PROCESSING AND CHARACTERIZATION OF NATURAL FIBER REINFORCED POLYMER COMPOSITES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology in Mechanical Engineering

BY

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DEPARTMENT OF MECHANICAL ENGINEERING
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CERTIFICATE

This is to certify that the thesis entitled "***Processing and Characterization of Natural Fiber Reinforced Polymer Composites***" submitted by ***Prakash Tudu*** *(Roll No.* **10503005***)* in partial fulfillment of the requirements for the award of ***Bachelor of Technology*** in the department of Mechanical Engineering, National Institute of Technology, Rourkela is an authentic work carried out under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to elsewhere for the award of any degree.

Place: Rourkela
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ABSTRACT

Polymeric materials reinforced with synthetic fibres such as glass, carbon, and aramid provide advantages of high stiffness and strength to weight ratio as compared to conventional construction materials, i.e. wood, concrete, and steel. Despite these advantages, the widespread use of synthetic fibre-reinforced polymer composite has a tendency to decline because of their high-initial costs, their use in non-efficient structural forms and most importantly their adverse environmental impact. On the other hand, the increase interest in using natural fibres as reinforcement in plastics to substitute conventional synthetic fibres in some structural applications has become one of the main concerns to study the potential of using natural fibres as reinforcement for polymers. In the light of this, researchers have focused their attention on natural fibre composite (i.e. bio-composites) which are composed of natural or synthetic resins, reinforced with natural fibres. Accordingly, manufacturing of high-performance engineering materials from renewable resources has been pursued by researchers across the world owning to renewable raw materials are environmentally sound and do not cause health problem. The present work includes the processing, characterization of coconut fiber reinforced epoxy composites. . It further outlines a methodology based on Taguchi's experimental design approach to make a parametric analysis of erosion wear behaviour. The systematic experimentation leads to determination of significant process parameters and material variables that predominantly influence the wear rate.

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Chapter 1

INTRODUCTION

CHAPTER 1

1. INTRODUCTION

1.1. Overview of composites

Over the last thirty years composite materials, plastics and ceramics have been the dominant emerging materials. The volume and number of applications of composite materials have grown steadily, penetrating and conquering new markets relentlessly. Modern composite materials constitute a significant proportion of the engineered materials market ranging from everyday products to sophisticated niche applications. While composites have already proven their worth as weight-saving materials, the current challenge is to make them cost effective. The efforts to produce economically attractive composite components have resulted in several innovative manufacturing techniques currently being used in the composites industry. It is obvious, especially for composites, that the improvement in manufacturing technology alone is not enough to overcome the cost hurdle. It is essential that there be an integrated effort in design, material, process, tooling, quality assurance, manufacturing, and even program management for composites to become competitive with metals. The composites industry has begun to recognize that the commercial applications of composites promise to offer much larger business opportunities than the aerospace sector due to the sheer size of transportation industry. Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years. Increasingly enabled by the introduction of newer polymer resin matrix materials and high performance reinforcement fibers of glass, carbon and aramid, the penetration of these advanced materials has witnessed a steady expansion in uses and volume. The increased volume has resulted in an expected reduction in costs. High performance FRP can now be found in such diverse applications as composite armoring designed to resist explosive impacts, fuel cylinders for natural gas vehicles, windmill blades, industrial drive shafts, support beams of highway bridges and even paper making rollers. For certain applications, the use of composites rather than metals has in fact resulted in savings of both cost and weight. Some examples are cascades for engines, curved fairing and fillets, replacements for welded metallic parts, cylinders, tubes, ducts, blade containment bands etc. Further, the need of composite for lighter construction materials and more seismic resistant structures has placed high emphasis on the use of new and advanced materials that not only decreases dead weight but also absorbs the shock & vibration through tailored microstructures. Composites are now extensively being used for rehabilitation/ strengthening of pre-existing structures that have to be retrofitted to make them seismic resistant, or to repair damage caused by seismic activity. Unlike conventional materials (e.g., steel), the properties of the composite material can be designed considering the structural aspects. The design of a structural component using composites involves both material and structural design. Composite properties (e.g. stiffness, thermal expansion etc.) can be varied continuously over a broad range of values under the control of the designer. Careful selection of reinforcement type enables finished product characteristics to be tailored to almost any specific engineering requirement. Whilst the use of composites will be a clear choice in many instances, material selection in others will depend on factors such as working lifetime requirements, number of items to be produced (run length), complexity of product shape, possible savings in assembly costs and on the experience & skills the designer in tapping the optimum potential of composites. In some instances, best results may be achieved through the use of composites in conjunction with traditional materials.

1.2. Definition of composite

The most widely used meaning is the following one, which has been stated by **Jartiz** “Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form”.

The weakness of this definition resided in the fact that it allows one to classify among the composites any mixture of materials without indicating either its specificity or the laws which should given it which distinguishes it from other very banal, meaningless mixtures.

Kelly very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength to resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Beghezan defines as “The composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their short comings”, in order to obtain improved materials.

Van Suchetclan explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

1.3. Merits of Composites

Advantages of composites over their conventional counterparts are the ability to meet diverse design requirements with significant weight savings as well as strength-to-weight ratio. Some advantages of composite materials over conventional ones are as follows:

- Tensile strength of composites is four to six times greater than that of steel or aluminium (depending on the reinforcements).
- Improved torsional stiffness and impact properties.
- Higher fatigue endurance limit (up to 60% of ultimate tensile strength).
- 30% - 40% lighter for example any particular aluminium structures designed to the same functional requirements.
- Lower embedded energy compared to other structural metallic materials like steel, aluminium etc.

- Composites are less noisy while in operation and provide lower vibration transmission than metals.
- Composites are more versatile than metals and can be tailored to meet performance needs and complex design requirements.
- Long life offer excellent fatigue, impact, environmental resistance and reduce maintenance.
- Composites enjoy reduced life cycle cost compared to metals.
- Composites exhibit excellent corrosion resistance and fire retardancy.
- Improved appearance with smooth surfaces and readily incorporable integral decorative melamine are other characteristics of composites.
- Composite parts can eliminate joints / fasteners, providing part simplification and integrated design compared to conventional metallic parts.

Broadly, composite materials can be classified into three groups on the basis of matrix material. They are:

a) Metal Matrix Composites (MMC)
b) Ceramic Matrix Composites (CMC)
c) Polymer Matrix Composites (PMC)

a) Metal Matrix Composites

Metal Matrix Composites have many advantages over monolithic metals like higher specific modulus, higher specific strength, better properties at elevated temperatures, and lower coefficient of thermal expansion. Because of these attributes metal matrix composites are under consideration for wide range of applications viz. combustion chamber nozzle (in rocket, space shuttle), housings, tubing, cables, heat exchangers, structural members etc.

b) Ceramic matrix Composites

One of the main objectives in producing ceramic matrix composites is to increase the toughness. Naturally it is hoped and indeed often found that there is a concomitant improvement in strength and stiffness of ceramic matrix composites.

c) Polymer Matrix Composites

Most commonly used matrix materials are polymeric. The reason for this are two fold. In general the mechanical properties of polymers are inadequate for many structural purposes. In particular their strength and stiffness are low compared to metals and ceramics. These difficulties are overcome by reinforcing other materials with polymers. Secondly the processing of polymer matrix composites need not involve high pressure and doesn't require high temperature. Also equipments required for manufacturing polymer matrix composites are simpler. For this reason polymer matrix composites developed rapidly and soon became popular for structural applications.

Composites are used because overall properties of the composites are superior to those of the individual components for example polymer/ceramic. Composites have a greater modulus than the polymer component but aren't as brittle as ceramics.

Two types of polymer composites are:

- Fiber reinforced polymer (FRP)
- Particle reinforced polymer (PRP)

Fiber Reinforced Polymer

Common fiber reinforced composites are composed of fibers and a matrix. Fibers are the reinforcement and the main source of strength while matrix glues all the fibers together in shape and transfers stresses between the reinforcing fibers. The fibers carry the loads along their longitudinal directions. Sometimes, filler might be added to smooth the manufacturing process, impact special properties to the composites, and / or reduce the product cost.

