CHAPTER 1

INTRODUCTION

1.1 GENERAL

The social and economical developments of human are largely dependent on better utilization of the available resources. Composites are an example for the same. They are engineered from two or more constituent materials with significantly different physical or chemical properties and which remain separate and distinct on a macroscopic level within the finished structure. Fiber Reinforced Polymers or FRPs include Wood comprising (cellulose fibers in a lignin and hemicellulose matrix), Carbon-fiber reinforced plastic or CFRP and Glass-fiber reinforced plastic or GFRP (also GRP). Bricks made of clay and reinforced with straw are an early example of application of composites as used by Israelites. The individual constituents, clay and straw, could not serve the function by themselves but did when put together. Some believe that the straw was used to keep the clay from cracking, but others suggest that it blunted the sharp cracks in the dry clay. Historical examples of composites are abundant in the literature. Significant examples include the use of reinforcing mud walls in houses with bamboo shoots, glued laminated wood by Egyptians (1500 B.C.), and laminated metals in forging swords (A.D.1800). Modern composites were used in the 1930s when glass fibers reinforced resins. Boats and aircraft were built out of these glass composites, commonly called fiberglass. Since 1970s, application of composites has widely increased due to development of new fibers such as carbon, boron and new composite systems with matrices made of metals and
ceramics. Composite materials have gained popularity (despite their generally high cost) in high performance products such as aerospace components (tails, wings, fuselages, propellors), boat and scull hulls and racing car bodies. More mundane uses include fishing rods and storage tanks. The synthetic fibres used in the composite material pollute the environment because of their non biodegradability. During the last decade there has been a renewed interest in the natural fibre as a substitute for glass, motivated by potential advantages such as weight saving, low raw material price and thermal recycling or the ecological advantages of using resources which are renewable. However, increased environmental consciousness particularly on recycling of traditional materials, unprecedented forest resources degeneration and global warming, have led to world wide efforts to develop natural fibre composites from non wood resources. On the other hand, natural fibres have their shortcomings and these have to be solved in order to be competitive with glass fibres. Environmentally sustainable lignocellulosic resources are available in different forms of non wood based fibres and agricultural residues. Natural commercial fibres include jute, sisal, kapok, kenaf, flax, hemp, ramie etc. Agriculture residues include stalks of most cereal crops, rice husks, coconut fibres, bagasse, peanut shells, and other waste. Most of the recent developments of bio-composites from non wood lignocellulosic resources have been aimed at improving the quality and performance of the product.

Natural fibres play an important role in developing high performing fully biodegradable ‘green’ composites, which will be a key material to solve the current ecological and environmental problems. The natural fibre reinforced composite is a lightweight, naturally attractive, cost effective application of renewable materials. It will help to improve cultivation of fibre plants and also economy of the country. Nowadays, wood substitute is found to be commercially cost effective and it plays a major and increasing role as alternative material in the composite industry.
1.2 NATURAL FIBRE

Environmental awareness and an increasing concern with the greenhouse effect have stimulated the construction, automotive and packing industries to look for sustainable materials that can replace conventional synthetic polymeric fibres. Natural fibres seem to be a good alternative since they are readily available in fibrous form and can be extracted from plant leaves at very low costs. Several years ago, nearly all resources for the production of commodities and many technical products, were materials derived from natural textiles. Textiles, ropes, canvas and also paper, were made of local natural fibres. Natural fibres are subdivided based on their origins, coming from plants, animals or minerals. Generally, plant or vegetable fibres are used to reinforce plastic. Plant fibres may include hairs (cotton, kapok), fibre sheaves of dicot plants or vessel sheaves of monocot plants, i.e. bast (flax, hemp, jute, and ramie) and hard fibres (sisal, henequen, and coir).

A single fibre of all plant based natural fibres consists of several cells. These cells are formed out of crystalline microfibrils based on cellulose, which are connected to a complete layer, by amorphous lignin and hemicellulose. Many of such cellulose-lignin/hemicellulose layers in one primary and three secondary cell walls stick together to a multiple layer composites. These cell walls differ in their composition and in the orientation of the cellulose microfibrils. These fibres are composed mainly of cellulose and some lignin and are sometimes called lingo-cellulosic fibres. The natural fibres are classified as shown in Figure 1.1.

