

Characterization of natural fibers and their application in bone grafting substitutes

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In the last decades, researchers have developed new materials to improve the quality of human life. Owing to the frequent occurrence of bone fractures, it is important to develop plate materials for the fixation of fractured bones. These plate materials have to be lightweight, compatible with human tissues and ought to allow stiffness. Natural fibers have the advantage that they are renewable resources and have marketing appeal. The Asian markets have been supplying natural fibers for many years, e.g., sisal, banana and roselle are common reinforcement in India. In this research, the fabrication of plate material from powdered natural fibers like sisal (*Agave sisalana*), banana (*Musa sapientum*) and roselle (*Hibiscus sabdariffa*), with bio-epoxy resin Grade 3554A and Hardner 3554B, using moulding method, is described. The present work deals with the prediction of flexural rigidity of the NFRP composite which is compared with that obtained using the ANSYS solution. They are found to be in good agreement. In this work, microstructure is scanned by the scanning electron microscope. The objective of this research was to utilize the advantages offered by renewable resources for the development of biocomposite materials based on biopolymers and natural fibers. In the future, this plate material externally coated with calcium phosphate and hydroxyapatite (hybrid) composite can be used for inside fixation and also external fixation of fractured bones.

Key words: natural fibers, bioepoxy resin, deflection test

1. Introduction

Composites comprise strong load-carrying material (known as reinforcement) embedded in weaker material (known as matrix). Reinforcement provides strength and rigidity, helping to support structural load. The matrix or binder (organic or inorganic) maintains the position and orientation of the reinforcement. Nowadays, the natural fibers such as sisal, banana and roselle have the potential to be used as a replacement for glass or other traditional reinforcement materials in composites. Other advantages of these fibers lie in their low density, high toughness, comparable specific strength, reduction in tool wear, ease of separation, and low energy of fabrication. They have high specific properties such as stiffness, impact resistance and flexibility. In addition, they

are available in large amounts, renewable and biodegradable. Other desirable properties include low cost and low density. Uses of these fibers satisfy both economic and ecological interests. The results showed that the best mechanical properties were observed when roselle and sisal (hybrid) fibers were incorporated. It is well understood now that both the strength and stiffness of fiber composites depend on fiber concentration, fiber aspect ratios, fiber–matrix adhesion, as well as fiber orientation and dispersion in powdered particles. The present contribution reports the utilization of untreated sisal, banana and roselle fibers as reinforcing fillers for bioepoxy resin Grade 3554A and Hardener 3554B as matrix for the first time. The effect of fiber content on mechanical properties such as flexural rigidity and hardness of the composites was investigated and reported.

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Biomaterials improve the quality of life for an ever-increasing number of people each year. The range of their applications is vast and includes such things as joint and limb replacements, artificial arteries and skin, contact lenses, and dentures. This ever-growing demand arises from an ageing population with higher quality of life expectations. The biomaterials' community not only produces new and better implant materials and improves techniques to meet this demand, but it also aids the treatment of younger patients where the necessary properties are even more demanding. A counter force to this technological push is the increasing level of regulation and the threat of litigation. To meet these conflicting needs it is necessary to have reliable methods of characterizing the material and material–host tissue interactions. The main property required for a biomaterial is that it does not produce an adverse reaction when placed into service. Various materials used in biomedical applications may be grouped into (a) metals, (b) ceramics, (c) polymers, and (d) composites made from the above groups. Metals and alloys that are successful as biomaterials include: gold, tantalum, stainless steel, NiTi (shape memory alloy), Co-Cr, and Ti alloys. The machining of such orthopaedic alloy implants, with high speed of machining, will offer advantages, but also has its own disadvantages, i.e., complexity and high machining cost. Titanium was used for bone replacements during past decades, and such implants are simple geometric approximations of the bone shape. Mismatches can occur between real bone and implants, often causing stress concentrations and premature implant failure. The machining stocks are uneven and more than the required levels. This leads to more weight at rough casting stage and contribution of more machining time and these castings are manufactured in green sand moulding process which leads to poor surface finish, more material in machining surfaces and less dimensional stability. The above said complexities open way for the next generation of bone implants, i.e. polymers and ceramics, which have better biocompatibility and good tensile properties.

2. Materials and methods

- An effort to utilize the advantages offered by renewable resources for the development of biocomposite materials based on biopolymers and natural fibers.

- Fabrication of natural fiber (sisal, banana and ro-selle (hybrid) composite fiber) reinforced polymer (NFRP) composite plate material by using bioepoxy resin. Instead of orthopaedic alloys (such as titanium, cobalt chrome, stainless steel and zirconium).