Common fiber reinforcing agents include asbestos, carbon / graphite fibers, beryllium, beryllium carbide, beryllium oxide, molybdenum, aluminium oxide, glass fibers, polyamide, natural fibers etc. Similarly common matrix materials include epoxy, phenolic, polyester, polyurethane, polyetherethrketone (PEEK), vinyl ester etc. Among these resin materials, PEEK is most widely used.

Epoxy, which has higher adhesion and less shrinkage than PEEK, comes in second for its high cost.

Particle Reinforced Polymer

Particles used for reinforcing include ceramics and glasses such as small mineral particles, metal particles such as aluminium and amorphous materials, including polymers and carbon black. Particles are used to increase the modules of the matrix and to decrease the ductility of the matrix. Particles are also used to reduce the cost of the composites. Reinforcements and matrices can be common, inexpensive materials and are easily processed. Some of the useful properties of ceramics and glasses include high melting temp., low density, high strength, stiffness, wear resistance, and corrosion resistance. Many ceramics are good electrical and thermal insulators. Some ceramics have special properties; some ceramics are magnetic materials; some are piezoelectric materials; and a few special ceramics are even superconductors at very low temperatures. Ceramics and glasses have one major drawback: they are brittle. An example of particle reinforced composites is an automobile tire, which has carbon black particles in a matrix of poly-isobutylene elastomeric polymer.

Polymer composite materials have generated wide interest in various engineering fields, particularly in aerospace applications. Research is underway worldwide to develop newer composites with varied combinations of fibers and fillers so as to make them useable under different operational conditions. Against this backdrop, the present work has been taken up to develop a series of PEEK based composites with glass fiber reinforcement and with ceramic fillers and to study their response to solid particle erosion.

1.4. Characteristics of the Composites

A composite material consists of two phases. It consists of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous phase and is called the ‘reinforcement‘ or ‘reinforcing material’, whereas the continuous phase is termed as the ‘ matrix’. The matrix is usually more ductile and less hard. It holds the dispersed phase and shares a load with it. Matrix is composed of any of the three basic material type i.e. polymers, metals or ceramics. The matrix forms the bulk form or the part or product. The secondary phase embedded in the matrix is a discontinuous phase. It is usually harder and stronger than the continuous phase. It servers to strengthen the composites and improves the overall mechanical properties of the matrix.

Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and size distribution) influences the properties of the composite to a great extent. The concentration distribution and orientation of the reinforcement also affect the properties.

The shape of the discontinuous phase (which may by spherical, cylindrical, or rectangular cross-sanctioned prisms or platelets), the size and size distribution (which controls the texture of the material) and volume fraction determine the interfacial area, which plays an important role in determining the extent of the interaction between the reinforcement and the matrix.

Concentration, usually measured as volume or weight fraction, determines the contribution of a single constituent to the overall properties of the composites. It is not only the single most important parameter influencing the properties of the composites, but also an easily controllable manufacturing variable used to alter its properties.

1.5. Natural Fiber Reinforced Composites

The interest in natural fiber-reinforced polymer composite materials is rapidly growing both in terms of their industrial applications and fundamental research. They are renewable, cheap, completely or partially recyclable, and biodegradable. Plants, such as flax, cotton, hemp, jute, sisal, kenaf, pineapple, ramie, bamboo, banana, etc., as well as wood, used from time immemorial as a source of lignocellulosic fibers, are more and more often applied as the reinforcement of composites. Their availability, renewability, low density, and price as well as satisfactory mechanical properties make them an attractive ecological alternative to glass, carbon and man-made fibers used for the manufacturing of composites. The natural fiber-containing composites are more environmentally friendly, and are used in transportation (automobiles, railway coaches, aerospace), military applications, building and construction industries (ceiling paneling, partition boards), packaging, consumer products, etc.

1.6. Classification of Natural Fibers

Fibers are a class of hair-like material that are continuous filaments or are in discrete elongated pieces, similar to pieces of thread. They can be spun into filaments, thread, or rope. They can be used as a component of composites materials. They can also be matted into sheets to make products such as paper or felt. Fibers are of two types: natural fiber and man made or synthetic fiber. Figure 1 shows the classification of natural fibers.

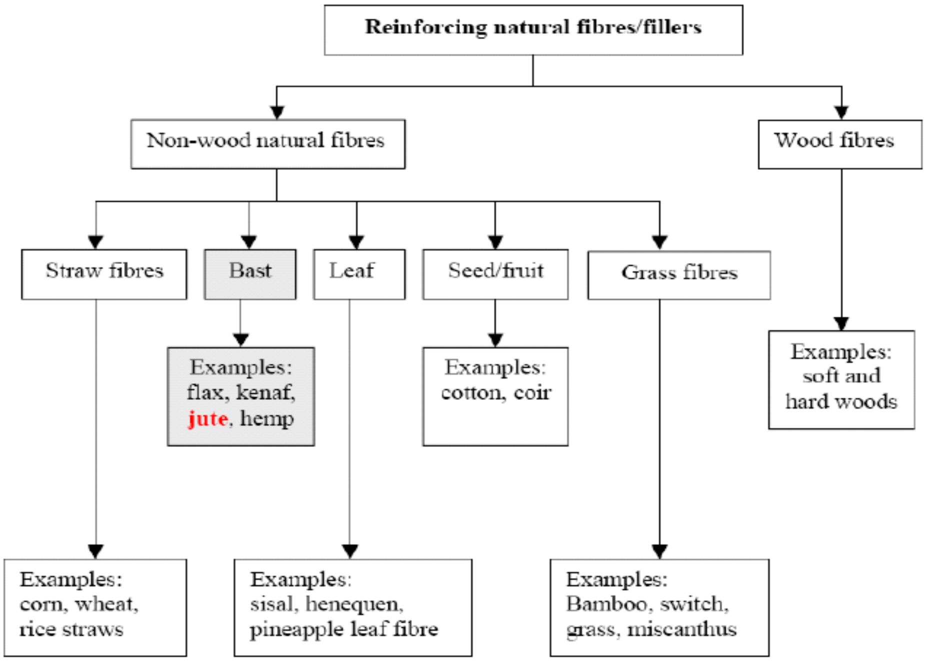

Figure 1. Classification of natural fibers which can be used as reinforcement of polymer[17]

Natural fibers include those made from plant, animal and mineral sources. Natural fibers can be classified according to their origin.

- Animal fiber
- Mineral fiber
- Plant fiber

Animal Fiber

Animal fiber generally comprise proteins; examples mohair, wool, silk, alpaca, angora.

Animal hair (wool or hair): Fiber taken from animals or hairy mammals. E.g. Sheep's wool, goat hair (cashmere, mohair), alpaca hair, horse hair, etc.

Silk fiber: Fiber collected from dried saliva of bugs or insects during the preparation of cocoons. Examples include silk from silk worms.

Avian fiber: Fibers from birds, e.g. feathers and feather fiber.

Mineral fiber

Mineral fibers are naturally occurring fiber or slightly modified fiber procured from minerals. These can be categorized into the following categories:

Asbestos: The only naturally occurring mineral fiber. Varietions are serpentine and amphiboles, anthophyllite.

Ceramic fibers: Glass fibers (Glass wood and Quartz), aluminum oxide, silicon carbide, and boron carbide.

Metal fibers: Aluminum fibers

Plant fiber

Plant fibers are generally comprised mainly of cellulose: examples include cotton, jute, flax, ramie, sisal and hemp. Cellulose fibers servers in the manufacture of paper and cloth. This fiber can be further categorizes into following.

Seed fiber: Fibers collected from the seed and seed case e.g. cotton and kapok.

Leaf fiber: Fibers collected from the leaves e.g. sisal and agave.

Skin fiber: Fibers are collected from the skin or bast surrounding the stem of their respective plant. These fibers have higher tensile strength than other fibers. Therefore, these fibers are used for durable yarn, fabric, packaging, and paper. Some examples are flax, jute, banana, hemp, and soybean.

Fruit fiber: Fibers are collected from the fruit of the plant, e.g. coconut (coir) fiber.

Stalk fiber: Fibers are actually the stalks of the plant. E.g. straws of wheat, rice, barley, and other crops including bamboo and grass. Tree wood is also such a fiber.