1.2.1 Bast Fibres

In general, the bast consists of a wood core surrounded by a stem. Within the stem there are a number of fibre bundles, each containing
individual fibre cells or filaments. The filaments are made of cellulose and hemicellulose, bonded together by a matrix, which can be lignin or pectin. The pectin surrounds the bundle thus holding them on to the stem. The pectin is removed during the retting process. This enables separation of the bundles from the rest of the stem. The bast fibres are found in the inner bast tissue of certain plant stems and are made up of overlapping cells, for example flax, hemp, jute, kenaf, ramie etc.,.

1.2.2 Leaf Fibres

In general, the leaf fibres are coarser than the bast fibres. Their applications are in manufacture of ropes, and coarse textiles. Among the total production of leaf fibres, sisal is the most important. Its stiffness is relatively high and it is often applied as binder twines. The leaf fibres are a part of the fibro vascular system of the leaves, for example sisal, henequen, palm and abaca.

1.2.3 Seed Fibres

Seed fibres are those that are borne on the seed coats as hairy structures or on the inner walls of the fruit, where each fibre consists of a single, long, narrow cell as in cotton and coir. Cotton is the most common seed fibre and is used for textile all over the world. Other seed fibres are applied in less demanding applications such as stuffing of upholstery. Coir is an exception to this. Coir is the fibre of the coconut husk; it is thick and coarse but durable and used to make ropes, mats and brushes.

1.2.4 Advantages of Natural Fibres

- Low specific weight, which results in a higher specific strength and stiffness than glass
- It is a renewable resource wherein the production requires little energy and CO₂ is used while oxygen is given back to the environment
- Production with low investment at low cost, which makes the material an interesting product for low wage countries
- Friendly processing, no wear of tooling and no skin irritation
- Thermal recycling is possible, where glass causes problems in combustion furnaces
- Good thermal and acoustic insulating properties

![Figure 1.1 Classification of natural fibres](image-url)
1.2.5 Disadvantages of Natural Fibres

- Lower strength properties, particularly its impact strength
- Variable quality, depending on unpredictable influences such as weather
- Moisture absorption, which causes swelling of the fibres
- Restricted maximum processing temperature
- Low durability but fibre treatment can improve this considerably
- Poor fire resistance
- Price can fluctuate by harvest results or agricultural politics

All vegetable fibres are made up of mainly cellulose and hemicellulose, lignin, pectin, and wax. All natural fibres are susceptible to microbial decomposition. Natural fibres based on cellulose have a relatively low density, and are relatively stiff and strong.

Natural fibres are amenable to modifications as they bear hydroxyl groups from cellulose and lignin. The hydroxyl groups may be involved in the hydrogen bonding within the cellulose molecules thereby reducing the activity towards the matrix. Chemical modifications may activate these groups that can effectively interlock with the matrix. Surface characteristics such as wetting, adhesion, surface tension, porosity, etc. can be improved upon modifications. Alkali treatment of the fibres may lead to major changes in fibre surface roughness. The irregularities of the fibre surface play an important role in the mechanical interlocking at the interface. The increase in adhesion of the resin onto the fibres happens due to the physical and chemical changes occurred to the fibre treatments. Physical changes may include removal of the waxy layer, changes in the surface roughness, changes in the
physical appearance of the fibre and density changes. This may lead to changes in the adhesive strength of the fibre onto the matrix and lead to improve interface properties of the composites. For the automotive industry, where weight reduction constantly is an issue, this way said to be the original reason for the development of interior parts with natural fibres as fillers.

Originally, wood fibres used by the automotive industry are quite short and do have the ability to make composites stiffer, but they do not make them stronger. Therefore, later, materials reinforced with other longer natural cellulose fibres were developed. Most of the interior parts in cars are still designed for stiffness and natural fibres are well matched for this application. Natural fibre reinforced composites are initially aimed at the replacement of glass fibre reinforced composites. If a natural fibre with an equivalent quality of glass fibre is characterized, then the former can replace the latter by its cheaper cost. Natural fibres are also expected to give less health problems for the people producing the composites.

Table 1.1 Composition of different cellulose based natural fibre (Bledzki and Gassan 1999)

