Synthesis and Characterization of PVA Biocomposites

A major project report submitted in partial fulfilment for the award of the degree of

Master of Technology

in

Polymer Technology

Submitted By

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Roll No - 2k12/PTE/10

Under the guidance of Prof. D.Kumar



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Certificate

This is to certify that M.Tech major project entitled **"Synthesis and Characterization of PVA Biocomposites"** has been submitted by Poonam in partial fulfilment of the requirements of the award of the degree of M.Tech (Polymer Technology) to the department of Applied Chemistry and Polymer Technology, Delhi Technological University. It is a record of students own work carried out by her under my guidance and supervision.

To the best of my knowledge and belief the content there in is her own original work and has not been submitted for the award of any other degree or diploma in any other college/university.

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DECLARATION

I POONAM YADAV hereby declares that the thesis entitled "Synthesis and Characterization of PVA Biocomposites" is an authentic rcord of research work done by me under the supervision of Dr D kumar , professor Delhi technological university, delhi. This work has not been submitted for the award of any other degree or diploma in any other college/university.

Date:01/08/2016

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(Poonam Yadav)

Abstract

Recently extensive studies have been carried out on natural fibres reinforced thermoplastics due to their low cost , high strength properties, simple fabrication methods, biodegradable nature and diverse application touching all commercial sectors. The present work reveals the effect of various natural fibres in a Bio-composite material. In this research work the effect of incorporation of small amounts (10%) of wood saw dust, wheat bran, rice husk and bagasse in thermoplastic were used to prepare the samples and mechanical characteristics of the prepared Bio-composite samples were analysed .The work shows the possibility of using biocomposite fibres , regarded as waste material, in producing the highest mechanical properties. The tensile strength of composite was measured with a universal testing machine in accordance with the ASTM standards. The results demonstrated that the saw dust and bagasse incorporated fibres shows significant improvement in mechanical properties, especially their tensile strength

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Introduction

1.1 INTRODUCTION

Natural fibres being renewable and amply accessible in many resources, have successfully grabbed the attention of many researchers. They are considered as a substitute material in replacing the inorganic fillers and fibers because of the serious environmental issues. Natural fibers are organically occurring polymers in our environment which are present in grasses, leaves, stalks of plants or even animals. In the composite industry, these are mentioned as plant fibers and further bifurcated into wood or non-wood sources. Lignin and cellulose have been used as main component in the structure of natural fibres composite so they are denoted as lignocellulose.

Apart from natural fibers, agro-wastes such as rice husks, wastes from rubber plants, cocoa cultivation, sugar cane cultivation and oil palm cultivation have also been extensively studied. In Malaysia, the palm oil industry is one of the biggest biomass producers. As per estimate a total of 17 million tons of empty fruit bunches waste are produced annually. The wastes from palm oil, i.e., excess fiber, empty fruit bunches and shell has been utilized on-site to provide energy for the mill and electricity exports to the grid. For low pressure systems with an assumed conversion rate of 2.5 kg of palm oil waste per kWh, potentially 7000 GW h can be generated [1].

Additionally, the empty fruit bunches have found alternative uses such as fibreboard in furniture making, eventually reducing the waste at palm oil mills. Further, in the making of nanocomposites, previous studies have explicitly exhibited that nanocellulose could be extracted from agro-wastes and can be used as nano-reinforcement in various polymer matrices [2]. Promoting the usage of agro-wastes in composites will help to solve part of the global agriculture refuse problem as huge loads of wastes could be transformed into affordable value-added products which make them more valuable for wider applications commercially. Besides, they are renewable, cheap and biodegradable.

The attributes of natural fibers have resemblance to the nature of cellulose and its crystallinity assets. For example, impressive specific mechanical properties of fibres are due to high cellulose content but it tend to be more flammable than those with higher hemicellulose content. Fibers with higher hemicellulose content tend to absorb more moisture and char formation is generally better with fibers that have higher lignin content as they experience degradation at relatively lower temperatures [3, 4]. For reinforcement purposes, natural fibers are employed for the extraction of cellulose which is further used to produce composites owing to its hierarchical structure and semi crystalline nature [5]. Despite their merits, the major hurdle of natural fibers is the apparent struggle to render them into the preferred form or film as they are incapable of being melted or dissolved into a common solvent owing to the strong intermolecular hydrogen bonding, high amount of polymerization, and elevated crystallinity degree [6].