The natural fibers can be used to reinforce both thermosetting and thermoplastic matrices. Thermosetting resins, such as epoxy, polyester, polyurethane, phenolic, etc. are commonly used today in natural fiber composites, in which composites requiring higher performance applications. They provide sufficient mechanical properties, in particular stiffness and strength, at acceptably low price levels. Considering the ecological aspects of material selection, replacing synthetic fibers by natural ones is only a first step. Restricting the emission of green house effect causing gases such as CO_2 into the atmosphere and an increasing awareness of the finiteness of fossil energy resources are leading to developing new materials that are entirely based on renewable resources.

1.7. Applications of Natural Fiber Composites

The natural fiber composites can be very cost effective material for following applications:

- Building and construction industry: panels for partition and false ceiling, partition boards, wall, floor, window and door frames, roof tiles, mobile or pre-fabricated buildings which can be used in times of natural calamities such as floods, cyclones, earthquakes, etc.
- Storage devices: post-boxes, grain storage silos, bio-gas containers, etc.
- Furniture: chair, table, shower, bath units, etc.
- Electric devices: electrical appliances, pipes, etc.
- Everyday applications: lampshades, suitcases, helmets, etc.
- Transportation: automobile and railway coach interior, boat, etc.
- Toys

The reasons for the application of natural fibers in the automotive industry include:

- Low density: which may lead to a weight reduction of 10 to 30%?
- Acceptable mechanical properties, good acoustic properties.
- Favorable processing properties, for instance low wear on tools, etc.
- Options for new production technologies and materials.
- Favorable accident performance, high stability, less splintering.
- Favorable ecobalance for part production.
- Favorable ecobalance during vehicle operation due to weight savings.
- Occupational health benefits compared to glass fibers during production.
- No off-gassing of toxic compounds (in contrast to phenol resin bonded wood and recycled Cotton fiber parts).
- Reduced fogging behavior.
- Price advantages both for the fibers and the applied technologies.

Applications of natural fiber reinforcement for automotive parts are shown in Figure 2.

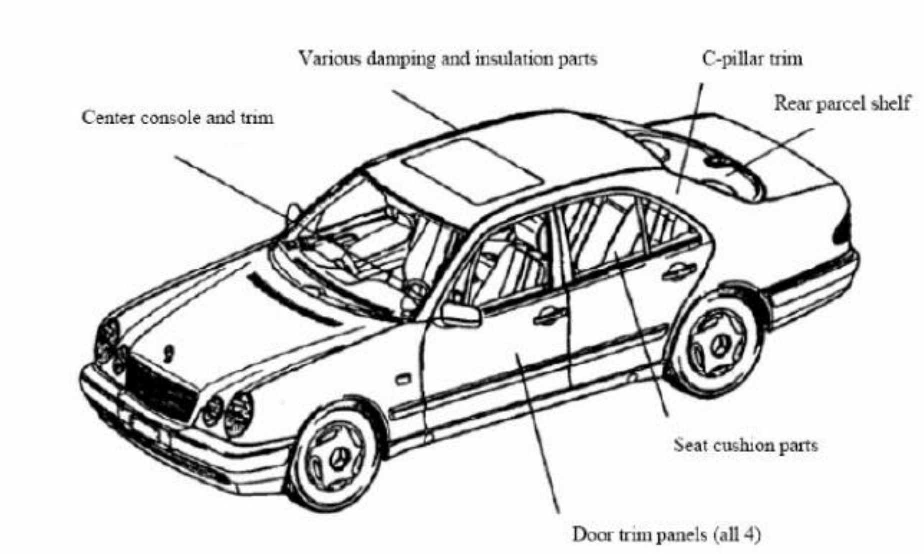

Figure 2. Plant fiber applications in the automobile sector.[35]

1.8. Advantages of Natural Fiber Composites

The main advantages of natural fiber composite are:

- Low specific weight, resulting in a higher specific strength and stiffness than glass fiber.
- It is a renewable source, the production requires little energy, and CO2 is used while oxygen is given back to the environment.
- Producible with low investment at low cost, which makes the material an interesting product for low wage countries.
- Reduced wear of tooling, healthier working condition, and no skin irritation.
- Thermal recycling is possible while glass causes problem in combustion furnaces.
- Good thermal and acoustic insulating properties.

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Chapter 2

LITERATURE SURVEY

2. LITERATURE SURVEY

This chapter outlines some of the recent reports published in literature on composites with special emphasis on erosion wear behavior of glass fiber reinforced polymer composites.

As a result of the increasing demand for environmentally friendly materials and the desire to reduce the cost of traditional fibers (i.e., carbon, glass and aramid) reinforced petroleum-based composites, new bio-based composites have been developed. Researchers have begun to focus attention on natural fiber composites (i.e., biocomposites), which are composed of natural or synthetic resins, reinforced with natural fibers. Natural fibers exhibit many advantageous properties, they are a low-density material yielding relatively lightweight composites with high specific properties. These fibers also offer significant cost advantages and ease of processing along with being a highly renewable resource, in turn reducing the dependency on foreign and domestic petroleum oil. Recent advances in the use of natural fibers (e.g., flax, cellulose, jute, hemp, straw, switch grass, kenaf, coir and bamboo) in composites have been reviewed by several authors [6–25].