<table>
<thead>
<tr>
<th>Fibre</th>
<th>Cotton</th>
<th>Jute</th>
<th>Flax</th>
<th>Ramie</th>
<th>Sisal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cellulose</td>
<td>82.7</td>
<td>64.4</td>
<td>64.1</td>
<td>68.6</td>
<td>65.8</td>
</tr>
<tr>
<td>Hemi-cellulose</td>
<td>5.7</td>
<td>12.0</td>
<td>16.7</td>
<td>13.1</td>
<td>12.0</td>
</tr>
<tr>
<td>Pectin</td>
<td>5.7</td>
<td>0.2</td>
<td>1.8</td>
<td>1.9</td>
<td>0.8</td>
</tr>
<tr>
<td>Lignin</td>
<td>-</td>
<td>11.8</td>
<td>2.0</td>
<td>0.6</td>
<td>9.9</td>
</tr>
<tr>
<td>Water soluble</td>
<td>1.0</td>
<td>1.1</td>
<td>3.9</td>
<td>5.5</td>
<td>1.2</td>
</tr>
<tr>
<td>Wax</td>
<td>0.6</td>
<td>0.5</td>
<td>1.5</td>
<td>0.3</td>
<td>0.3</td>
</tr>
</tbody>
</table>
Natural fibres offer several advantages over glass fibres

- Plant fibres are renewable raw materials and their availability is more. When natural reinforced plastics were subjected, at the end of their life cycle, to a combustion process, the released amount of CO$_2$ of the fibres is natural with respect to the assimilated amount during their growth.

- The abrasive nature of natural fibres is much lower compared to that of glass fibres, which leads to advantages with regard to technical, material recycling or processing of composite materials in general.

**Table 1.2  Mechanical properties of natural fibres as compared with conventional reinforcing fibres (Bledzki and Gassan 1999)**

<table>
<thead>
<tr>
<th>Fibre</th>
<th>Density (g/cm$^3$)</th>
<th>Elongation (%)</th>
<th>Tensile strength (MPa)</th>
<th>Young’s modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton</td>
<td>1.5-1.6</td>
<td>7.0-8.0</td>
<td>287-597</td>
<td>5.5-12.6</td>
</tr>
<tr>
<td>Jute</td>
<td>1.3</td>
<td>1.5-1.8</td>
<td>393-773</td>
<td>26.5</td>
</tr>
<tr>
<td>Flax</td>
<td>1.5</td>
<td>2.7-3.2</td>
<td>345-1035</td>
<td>27.6</td>
</tr>
<tr>
<td>Hemp</td>
<td>-</td>
<td>1.6</td>
<td>690</td>
<td>-</td>
</tr>
<tr>
<td>Ramie</td>
<td>-</td>
<td>3.6-3.8</td>
<td>400-938</td>
<td>61.4-128</td>
</tr>
<tr>
<td>Sisal</td>
<td>1.5</td>
<td>2.0-2.5</td>
<td>511-635</td>
<td>9.4-22.0</td>
</tr>
<tr>
<td>Coir</td>
<td>1.2</td>
<td>30.0</td>
<td>175</td>
<td>4.0-6.0</td>
</tr>
<tr>
<td>Viscose(cord)</td>
<td>-</td>
<td>11.4</td>
<td>593</td>
<td>11.0</td>
</tr>
<tr>
<td>Soft wood Kraft</td>
<td>1.5</td>
<td>-</td>
<td>1000</td>
<td>40.0</td>
</tr>
<tr>
<td>E-glass</td>
<td>2.5</td>
<td>2.5</td>
<td>2000-3500</td>
<td>70.0</td>
</tr>
<tr>
<td>S-glass</td>
<td>2.5</td>
<td>2.8</td>
<td>4570</td>
<td>86.0</td>
</tr>
<tr>
<td>Aramide (normal)</td>
<td>1.4</td>
<td>3.3-3.7</td>
<td>3000-3150</td>
<td>63.0-67.0</td>
</tr>
<tr>
<td>Carbon</td>
<td>1.4</td>
<td>1.4-1.8</td>
<td>4000</td>
<td>230-240</td>
</tr>
</tbody>
</table>
1.3 MATRIX

The role of the matrix in a fibre reinforced composite is: (i) to keep the fibres in place, (ii) to transfer stresses between the fibres, (iii) to provide a barrier against an adverse environment, such as chemicals and moisture and (iv) to protect the surface of the fibres from mechanical degradation. The matrix plays a minor role in the tensile load carrying capacity of a composite structure. However, selection of a matrix has a major influence on the compressive, interlaminar shear as well as in-plane shear properties of the composite material. Polymer matrix is a long chain molecule containing one or more repeating units of atoms joined together by strong covalent bonds for which classification is shown in Figure 1.2.