- NFRP composite (biocomposite plate material) can be coated with bone graft substitutes such as calcium phosphate and hydroxyl apatite and this plate material can be used for both inside fixation and external fixation of fractured human bone.

2.1. Natural fibers

Natural fibers present important advantages such as low density, appropriate stiffness and mechanical properties and high disposability and renewability. Moreover, they are recyclable and biodegradable. Over the last decade, the composites of polymers reinforced with natural fibers have received increased attention. Natural fibers such as sisal, flax, jute and wood-fibers possess good reinforcing capability when properly compounded with polymers. One of the unique aspects of designing the parts with fiber-reinforced composite materials is that the mechanical properties of the material can be tailored to fit a certain application. By changing the orientation or placement of the fibers the material can be designed to exhibit properties that are isotropic or highly anisotropic, depending on the desired end result.

2.1.1. Sisal

Sisal is valued for cordage use because of its strength, durability, ability to stretch, affinity for certain dyestuffs, and resistance to deterioration in salt-water. Three grades of sisal were used in industry. The lower-grade fiber is processed by the paper industry because of its high content of cellulose and hemicelluloses. The medium-grade fiber is used in the cordage industry for making ropes, baler and binders twine. Ropes and twines are widely employed for marine, agricultural, and general industrial use. The higher-grade fiber after treatment is converted into yarns and used by the carpet industry.

Uses of sisal: products made from sisal are being developed rapidly, such as furniture and wall tiles made of resonated sisal. A recent development expanded the range, even to car parts for cabin interiors. Other products developed from sisal fiber include spa products, cat scratching posts, lumbar support belts, rugs, slippers, cloths and disc buffers.

Sisal wall covering meets the abrasion and tearing resistance standards of the American society for testing materials and of the national fire protection association. Apart from ropes, twines and general cordage, sisal is used in low-cost and specialty paper, dartboards, buffing cloth, filters, geotextiles, mattresses, carpets, handicrafts, wire rope cores and macramé. In recent years, sisal has been utilized as a strengthening agent to replace asbestos and fiberglass as well as an environmentally friendly component in the automobile industry. Products made from sisal fiber are purchased throughout the world and for use by the military, universities, churches and hospitals.

2.1.2. Roselle

The roselle (*Hibiscus sabdariffa*) is a species of hibiscus native to the old world tropics. It is an annual or perennial herb or woody-based subshrub, growing to 2–2.5 m tall. The leaves are deeply three- to five-lobed, 8–15 cm long, arranged alternately on the stems. The flowers are 8–10 cm in diameter, white to pale yellow with a dark red spot at the base of each petal, and have a stout fleshy calyx at the base, 1.5–2 cm wide, enlarging to 3–3.5 cm, fleshy and bright red as the fruit matures. It is an annual plant, and takes about six months to mature. Roselle is native to the areas from India to Malaysia, where it is commonly cultivated, and must have been carried at an early date to Africa. It has been widely distributed in the tropics and subtropics of both hemispheres, and in many areas of the West Indies and Central America has become naturalized.

Uses of roselle: The seeds are considered excellent feed for chickens. The residue after oil extraction is valued as cattle feed when available in quantity. Nutritionists have found roselle calyces, sold in Central American markets, to be rich in calcium, niacin, riboflavin and iron.

2.1.3. Banana fiber

Banana fiber, a ligno-cellulosic fiber, obtained from the pseudo-stem of banana plant (*Musa sapientum*), is a bast fiber with relatively good mechanical properties. The “pseudo-stem” is a clustered, cylindrical aggregation of leaf stalk bases. Banana fiber at present is a waste product of banana cultivation and either not properly utilized or partially done so. The extraction of fiber from the pseudo-stem is not a common practice and much of the stem is not used for production of fibers. The buyers for banana fibers are

erratic and there is no systematic way to extract the fibers regularly. Useful applications of such fibers would regularize the demand which would be reflected in a fall of the prices.

Uses of banana and plantain. Culinary uses: banana leaves, pseudo-stems, fruit stalks and peels can all be used for various culinary purposes. Bananas are primarily eaten as a fruit, either on its own or as a part of a salad. All parts of the banana have medicinal applications as well.

2.2. Types of composite materials

1. Fibrous composites which consist of fibers in a matrix.

2. Laminated composites which consist of layers of various materials.

3. Particulate composites which are composed of particles in a matrix.

According to the matrix phase, composite materials are divided into three types:

1. Polymer matrix composites (PMCs). These are the most common and will be discussed here. Also known as fiber reinforced polymers (FRP) – in these materials, a polymer-based resin is the matrix, and a variety of fibers such as glass, carbon and aramid provide the reinforcement.