Harish et al. [2] developed coir composite and mechanical properties were evaluated. Scanning electron micrographs obtained from fracture surfaces were used for a qualitative evaluation of the interfacial properties of coir /epoxy and compared with glass fibers. Wang and Huang [1] had taken a coir fiber stack, characters of the fibers were analyzed. Length of the fibers was in the range between 8 and 337 mm. The fibers amount with the length range of 15~145 mm was 81.95% of all measured fibers. Weight of fibers with the length range of 35~225 mm accounted for 88.34% of all measurement. The average fineness of the coir fibers was 27.94 tex. Longer fibers usually had higher diameters. Composite boards were fabricated by using a heat press machine with the coir fiber as the reinforcement and the rubber as matrix. Tensile strength of the composites was investigated. Nilza et al. [3] use three Jamaican natural cellulosic fibers for the design and manufacture of composite material. They took bagasse from sugar cane, banana trunk from banana plant and coconut coir from the coconut husk. Samples were subjected to standardized tests such as ash and carbon content, water absorption, moisture content, tensile strength, elemental analysis and chemical analysis. Bilba et al. [4] examined Four fibers from banana-trees (leaf, trunk) and coconut-tree (husk, fabric) before their incorporation in cementitious matrices, in order to prepare insulating material for construction. Thermal degradation of these fibers was studied between 200 and 700 °C under nitrogen gas flow. Temperature of pyrolysis was the experimental parameter investigated. The solid residues obtained were analyzed by classical elemental analysis, Fourier Transform Infra Red (FTIR) spectroscopy and were observed by Scanning Electron Microscopy (SEM). This study has shown (1) the relation between botanical, chemical composition with both localization of fiber in the tree and type of tree; (2) the rapid and preferential decomposition of banana fibers with increasing temperature of pyrolysis and (3) the rough samples are made of hollow fiber. Conrad [5] investigates the connection between the distribution of lignin and pectin and the loading of Pb and Zn on coir. The coir consisted mainly of xylem and a fiber sheath. The lignin was evenly distributed in the cell walls of the fiber sheath, but in the xylem, there was no detectable content in the compound middle lamella, and a smaller content of lignin in the secondary walls than in the walls of the fiber sheath. The only detectable content of pectin in the fiber sheath walls was in the middle lamella, cell corners and extracellular matrix, while in the xylem, the pectin was almost evenly distributed in the wall, with a higher concentration in the middle lamella and cell corners. All cell walls facing the lacuna had a high content of pectin. Simple correlation between the loading of metal ions and the distribution of lignin or pectin, these investigations point at no correlation with lignin and a positive correlation with pectin. Passipoularidis and Philippidis [6] studied the influence of damage accumulation metric, constant life diagram formulation and cycle counting method on life prediction schemes for composite materials under variable amplitude (VA) loading. Results indicate that a net improvement is achieved when linear strength degradation is implemented as damage metric in life prediction schemes, over the state-of-the-art PM summation. Din et al. [7] investigated the liquid-phase adsorption of phenol onto coconut shell-based activated carbon for its equilibrium studies and kinetic modeling. Coconut shell was converted into high quality activated carbon through physiochemical activation at 850 ◦C under the influence of CO2 flow. Beforehand, the coconut shell was carbonized at 700 ◦C and the resulted char was impregnated with KOH at 1:1 weight ratio. A series of batch adsorption experiments were conducted with initial phenol concentrations ranging from 100 to 500mgl^{-1}, adsorbent loading of 0.2 g and the adsorption process was maintained at 30±1 ◦C. Chemical reaction was found to be a rate-controlling parameter to this phenol-CS850A batch adsorption system due to strong agreement with the pseudo-second-order kinetic model. Adsorption capacity for CS850A was found to be 205.8mgg−1. Rao et al. [8] aims at introducing new natural fibers used as fillers in a polymeric matrix enabling production of economical and lightweight composites for load carrying structures. An investigation of the extraction procedures of vakka, date and bamboo fibers has been undertaken. The cross-sectional shape, the density and tensile properties of these fibers, along with established fibers like sisal, banana, coconut and palm, are determined experimentally under similar conditions and compared. The fibers introduced in the present study could be used as an effective reinforcement for making composites, which have an added advantage of being lightweight. Dick et al. [9] conduct static and cyclic 4-point bending tests on glass-filled polycarbonate, to collect results for evaluation of a theoretical model on its capability to predict the fatigue life and the residual strength after the cyclic loading The study quantifies the effects of loading conditions, i.e. the stress ratio and the maximum stress level, on the damage development. The paper demonstrates the possibility of expressing each of the model parameters as a function of single variable that is stress ratio, maximum stress level, or a material-dependent constant. Ersoy and Kucuk [10] investigated the sound absorption of an industrial waste, developed during the processing of tea leaves. Three different layers of tea-leaf-fiber waste materials with and without backing provided by a single layer of woven textile cloth were tested for their sound absorption properties. The experimental data indicate that a 1 cm thick tea-leaf-fiber waste material with backing provides sound absorption which is almost equivalent to that provided by six layers of woven textile cloth. Twenty millimeters thick layers of rigidly backed tea-leaf-fibers and non-woven fiber materials exhibit almost equivalent sound absorption in the frequency range between 500 and 3200 Hz. Jacquemin et al [11] proposed an analytical micro-mechanical self-consistent approach dedicated to mechanical states prediction in both the fiber and the matrix of composite structures submitted to a transient hygroscopic load. The time and space dependent macroscopic stresses, at ply scale, are determined by using continuum mechanics formalism. The reliability of the new approach is checked, for carbon–epoxy composites, through a comparison between the local stress states calculated in both the resin and fiber according to the new closed-form solutions and the equivalent numerical model. Wang et al. [12] investigated the effective thermal conductivity enhancement of carbon fiber composites using a three-dimensional numerical method First a more realistic three-dimensional distribution of fibers dispersed in a matrix phase is reproduced by a developed random generation-growth method to eliminate the overrated inter-fiber contacts by the two-dimensional simulations. The energy transport governing equations are then solved through the three-dimensional structures using a high-efficiency lattice Boltzmann scheme. The resultant predictions agree well with the available experimental data. Compared with the existing theoretical models, the present method does not depend upon empirical parameters which have to be determined case by case, so that it is useful for design and optimization for new materials, beyond prediction and analysis just for existing composites. Yetgin et al. [13] studied the compression and tensile tests for five different adobe mixtures. The important part of this study consisted of uniaxial compressive tests done with natural fiber mixtures. Thus, the results obtained from mechanical tests were presented in the form of stress–strain graphs. In addition, mechanical properties were related to the water content for workability, unit weight and fiber contents and discussions were given. The results show that as fiber content increases, compressive and tensile strengths decrease, and shrinkage rates decrease. Rahman et al. [14] studied the surface treatment of the coir fiber and its mechanical properties. Fiber surface modification by ethylene dimethylacrylate (EMA) and cured under UV radiation. Pretreatment with UV radiation and mercerization were done before grafting with a view to improve the physico-mechanical performance of coir fibers. The effects of mercerization on shrinkage and fiber weight losses were monitored at different temperature and alkali concentration. They observed that, fiber shrinkage is higher at low temperature and 20% alkali treated coir fibers yielded maximum shrinkage and weight losses. It was found that higher shrinkage of the polymer grafted fiber showed enhanced physico-mechanical properties. The grafting of alkali treated fiber shows an increase of polymer loading (about 56% higher) and tensile strength (about 27%) than 50% EMA grafted fiber. The fiber surface topology and the tensile fracture surfaces were characterized by scanning electron microscopy and were found improved interfacial bonding to the modified fiber–matrix interface.

A lot of research has been done on natural fiber reinforced polymer composites but research on coconut based polymer composites is very rare. Against this background, the present research work has been undertaken, with an objective to explore the potential of coconut fiber polymer composites and to study the mechanical and wear characterization of different composites.

2.1 Objectives of the Research Work

The objectives of the project are outlined below.

- Fabrication of coconut fibre reinforced epoxy based composite.
- Evaluation of mechanical properties (tensile strength, flexural, hardness, impact strength etc.).
- Besides the above all the objective is to develop new class of composites by incorporating coconut fiber reinforcing phases into a polymeric resin.

Also this work is expected to introduce a new class of polymer composite that might find many engineering applications.

- Erosion analysis of all the composites are studied by using Taguchi experimental design.

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Chapter 3

MATERIALS AND METHODS

CHAPTER 3

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3. MATERIALS AND METHODS

3.1 Introduction

This chapter describes the details of processing of the composites and the experimental procedures followed for their characterization and tribological evaluation. The raw materials used in this work are

1. Coconut Fiber
2. Epoxy resin

3.2 Processing of the Composites

Coconut fibers are reinforced with Epoxy LY 556, chemically belonging to the ‘epoxide’ family which is used as the matrix material. The epoxy resin and the hardener are supplied by Ciba Geigy India Ltd. Red mud collected from NALCO aluminium refinery at Damanjodi. Epoxy resin have modulus of 3.42GPa and possess density 1100 kg/m3. Composites of three different compositions i.e. 30wt%, 40wt% and 50wt% are made. Specimens of suitable dimension are cut for different tests. Figure 3. Specimen of coconut fiber reinforced epoxy composites

Figure 3. Specimen of coconut fiber reinforced epoxy composites

3.3 Characterization of the Composites

Density

The theoretical density of composite materials in terms of weight fraction can easily be obtained as for the following equations given by Agarwal and Broutman [26].

$$\rho_{ct} = \frac{1}{(W_f/\rho_f) + (W_m/\rho_m)} \qquad (1)$$

Where, W and ρ represent the weight fraction and density respectively. The suffix f, m and ct stand for the fiber, matrix and the composite materials respectively.

The composites under this investigation consists of three components namely matrix, fiber and particulate filler. Hence the modified form of the expression for the density of the composite can be written as

$$\rho_{ct} = \frac{1}{(W_f/\rho_f) + (W_m/\rho_m) + (W_p/\rho_p)} \tag{2}$$

Where, the suffix '*p*' indicates the particulate filler materials.

The actual density (ρ_{ce}) of the composite, however, can be determined experimentally by simple water immersion technique. The volume fraction of voids (V_v) in the composites is calculated using the following equation:

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \tag{3}$$

Micro-hardness measurement

Micro-hardness measurement is done using a Leitz micro-hardness tester. A diamond indenter, in the form of a right pyramid with a square base and an angle 136^0 between opposite faces, is forced into the material under a load F. The two diagonals X and Y of the indentation left on the surface of the material after removal of the load are measured and their arithmetic mean L is calculated. In the present study, the load considered F = 24.54N and Vickers hardness number is calculated using the following equation.

$$H_V = 0.1889 \frac{F}{L^2} \tag{4}$$

$$\text{and} \quad L = \frac{X + Y}{2}$$

Where F is the applied load (N), L is the diagonal of square impression (mm), X is the horizontal length (mm) and Y is the vertical length (mm).