Figure 1.2 Classification of polymer matrix

1.3.1 Thermosets Polymer

In a thermoset, polymer molecules are chemically joined together by cross links, forming a rigid and three dimensional network structures.
Once these cross links are formed during the polymerization reaction, the thermoset polymer cannot be melted by the application of heat. However, if the number of cross links is low, it may still be possible to soften them at elevated temperature. Traditionally, thermoset polymers have been used as a matrix material for fibre reinforced composites. The viscosity of the polymer at the time of fibre incorporation is very low. It is possible to achieve a good wet out between the fibres and the matrix without the aid of either high temperature or pressure. Among other advantages of using thermoset polymers are their thermal stability and chemical resistance.

1.3.2 Epoxy Resin

The epoxy matrix are low molecular weight organic liquid resins containing a number of epoxide groups, which are three member rings of one oxygen atom and two carbon atoms. Epoxy or Polyepoxide is a thermosetting epoxide polymer that cures (polymerizes and cross links) when mixed with a catalyzing agent or "hardener". The most common and widely used epoxy resins are made by allowing polycondensation involving epichlorohydrin and bisphenol-A. The quality of the epoxy resin is determined by the viscosity, hydroxyl equivalent and epoxy equivalent, and molecular weight.

These high performance adhesives are used in the construction of aircraft, automobiles, bicycles, golf clubs, skis, snow boards, and other applications where high strength bonds are required. Epoxy adhesives capable of meeting almost any application can be developed. They can be made flexible or rigid, transparent or opaque/coloured, fast setting or extremely slow. In general, epoxy adhesives cured with heat will be more heat and chemical resistant than when cured at room temperature. Epoxies are sold in hardware stores, typically as two component kits.
1.3.2.1 Epoxy Hardeners

Epoxy hardeners are not catalysts and they react with the epoxy resins, greatly contributing to the ultimate properties of the cured epoxy resin system. Epoxy hardeners provide: gel time, mixed viscosity, demould time of the epoxy resin system.

Physical properties of the epoxy resin system such as tensile, compression, flexural properties, etc., are also influenced by epoxy hardeners. The performance of epoxy hardeners in the epoxy resins system depend on the chemical characteristics of the epoxy resins and the physical characteristics while applying the epoxy resins system. The chemical characteristics of the epoxy resins that influence epoxy hardeners are: viscosity amount and kind of diluents and fillers in epoxy resins.

The physical characteristics of the epoxy resins system influencing the behaviour of epoxy hardeners in the epoxy resins system are: temperature of the work area, temperature of the resins system (i.e. the heated resins), and moisture (dampness).

1.3.2.2 Chemistry

![Figure 1.3 Structure of unmodified epoxy prepolymer.](image)

Where, n denotes the number of polymerized subunits and is in the range from 0 to about 25.
When epoxy is mixed with the appropriate catalyst, the resulting reaction is exothermic, and the oxygen on the epoxy monomers is "flipped." This occurs throughout the epoxy, and a matrix with a high stress tolerance is formed, and "glues" the materials together (Figures 1.3 and 1.4).

![Chemical reaction between Bisphenol-A and Epichlorohydrin](image)

**Figure 1.4 Chemical reaction between Bisphenol-A and Epichlorohydrin**

### 1.4 AGAVE PLANT

*Agave americana* (Agave) is a succulent plant of a large botanical genus of the same name, belonging to the family Agavaceae (Figure 1.5). Agave is a native of central and North America. This species is probably known as the Agave most commonly grown as an ornamental plant, and it is spread throughout the temperate and tropical areas of the world. It is abundantly found in arid and semi-arid regions. It has been successfully imported to India as well. The plant has a large rosette of thick fleshy leaves with a spiny margin and ending in a sharp point. The leaves are long, tough and rigid, with very sharp, hard points which can easily penetrate through clothes and leather. In general the leaf fibres are coarse than the bast fibres. The Agave reacts dynamically to their environment in the dry season wherein they will pucker up to reduce the amount of surface for evaporation. In times of excess heat or sun, they will adapt the angle of their leaves to either get more sun or reduce its effects. Agave fibre is extracted by rural people for the manufacturing of ropes and coarse textiles.
Natural fibre composites, may in the future, become materials to replace synthetic fibre polymer composites. Natural fibre incorporated polymers have been very fashionable due to their flexibility, their lightness and the ease of fabrication of complicated shapes with economic saving. The quality and performance of plant fibre based composites can further be improved by adopting appropriate engineering techniques. In addition, these composites can easily substitute for conventional materials in several areas such as the automotive industry, building industry, consumer goods and sport goods. Many automotive and household components are produced using natural composites, mainly based on polyester and fibre like flax, hemp, pineapple, coir and sisal. The application of natural fibre composites in this industry is lead by motives of price, weight reduction, and biodegradability.
1.6 NEED FOR NATURAL FIBRE COMPOSITES