In accordance with the entirely green environmental policies, it is desirable to combine the natural fibers with biodegradable polymers such as polyvinyl alcohol (PVA) to yield ecosustainable composites. PVA is a commonly used polymer that holds certain significant characteristics such as water solubility, efficient usability, the property of film-forming and biodegradability. The worldwide manufacturing of PVA is about 650,000 tons per annum [8]. Polyvinyl alcohol has been extensively employed for preparing blends and composites with quite a few natural, renewable polymers like chitosan, nanocellulose, starch or lignocellulosic fillers.

The injurious consequences of our throwaway customer lifestyle, on the environment are very apparent and also, expressively documented and as a preventive measure, the government is progressively presenting regulations to normalise the detrimental effects on environment resultant of the supplies used in the production of countless of common place products. The composite materials ought to be able to be recycled, reprocessed or biodegradable, to diminish its effect on the environment. Also, the source of the resources ought to be sustainable and renewable. Plant based fiber is sustainable in its stock and it is also aptly biodegradable.

PVA is an example of biodegradable matrix polymers employed extensively in mulch films. These polymers have a decent potential as biodegradable matrices in eco-friendly composites when compared to carbon fibers mixtures or any other non-biodegradable, recyclable fillers. For many ground-breaking and ecofriendly manufacturers, composite consists of PVA, a biopolymer, with natural fibers, which shall furthermore increase PVA biodegradability and physical attributes, is the right selection for ecosustainable materials. A typical to the majority of polymers, PVA is not capable of undergoing polymerization from its own monomer, but it can do so via polyvinyl acetate (PVAc) due to the instability of vinyl monomer. Furthermore, PVA can only be acquired through the process of saponification from PVAc or alcoholysis by reacting PVAc with methanol [9,10]. PVA can also be hydrolyzed from PVAc into two distinct grades, wholly or partly hydrolyzed based on their function. The amount of hydrolysis directs the amount of remaining acetate groups that are there in the polymer in which saponification or alcoholysis has not happened [11]. The quantity of hydrolysis will ultimately influence the attributes of PVA which is also inclusive of its solubility [12].

1.2 PVA/Natural Fiber Composite

The mixture of PVA along with natural fibers leads to the formation of eco-sustainable composites. Specifically, the nanocomposites have become highly popular around the world owing to their exceptional microstructure and superior attributes. The inter-relationship of reinforcement-matrix, the dispersal of reinforcement in matrix, and the attributes of matrix and reinforcement are the four integral aspects that can influence the effectiveness of polymer composites. The existence of –OH groups in the natural fibers and their hydrophilic character are well-matched with PVA. Fine interaction involving fibers and PVA has been seen in a lot of micrographs, consequently, leading to good composite attributes and acceptable performance. The green composite based on continuous and unidirectional bamboo fibers reinforced PVA composites showed good fiber/matrix bonding and showed high tensile strength and Young's modulus with the highest fiber substance. Their mechanical attributed were also well calculated from the rule of mixture. However, a few attributes and processing inadequacies of PVA/natural fibers composites have also been reported. With certain treatments and modifications, these composites could be viewed with a transformed curiosity as advanced materials in the future.