Tensile and flexural strength

The tensile test is generally performed on flat specimens. The commonly used specimens for tensile test are the dog-bone type and the straight side type with end tabs. During the test a uni-axial load is applied through both the ends of the specimen. The ASTM standard test method for tensile properties of fiber resin composites has the designation D 3039-76. The length of the test section should be 200 mm. The tensile test is performed in the universal testing machine (UTM) Instron 1195 and results are analyzed to calculate the tensile strength of composite samples. The short beam shear (SBS) tests are performed on the composite samples at room temperature to evaluate the value of flexural strength (FS). It is a 3-point bend test, which generally promotes failure by inter-laminar shear. The SBS test is conducted as per ASTM standard (D2344-84) using the same UTM. Span length of 40 mm and the cross head speed of 1 mm/min are maintained. The flexural strength (F.S.) of any composite specimen is determined using the following equation.

$$\mathrm{F.S} = \frac{3\mathrm{PL}}{2\mathrm{bt}^2} \tag{5}$$

Where, *L* is the span length of the sample. P is the load applied; b and t are the width and thickness of the specimen respectively.

3.4. Scanning electron microscopy

The surfaces of the raw fish scales and the composite specimens are examined directly by scanning electron microscope JEOL JSM-6480LV. The scales are washed, cleaned thoroughly, air-dried and are coated with 100 Å thick platinum in JEOL sputter ion coater and observed SEM at 20 kV. Similarly the composite samples are mounted on stubs with silver paste. To enhance the conductivity of the samples, a thin film of platinum is vacuum-evaporated onto them before the photomicrographs are taken.

3.5. Test Apparatus

Figure 4 shows the schematic diagram of erosion test rig confirming to ASTM G 76. The set up is capable of creating reproducible erosive situations for assessing erosion wear resistance of the prepared composite samples. It consists of an air compressor, an air particle mixing chamber and an accelerating chamber. Dry compressed air is mixed with the particles which are fed at constant rate from a sand flow control knob through the nozzle tube and then accelerated by passing the mixture through a convergent brass nozzle of 3 mm internal diameter. These particles impact the specimen which can be held at various angles with respect to the direction of erodent flow using a swivel and an adjustable sample clip. The velocity of the eroding particles is measured using double disc method. In the present study, dry silica sand (angular) of different particle sizes (300, 400 and 500 μm) is used as erodent. Each sample is cleaned in acetone, dried and weighed to an accuracy of ±0.1 mg using a precision electronic balance. It is then eroded in the test rig for 10 min and weighed again to determine the weight loss. The process is repeated till the erosion rate attains a constant value called steady-state erosion rate. The ratio of this weight loss to the weight of the eroding particles causing the loss is then computed as a dimensionless incremental erosion rate. The erosion rate is defined as the weight loss of the specimen due to erosion divided by the weight of the erodent causing the loss.

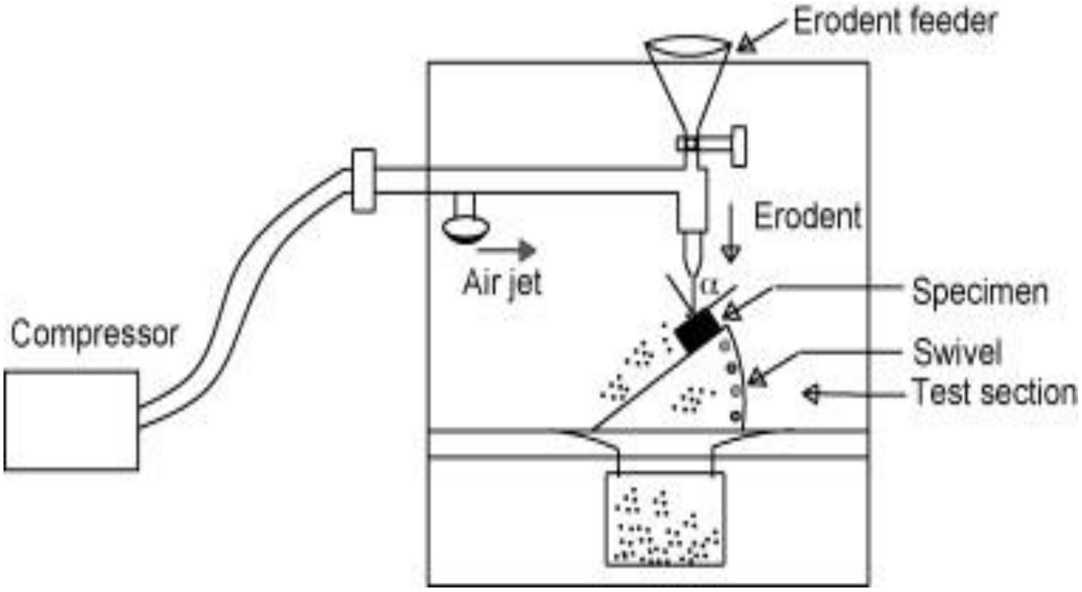

Figure 4. A schematic diagram of the erosion test rig

Chapter 4

COMPOSITE CHARACTERIZATION: RESULTS & DISCUSSION

4.COMPOSITE CHARACTERIZATION: RESULTS AND DISCUSSION

4.1 Introduction

This chapter presents the physical and mechanical characterization of the class of polymer matrix composites developed for the present investigation. They are

- Unidirectional coconut fiber reinforced epoxy resin composites.

Details of processing of these composites and the tests conducted on them have been described in the previous chapter. The results of various characterization tests are reported here. They include evaluation of tensile strength, flexural strength, measurement of density and micro-hardness has been studied and discussed.

4.2 Composite Characterization

Mechanical properties

Figure 5 shows the micro-hardness values for different compositions. It is seen that with the increase in fiber content in the composite, its hardness value improves although the increment is marginal. Figures 6 and 7 show the variation of tensile and flexural strengths of the composites with the fiber content. A gradual increase in tensile strength as well as flexural strength with the weight fraction of fiber is noticed. It clearly indicates that inclusion of glass fiber improves the load bearing capacity and the ability to withstand bending of the composites. Similar observations have been reported by Harsha et al. [25] for other fiber reinforced thermoplastics such as polyaryletherketone composites. It may be mentioned here that both tensile and flexural strengths are important for recommending any composite as a candidate for structural applications.

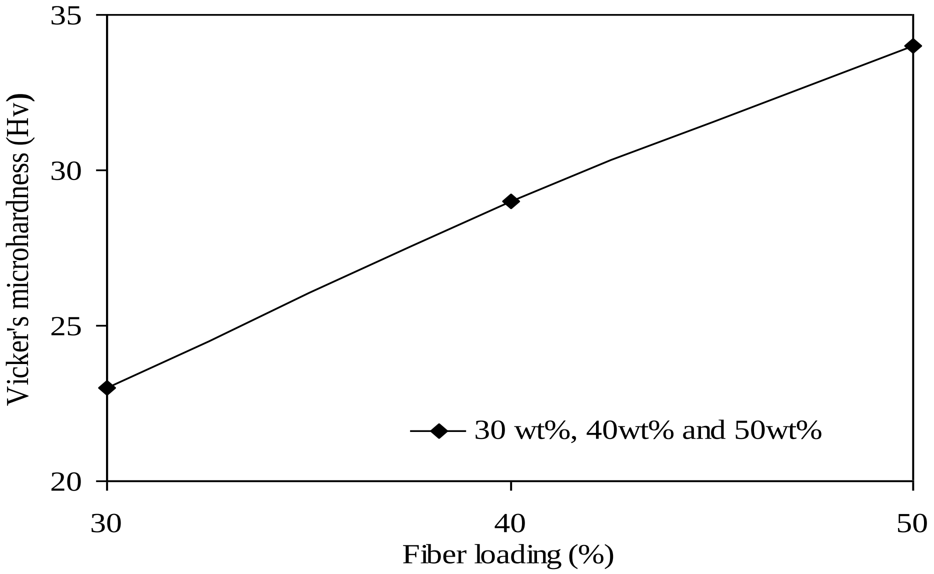

Figure 5. Variations of micro-hardness of the composites with different fiber loading