The development of natural fibre composites has profited from the policy of a number of (Indian) governments to support the development of technical applications for renewable resources. Establishment of disposal methods for glass fibre reinforced plastics and their recycling laws are important contemporary subjects because many environmental problems have appeared. It is necessary to reduce environmental impacts such as global warming, which are generated by consumption of petroleum, a non-renewable resource. The driving forces of natural fibre composites are (i) cost reduction, (ii) weight reduction and (iii) marketing (application of renewable materials). The use of natural fibre reinforced polymer represents an attractive and suitable method for replacing. Natural fibres are low cost, renewable and high specific strength and its composites are used for fabricating some products such as furniture and architectural materials. Recently, they have gained widespread use in the automobile industry.

1.7 SCOPE OF THE PRESENT WORK

In this thesis the relation between the structure and the mechanical properties of untreated and alkali treated Agave fibres and their reinforced epoxy composites were carried out with the following objectives.

- To develop a procedure for extraction of fibre from the plant.
- To do a detailed study of the physical, chemical and mechanical properties of Agave fibre and also the surface morphology of the fibre.
- To design and fabricate a mould.
• To make the unidirectional continuous Agave fibre (treated and untreated) reinforced epoxy composite samples.

• To investigate the mechanical properties (tensile, compressive, flexural and impact), moisture absorption test, dynamic mechanical analysis, Fourier transform infrared spectroscopy and scanning electron microscopy of the continuous Agave fibre reinforced epoxy composites.

• To make the chopped Agave fibre (treated and untreated) reinforced epoxy composite samples.

• To study the performance of the chopped Agave fibre reinforced epoxy composites by analysing the tensile, compressive, flexural and impact properties, moisture absorption, dynamic mechanical property, machinability and morphology.

• To analyse the wear performance and the worn surface of the chopped Agave fibre reinforced epoxy composites for tribological reasons.

• To investigate the mechanical properties (tensile, flexural and impact), Fourier transform infrared spectroscopy and scanning electron microscopy of the continuous/short Agave/glass fibre reinforced epoxy hybrid composites.

• To select the Agave fibre reinforced epoxy composite of best mechanical qualities from the above experimental results and to fabricate a house hold table, two wheeler automotive bumper and hockey stick as to show its application in lightweight manufacture.
1.8 OUTLINE OF THESIS

Studies on Agave Americana Fibre Reinforced Composite Materials

Chapter 1 Introduction
1.1 General
1.2 Natural fibre (NF)
1.3 Matrix
1.4 Agave plant
1.5 Natural fibre composite materials (NFC)
1.6 Need for NFC
1.7 Aim and Scope of work
1.8 Outline of thesis

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2.1 Introduction
2.2 Characterization and mechanical properties of NF
2.3 Mechanical and morphological properties of continuous NFC
2.4 Mechanical and morphological properties of short NFC
2.5 Wear behaviour of NFC
2.6 Application of NFC

Chapter 3 Materials and Experiments
3.1 Harvest of Agave americana plant
3.2 Fibre extraction from plant
3.3 Alkali treatment of fibre
3.4 Physical and chemical analysis of Agave fibre
3.5 Making of mould
3.6 Specimen preparation
3.7 - 3.12 Tensile, Compressive, Flexural, Impact, Water absorption and Machinability tests
3.13 - 3.16 DMA, AFM, and SEM tests
3.17 Wear test
3.18 Agave/glass fibre reinforced hybrid composite specimen preparation

Chapter 4 Results and Discussion
4.1 Analysis of Agave americana fibre
4.2 Analysis of continuous Agave fibre (untreated and alkali treated) epoxy composites
4.3 Analysis of chopped Agave fibre (untreated and alkali treated) epoxy composites
4.4 Wear analysis of chopped Agave fibre
4.5 Investigation on Agave/glass hybrid composite materials

Chapter 5 Application of the experimental materials
5.1 Fabrication of household table
5.2 Fabrication of hockey stick
5.3 Fabrication of two wheeler bumper

Chapter 6 Summary and Conclusions
6.1 Analysis of Agave americana fibre
6.2 Analysis of continuous Agave fibre epoxy composites
6.3 Analysis of chopped Agave fibre epoxy composites
6.4 Wear analysis of chopped Agave fibre
6.5 Investigation on Agave/glass hybrid composite materials
6.5 Applications of the Agave fibre reinforced composite materials
6.6 Scope of future study

Figure 1.6 Outline of thesis