic properties have shown a downturn with the 3 wt% of HA. The overall trend of the storage modulus increased with the rise in frequency. As discussed earlier, the surge in the stiffness of the fibers due to the infusion of HA particles caused the improvement in storage modulus. Although the trend is discontinuous for the loss modulus, overall, there is no significant change with the increase in frequency.

4 Conclusions

The HA particles were synthesized from the biowaste of carpa fish scales and characterized using SEM, TEM and XRD. These results confirmed that the nanomilling process used in synthesis of HA particles effectively reduced the size of the particles to nanoscale. The XRD analysis of calcined and nanomilled fish scales conforms to the HA standard. The PCL/HA fibers were successfully produced using the forcespinning technique that uses centrifugal force to generate the fibers. The influence of the amount of HA filler percentage on thermal and mechanical properties of PCL/HA fibers was analyzed. The infusion of HA particles enhanced the thermomechanical properties of the fibers. The diameters of the fibers increased with the infusion of HA particles. Thermal analysis has shown that crystallinity and glass transition temperature of the fibers increased with the amount of infused HA. However, the thermal properties of the fibers started to decline for 3 wt% of HA due to the increased HA aggregations that have curtailed the mobility of the polymer chains. The tensile and viscoelastic properties have also shown deterioration for 3 wt% of HA. This can be attributed to the undermined energy-dissipating ability of the HA due to agglomeration. The thermomechanical properties of the PCL/HA fibers that were addressed in this study help to understand the suitability of the forcespun fibers for bone tissue engineering applications.

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