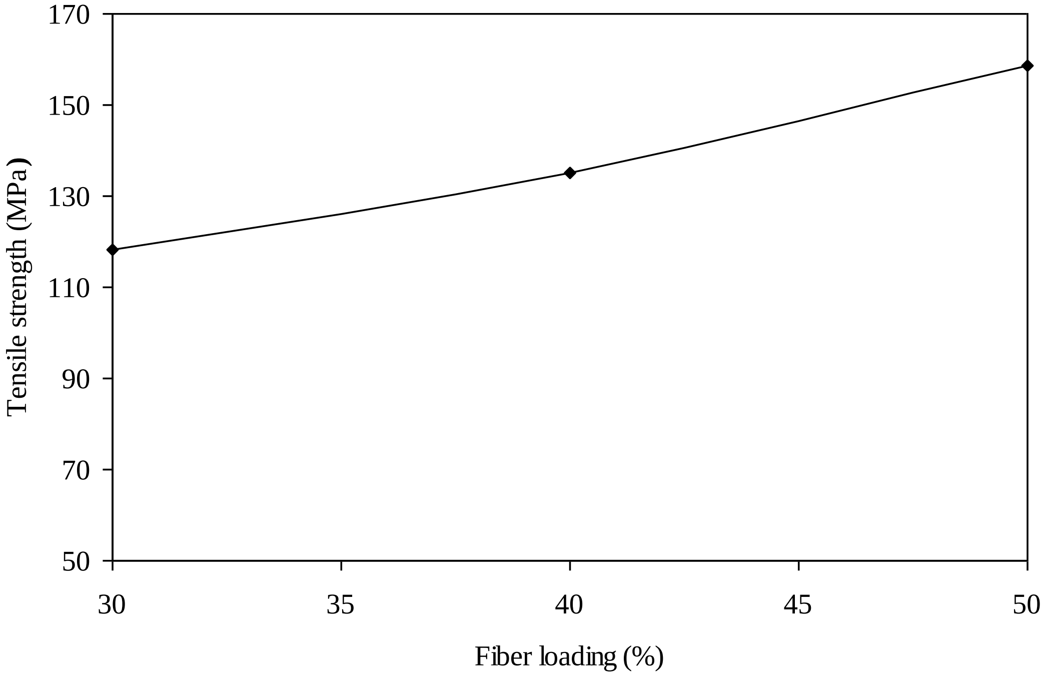

Figure 6. Variations of tensile strength of the composites with fiber loading

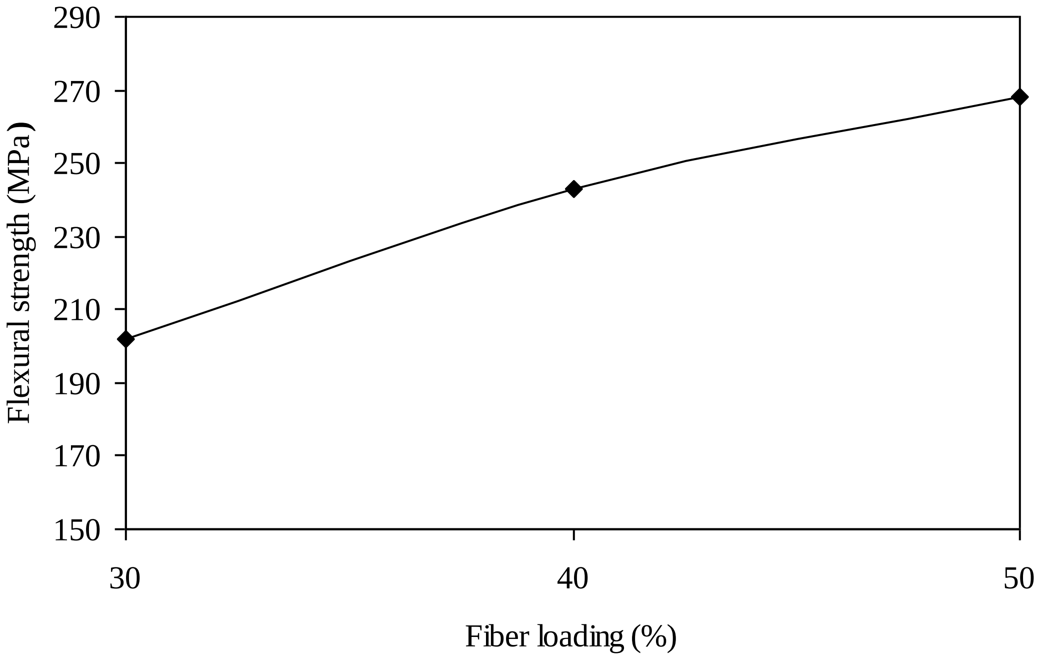

Figure 7. Variations of flexural strength of the composites with fiber loading

4.3. Design of experiments via Taguchi method

The Taguchi method involves reducing the variation in a process through robust design of experiments. The overall objective of the method is to produce high quality product at low cost to the manufacturer. The Taguchi method was developed by Dr. Genichi Taguchi of Japan who maintained that variation. Therefore, poor quality in a process affects not only the manufacturer but also society. He developed a method for designing experiments to investigate how different parameters affect the mean and variance of a process performance characteristic that defines how well the process is functioning. The experimental design proposed by Taguchi involves using orthogonal arrays to organize the parameters affecting the process and the levels at which they should be varied; it allows for the collection of the necessary data to determine which factors most affect product quality with a minimum amount of experimentation, thus saving time and resources. Analysis of variance on the collected data from the Taguchi design of experiments can be used to select new parameter values to optimize the performance characteristic.

The general steps involved in the Taguchi Method are as follows:

1) Define the process objective, or more specifically, a target value for a performance measure of the process. This may be a flow rate, temperature, etc. The target of a process may also be a minimum or maximum; for example, the goal may be to maximize the output flow rate. The deviation in the performance characteristic from the target value is used to define the loss function for the process.
2) Determine the design parameters affecting the process. Parameters are variables within the process that affect the performance measure such as temperatures, pressures, etc. that can be easily controlled. The number of levels that the parameters should be varied at must be specified. For example, a temperature might be varied to a low and high value of 40^oC and 80^oC. Increasing the number of levels to vary a parameter at increases the number of experiments to be conducted.
3) Create orthogonal arrays for the parameter design indicating the number of and conditions for each experiment. The selection of orthogonal arrays will be discussed in considerably more detail.
4) Conduct the experiments indicated in the completed array to collect data on the effect on the performance measure.
5) Complete data analysis to determine the effect of the different parameters on the performance measure.

The most important stage in the design of experiment lies in the selection of the control factors. Therefore, a large number of factors are included so that non-significant variables can be identified at earliest opportunity**.** Exhaustive literature review on erosion behaviour of polymer composites reveal that parameters viz., impact velocity, fiber loading, impingement angle and erodent size etc largely influence the erosion rate of polymer composites. The impact of four such parameters are studied using L_9 (3^4) orthogonal design. The tests are conducted as per experimental design given in Table 1.

The fixed and variable parameters chosen for the test are given in Table 2. The selected levels of the four control parameters are listed in Table 3.

Table 1. Orthogonal array for L9 (3^4) Taguchi design

Experiment Number	**Column**			
	1(A)	**2(B)**	**3(C)**	**4(D)**
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

Table 2. Parameters of the setting

Control Factors	Symbols	Fixed parameters	
Velocity of impact	Factor A	Erodent	Silica sand
Fiber loading	Factor B	Erodent feed rate (g/min)	10.0 ± 1.0
Impingement angle	Factor C	Nozzle diameter (mm)	3
Erodent size	Factor D	Length of nozzle (mm)	80

Table 3. Levels for various control factors

Control factor	Level			
	I	II	III	units
A: Impact velocity	43	54	65	m/sec
B: Fiber loading	30	40	50	%
D:Impingement angle	30	60	90	degree
E: Erodent size	300	400	500	µm

In Table 2, each column represents a test parameter whereas a row stands for a treatment or test condition which is nothing but combination of parameter levels. In conventional full factorial experiment design, it would require $3^4 = 81$ runs to study five parameters each at three levels whereas, Taguchi's factorial experiment approach reduces it to only 9 runs offering a great advantage in terms of experimental time and cost. The experimental observations are further transformed into signal-to-noise (S/N) ratio. There are several S/N ratios available depending on the type of performance characteristics. The S/N ratio for minimum erosion rate can be expressed as "lower is better" characteristic, which is calculated as logarithmic transformation of loss function as shown below.