2. Metal matrix composites (MMCs) increasingly employed in the automotive industry. In these materials, a metal such as aluminium is used as the matrix reinforced with fibers such as silicon carbide.

3. Ceramic metal composites (CMCs) used in a very high-temperature environments. In these materials, a ceramic is used as the matrix reinforced with short fibers, or whiskers such as those made from silicon carbide and boron nitride.

This formula is used in the preparation of composites.

2.3. Chemical treatment

The fibers are powdered, then they are cleaned normally in clean running water and dried.

A glass beaker was filled with 6% NaOH and 80% of distilled water which make a solution. After the adequate drying of the fibers in normal shading for 2–3 hours, the fibers are taken and soaked in the prepared NaOH solution. Soaking is carried out for different time intervals, depending upon the strength of the fiber required. For this project the fibers are soaked in the solution for three hours.

After completing the soaking process, the fibers are taken out, washed in running water and dried for another 2 hours. Now the fibers are taken for the next fabrication process, namely the PROCASTING process.

2.4. Advantages of chemical treatment

First and foremost, chemical treatment with NaOH removes moisture content from the fibers, thereby increasing their strength. Second, the chemical treatment also enhances the flexural rigidity of the fibers. Third, this treatment removes all impurities adjoining the fiber material and also stabilizes the molecular orientation.

2.5. Manufacturing process

A mould is made of wood in dimensions given in ASTM D790. An OHP sheet is taken and releasing agent is applied over it and fitted with the inner side of the mould and allowed it to dry. A glass beaker and a glass rod or a stirrer are taken and cleaned well with running water and then with warm water. Thereafter the calculated quantity of bioepoxy resin and the measured quantity of hardener are added to a breaker and the mixture is stirred for nearly 25 minutes. The reason behind this stirring is to create a homogeneous mixture. Then, after the mixing is over, the calculated quantity of fibers is added and stirring process is continued for the next 45 minutes. Then the mixture is poured into the mould and rammed mildly for uniform settlement. Then the mould is allowed to solidify for nearly 24 hours.

2.6. Fiber volume fraction

The volume fraction of fiber was calculated by a method which enables the rule of mixtures to be applied and the measured composite properties to be analysed. The method involves measuring the density of the composite (ρ_c) of mass (M_C) at a given mass fraction of the resin (M_R) Volume fraction of resin (V_R) was calculated using the formula

$$V_R = \frac{M_R \cdot \rho_c}{M_C \cdot \rho_R}, \quad (1)$$

where ρ_R is the density of resin in g/m^3 .

Then the fiber volume fraction is determined

$$V_F = 1 - V_R. \quad (2)$$

2.7. Moisture absorption test

Flexural specimens as per ASTM standards were cut from the fabricated plate. Edges of the samples were sealed with polyester resin and subjected to moisture absorption. The composite specimens to be used for moisture absorption test were first dried in an air oven at 50 °C. Then these conditioned composite specimens were immersed in distilled water at 30 °C for about 5 days. At regular intervals, the specimens were removed from water and wiped with filter paper to remove surface water and weighed with digital balance of 0.01 mg resolution. The samples were immersed in water to permit the continuation of sorption until saturation limit was reached. The weighing was done within 30 s in order to avoid the error due to evaporation. The test was carried out according to ASTM D570 to find out the swelling of specimen. After 5 days, the test specimens were again taken out of the water bath and weighed.

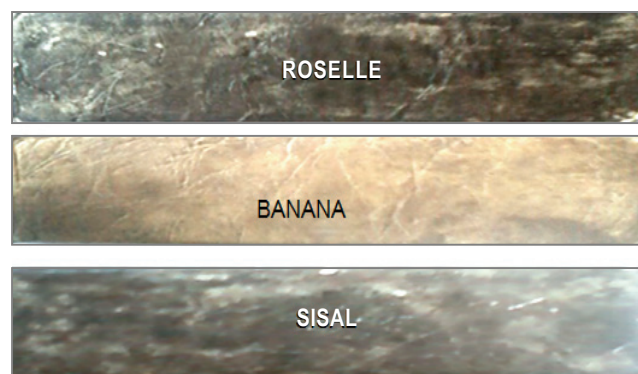


Fig. 1. Specimens for deflection test

3. Results

3.1. Flexural test

Flexural test is also known as bending test and consists in applying a point load at the centre of composite material. The flexural tests were performed on the universal testing machine, using the 3-point bending fixture according to ASTM D790 with the cross-head speed of 2 mm/min.