Smaller is the better characteristic: $\frac{S}{N} = -10 \log \frac{1}{n} \left(\sum y^2 \right)$ (6)

Where ‘n’ the number of observations and y the observed data. The standard linear graph, as shown in Figure 8, is used to assign the factors and interactions to various columns of the orthogonal array [21].

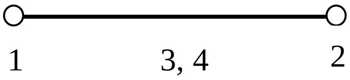

Figure 8. Linear Graph for L_9 orthogonal array

The plan of the experiments is as follows: the first column of this orthogonal array is assigned to impact velocity (A), the second column to fiber loading (B), the third column to impingement angle (C) and fourth column to erodent size (D) respectively.

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Chapter 5

RESULTS & ANALYSIS

CHAPTER 5

5.0 RESULTS AND ANALYSIS

The experimental results are analyzed using Taguchi method and the significant parameters affecting material erosion have been identified. The results of the Taguchi analysis are also presented here.

5.1. Taguchi experimental analysis

From Table 4, the overall mean for the S/N ratio of the erosion rate is found to be -48.73 db. Figure 6 shows graphically the effect of the four control factors on erosion rate. The analysis is made using the popular software specifically used for design of experiment applications known as MINITAB 14. Before any attempt is made to use this simple model as a predictor for the measures of performance, the possible interactions between the control factors must be considered. Thus factorial design incorporates a simple means of testing for the presence of the interaction effects. Analysis of the result leads to the conclusion that factor combination of A_2, B_2, C_1 and D_3 gives minimum erosion rate. As for as minimization of erosion rate is concerned, factors A, C and D have significant effect whereas factor B has least effect as shown in Table 5. It is also observed from Figure 9 that the significant level of each factor for minimization of erosion rate.

Table 4. Experimental design using L_9 orthogonal array

Sl. No.	A	B	C	D	Er (mg/kg)	S/N ratio (db)
1	43	30	30	65	255.25	-48.1393
2	43	40	60	75	288.86	-49.2137
3	43	50	90	85	249.80	-47.9518
4	54	30	60	85	255.25	-48.1393
5	54	40	90	65	239.76	-47.5955
6	54	50	30	75	249.18	-47.9303
7	65	30	90	75	298.23	-49.4910
8	65	40	30	85	261.17	-48.3385
9	65	50	60	65	364.31	-51.2294

Table 5. Response Table for Signal to Noise Ratios

Level	A	B	C	D
1	-48.43	-48.59	-48.14	-48.99
2	-47.89	-48.38	-49.53	-48.88
3	-49.69	-49.04	-48.35	-48.14
Delta	1.80	0.65	1.39	0.84
Rank	1	4	2	3

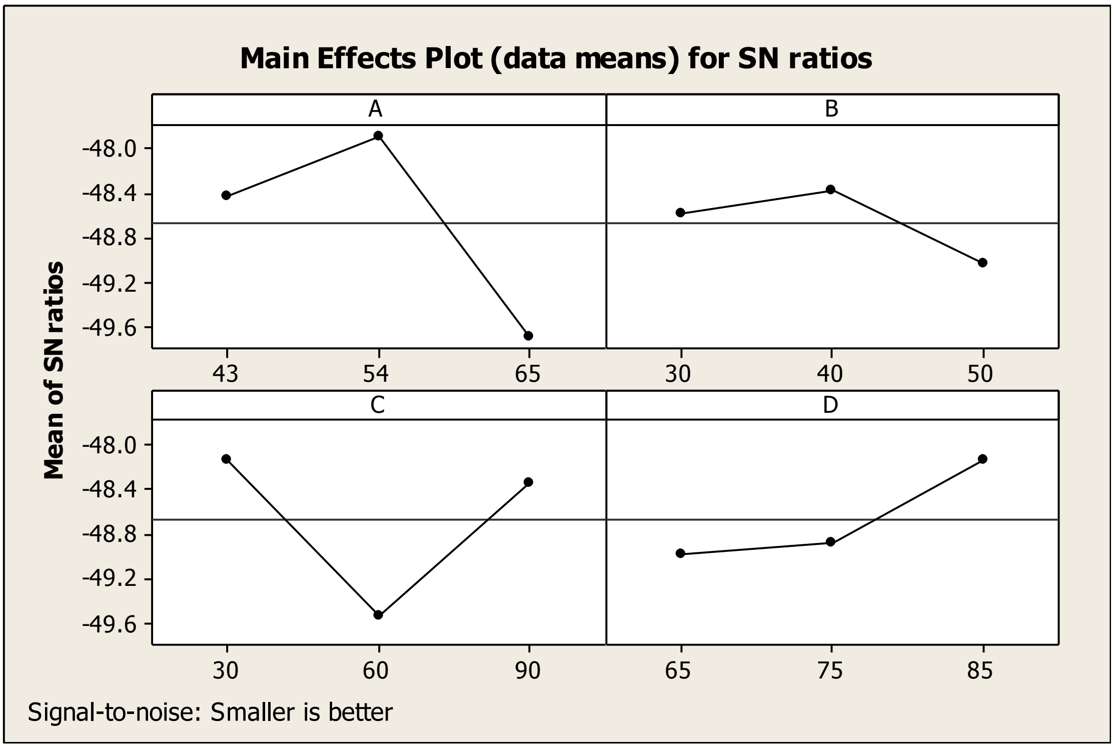

Figure 9. Effect of control factors on erosion rate

5.2. Surface morphology of the composites

To identify the mode of material removal, the morphologies of eroded surfaces are observed under scanning electron microscope. Figure 10 shows the local removal of resin material from the impacted surface resulting in exposure of the fibers to the erodent flux. This micrograph also reveals that due to sand particle impact on fibers there is formation of transverse cracks that break these fibers. Figure 11 presents the microstructure of the composite eroded at high impact velocity (65m/sec) and at an impingement angle of 60^0. Here the propagation of crack along transverse as well as longitudinal direction is well visualized. On comparing this micro-structure with that of the same composite eroded at a lower impact velocity (43m/s) and higher impingement angle (90^0), it can be seen that in the second case the breaking of coconut fibers is more prominent (Figure 11). It appears that cracks have grown on the fibers giving rise to breaking of the fibers into small fragments. Further the cracks have been annihilated at the fiber matrix interface and seem not to have penetrated through the matrix. Change in impact angle from oblique to normal changes the topography of the damaged surface very significantly. Figure 12 shows the dominance of micro-chipping and micro-cracking phenomena. It can be seen that multiple cracks originate from the point of impact, intersect one another and form wear debris due to brittle fracture in the fiber body. After repetitive impacts, the debris in platelet form are removed and account for the measured wear loss. The occurrence of peak erosion rate at 60^0 impact is understandable. In this case, both abrasion and erosion processes play important roles. The sand particles after impacting, slide on the surface and abrade while dropping down. The wear and subsequently the damage are therefore more than that in the case of normal impact. Marks of micro-ploughing on the ductile polyester matrix region seen in Figure 8 support this argument.

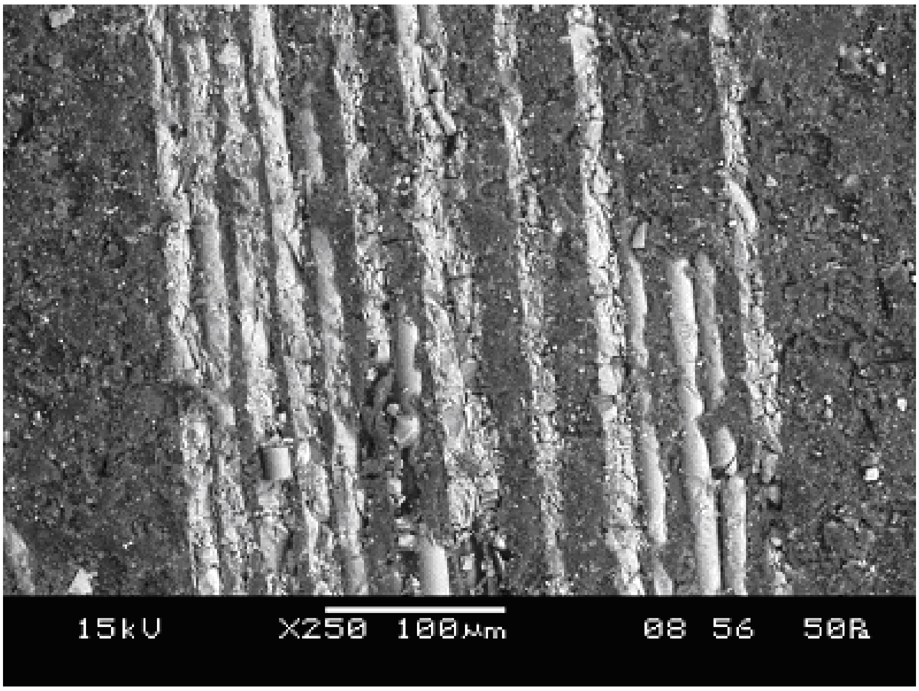

Figure 10. SEM micrograph (X 250) of coconut fiber epoxy resin composite eroded surface (impact velocity 65 m/sec, fiber loading 50%, impingement angle 60^0 and erodent size 300µm).

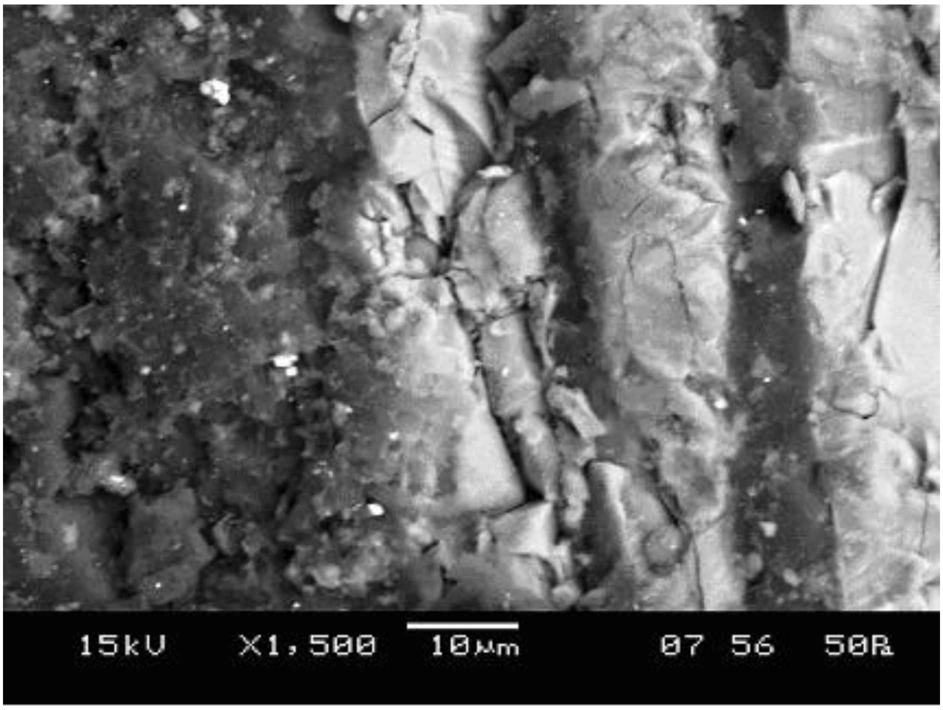

Figure 11. SEM micrograph (X 1000) of GF Polymer composite eroded surface (impact velocity 65 m/sec, fiber loading 50%, impingement angle 60^0 and erodent size 300µm)

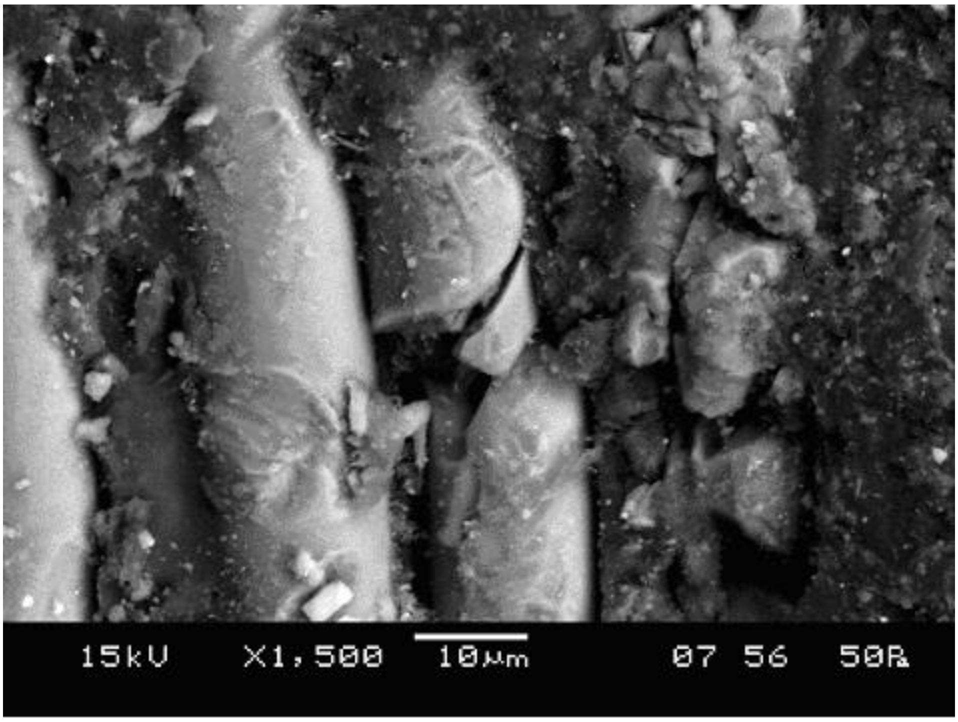

Figure 12. SEM micrograph (X 1000) of GF Polymer composite eroded surface (impact velocity 43 m/sec, fiber loading 50%, impingement angle 90^0 and erodent size 500µm)

5.3. ANOVA and the effects of factors

In order to understand a concrete visualization of impact of various factors and their interactions, it is desirable to develop analysis of variance (ANOVA) table to find out the order of significant factors as well as interactions. Table 6 shows the results of the ANOVA with the erosion rate. This analysis was undertaken for a level of confidence of significance of 5 %. The last column of the table indicates that the main effects are highly significant (all have very small p-values).

From Table 6, one can observe that the impact velocity (p=0.300), erodent size (p=0.466) and fiber loading (p=0.692) have great influence on erosion rate.

Table 6. ANOVA table for erosion rate

Source	DF	Seq SS	Adj SS	Seq MS	F	P
A	1	2.349	2.349	2.349	1.42	0.300
B	1	0.300	0.300	0.300	0.18	0.692
C	1	0.066	0.066	0.066	0.04	0.851
D	1	1.071	1.071	1.071	0.65	0.466
Error	4	6.625	6.625	1.656		
Total	8	10.411				

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Chapter 6

CONCLUSIONS

6. CONCLUSIONS

- This work shows that successful fabrication of a coconut fiber reinforced epoxy composites by simple hand lay-up technique.
- Solid particle erosion characteristics of these composites can be successfully analyzed using Taguchi experimental design scheme. Taguchi method provides a simple, systematic and efficient methodology for the optimization of the control factors. This approach not only needs engineering judgment but also requires a rigorous mathematical model to obtain optimal process settings.
- The results indicate that impact velocity, erodent size and fiber loading are the significant factors in a declining sequence affecting the erosion wear rate.
- The composites exhibit semi-ductile erosion characteristics with the peak erosion wear occurring at 60^0 impingement angle. This nature has been explained by analyzing the possible damage mechanism with the help of SEM micrographs. It is concluded that the inclusion of brittle fibers in ductile polyester matrix is responsible for this semi-ductility.

6.1. Scope for Future Work

- This study leaves wide scope for future investigations. It can be extended to newer composites using other reinforcing phases and the resulting experimental findings can be similarly analyzed.
- Tribological evaluation of coconut fiber reinforced epoxy resin composite has been a much less studied area. There is a very wide scope for future scholars to explore this area of research. Many other aspects of this problem like effect of fiber orientation, loading pattern, weight fraction of ceramic fillers on wear response of such composites require further investigation.

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