Table 1. Characteristics of natural fibers

Fiber type	Density (kg/m ³)	Modulus of elasticity <i>E</i> (N/mm ²)	Poisson ratio
Sisal	800–700	9·10 ³ –22·10 ³	0.32
Roselle	800–750	10·10 ³ –17·10 ³	0.33
Banana	950–750	11·10 ³ –23·10 ³	0.30

Compiled from Refs. [8], [15], [16].

such as electrical conductivity. SEM can produce very high-resolution images of a sample surface, revealing details about less than 2 to 5 nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field, yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. For conventional imaging in the SEM, specimens must

Table 2. Comparison of results





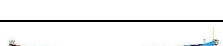
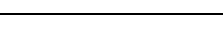
Material	Maximum deflection in experimental results (mm)	Maximum deflection in ANSYS (mm)	ANSYS
Roselle fiber without moisture	119.24	122.861	
Sisal fiber without moisture	73.57	77.410	
Banana fiber without moisture	52.54	59.230	
Roselle fiber with moisture	119.64	122.950	
Sisal fiber with moisture	73.27	77.662	
Banana fiber with moisture	50.18	59.338	

Table 3. Moisture absorption and volume fraction

Material	% moisture absorption	Volume fraction
Banana	6	0.72121
Roselle	5.4	0.77905
Sisal	5.7	0.7417

3.2. Scanning electron microscope

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample, producing signals that contain information about the sample's surface topography, composition and other properties

be electrically conductive, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge at the surface. Metal objects require little special preparation for SEM except for cleaning and mounting on a specimen stub. Nonconductive specimens tend to charge when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. They are therefore usually coated with an ultrathin coating of electrically-conducting material, commonly gold, deposited on the sample either by low-vacuum sputter coating or by high-vacuum evaporation. Conductive materials in current use for specimen coating include gold, gold/palladium alloy, platinum, osmium, iridium, tungsten, chromium and graphite. Coating prevents the accumulation of static

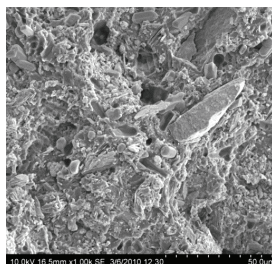


Fig. 2. SEM of sisal with moisture

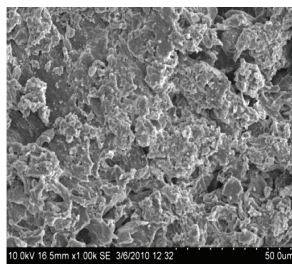


Fig. 3. SEM of roselle with moisture

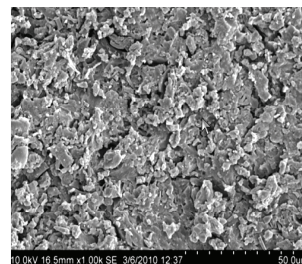


Fig. 4. SEM of banana with moisture

electric charge on the specimen during electron irradiation. The image may be captured by photography from a high-resolution cathode ray tube, but in modern machines is digitally captured and displayed on a computer monitor and saved to a computer's hard disc. All samples must also be of an appropriate size to fit in the specimen chamber and are generally mounted rigidly on a specimen holder, called a specimen stub. Several models of SEM can examine any part of a 6-inch (25 cm) semiconductor wafer, and some can tilt an object of that size to 45°.

4. Conclusion

After determining the material properties of natural fiber-reinforced epoxy composite using three different tool materials and geometries subjected to flexural test, the following conclusions can be drawn: The composites showed comparatively better performance. Sisal and banana fibers in flexural loading condition showed a brittle like failure. Elliptical cracks and their fast propagation could be observed. Less fiber pull-out was observed and this could be a reason for the reduction in the flexural strength. In bending fracture micrograph for the roselle and banana fiber-based composites, plastic deformation and more fiber pull-out could be observed. This is justified in the case where more percentage elongation could be observed for the roselle fiber as shown in figures 2, 3 and 4. The presence of moisture in the composites reduces their flexural properties. Since the absorption of moisture leads to the degradation of fiber matrix, the interface region creates poor stress transfer, resulting in a reduction of the flexural strength. Both in the roselle and sisal fiber composites, the percentage elongation is found to be increasing after immersing the components in water. The reason could be the water attack on the cellulose structure which allows the cellulose molecules to move smoothly. Hence, for the applications where flexural loading conditions are dominating, sisal and roselle composites could be selected. This could be attributed to fiber bridging through fiber pull-out. The complete breaking of the fiber rather than pull-out is observed in SEM analysis. Table 1 shows the maximum deflection of sisal and roselle fibers with and without moisture. Also in table 2, the maximum deflections from experimental test and ANSYS solution are compared. Finally, it can be concluded that one of the best materials is sisal, while roselle can be used for internal fixation and also external fixation. In the future, this plate

material externally coated with calcium phosphate and hydroxyapatite (hybrid) composite will be used for both internal and external fixation of human fractured bones. The researchers are of the opinion that the most important thing is that these steps taken now will help humans to develop and to have a more pleasant life.

4.1. Future development

When a bone is severely crushed, physicians usually cannot reset it and bone graft or amputation – until now – has remained a primary option. The same is true for bones damaged by disease, such as cancer. If, for instance, the humerus bone in the arm is injured and damaged, CT scan or MRI image can be made of the good arm bone, and converted into a “growth code” – a 3-D virtual image – of the replacement bone segment needed. In order to replace orthopaedic alloys such as titanium, cobalt chrome, stainless steel and zirconium, this project aims to fabricate natural fiber-reinforced polymer composite plate material by using bioepoxy resin. The materials used in this project (sisal, banana and roselle fiber-reinforced composites) will be coated with calcium phosphate and hydroxyapatite (hybrid) composite. This plate material can be used for both inside and external fixation of human fractured bone.

4.2. Calcium phosphate

Calcium phosphate salts were first applied as powders. The ceramic form first became available in the 1160 and was later evaluated as a good substitute for bone graft. The synthetic hydroxyapatite is one of the most commonly used calcium phosphate ceramics. Synthetic ceramics provide an osteoconductive scaffold to which chemotactic, circulating proteins and cells can migrate and adhere, and within which progenitor cells can differentiate into functioning osteoblasts. Ceramics do not supply osteogenic cells as is found in autograft. They do not have even the weak osteoinductive potential found with allograft. However, ceramics are readily available and allow us to avoid the known risks of allograft-induced immunogenic response or disease conveyance, as well as surgical complications due to retrieving bone from an autogenous second site. The chemistry, architecture, shape, and positioning of the ceramic material influence the speed and extent of bone remodelling. Its bioresorbability depends on the amount of surface

area exposed, which is governed, in turn, by crystal size, the form supplied, and density. A ceramic material formed as a dense block exposes only a small surface area, thus slowing or confining surfaces accessible for resorption. Most calcium phosphates are classified as resorbable biomaterials. This means that under physiological conditions they will dissolve. The benefit of calcium phosphate biomaterials is that the dissolution products can be readily assimilated by the human body. Calcium phosphate is mainly used in filling defects (for example, areas of bone loss such as in tibial plateau fracture), in composite grafts to supplement autograft, and at sites where compression (rather than tension, bending, or torsion) is the dominant mode of mechanical loading. Variation in the properties of calcium phosphate coatings has an effect on the bone-bonding mechanism and the rate of bone formation. Both the composition and the crystallinity of the calcium phosphate coating are important parameters that determine its bioactivity characteristics. Hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) has the ability to bond to osseous and epithelial tissues. Hydroxyapatite, with beneficial bone tissue growth effects, is used as a coating material since it does not have sufficient strength and toughness to be used by itself as biomedical implant. Unlike other calcium phosphates, hydroxyapatite does not break down under physiological conditions. Under normal physiological conditions of pH 7.2 hydroxyapatite is the stable calcium phosphate compound. This may drop to as low as pH 5.5 in the region of tissue damage, although this would eventually return to pH 7.2 over a period of time. Even under these conditions hydroxyapatite is still the stable phase. It actively takes part in bone bonding, forming strong chemical bonds with surrounding bone. This property has been exploited for rapid bone repair after major trauma or surgery.

4.3. Hydroxyapatite (HA)

Hydroxyapatite belonging to a class of calcium phosphate-based bioceramics is frequently used as bone graft substitute owing to its chemical and structural similarity to the natural bone mineral. Its chemical composition is given as $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. The Young's modulus of HA ranges between 80 and 110 (GPa). The elastic modulus of HA is 114 GPa. Fracture toughness is predominant up to 0.7–1.2. Its biocompatibility is high. Although HA is an excellent bone graft, its inherent low fracture toughness has limited use in certain orthopaedic application, in particular in heavy load bearing implantations.

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