In the basic manufacturing of natural fibers composites, the natural fibers are typically gathered, dried out, ground and sieved to acquire the required size before processing. The

typical processing methodology to set up natural fiber composites is injection moulding [13]. One of the demerits of natural fibers is their low thermal stability. It is preferred to process the natural fiber based composites at low temperature usually limited to 200° C with a relatively shorter processing time [14]. At lower processing temperatures, the degradation of natural fibers can be successfully prevented. Cinelli et al. [13] discovered that compounding of PVA/corn fibers could be attained at low temperatures ($<170^{\circ}$ C) in the occurrence of a plasticizer. Additionally, the fibers' decay can be prevented whilst processing. Adding of high loading plasticizer decreases the melt viscosity throughout extrusion which is pointed out by a lessening of die pressure. Lower melt viscosity implies an effortless processing of composites. The certain specific plasticizers reported by Cinelli et al. were polyethylene glycol (PEG), pentaerythritol and glycerol. The use of plasticizer gives way to uniform and flexible PVA/corn fibers composites. PEG and glycerol have been typically used in PVA. The addition of PEG plasticizer depressed the melting temperature, elastic modulus and also tensile strength of composites, while increasing the elongation at break, thus stirring up plastic conduct to the composites [15]. Also, glycerol content in the PVA was significant to forecast the decisive tensile strength of PVA based composites [16]. Glycerol was found to have a more weighty influence on the overall mechanical properties of the composite, indicating that the glycerol is a more effective plasticizer than the others. Cinelli et al. also pointed out that water is a superior plasticizer than pentaerythritol as water may be capable of a plasticization effect over the composites. Some researchers added both urea and glycerol as plasticizers in PVA/lignocellulose fiber based composites for mulch films purposes. Thermogravimetric analysis (TGA) has evidenced a small decline of the commencement temperature to be at 168° C, demonstrating the lessening of thermal stability due to early decay of low molecular weight components such as urea and glycerol. In a biodegradation study done by Imam et al. [17], it was viewed that the amount of CO₂ discharged from the glycerol and urea had surpassed the worth corresponding to 100% mineralization, pointed out that these additives perhaps possibly may be the potential carbon and nitrogen sources for the microorganisms which enhanced the mineralization capacity of the soil. Similar findings were also accounted by Cinelli et al. [18]. Certainly, the usage of plasticizers in polymer composite lends a certain number of advantages to the polymers whilst concurrently sacrificing some of the attributes. Plasticizer selection must be fundamentally based on its effectiveness in terms of essential quantity, companionability with polymer and its permanency. Due to some apprehension about ecological defence, natural based plasticizers are de facto purported to be the preference for the future. In fact, various types of natural based plasticizers have been well developed and studied.

1.3 COMPOSITE MATERIALS

A composite material consists of two phases mainly as is known to all. It consists of one or more discontinuous phases embedded in a continuous phase [43]. 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. The matrix is composed of three basic material types, i.e., polymers, metals or ceramics. The matrix forms the bulk form of the product. The secondary phase embedded in the matrix is a discontinuous phase. It is usually harder and stronger than the continuous phase. It serves 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 such as 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-sectioned 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.

The composite materials have got wide applications in all cutting-edge ranges of advanced materials such as aeronautics, automotives, boats, sports parts and medical devices etc. A composite is a structural material which at macroscopic level comprises two or more combined constituents and is insoluble among them. The two constituent used over here are reinforcing phase and the other one is matrix in which it is embedded. The reinforcing phase material is mainly present in the form of fibers, particles or flake. The matrix phase materials are normally continuous. Specimens of naturally found composite include wood in which the lignin matrix is reinforced with cellulose fibers. The composite materials are embedded with matrices to provide shape to the composite part, defend the reinforcements from the atmosphere and increase toughness of the material. The purpose of reinforcements is basically to achieve strength, stiffness and other mechanical properties. The composites materials have some advantages over metal such as:

- Light weight
- High specific stiffness and strength
- Easy mouldable to complex forms
- Easy bondable
- Good dumping
- Low electrical conductivity and thermal expansion
- Low radar visibility
- Internal energy storage and release

There are some disadvantages of composites which are presented below:

- Cost of materials
- Long development time
- Difficulty in manufacturing
- Fasteners
- Low ductility
- Temperature limits
- Solvent or moisture attack

1.3.1 Classification of Composite Materials

Composites are categorized on the foundation of (i) geometry of the reinforced particulate, flake, fibres, filled and directionally solidified eutectics and by (ii) the nature of matrix-polymer, metal, ceramic and carbon. Polymer matrix material is subdivided into thermosets and thermoplastics. Further, thermosets and thermoplastics are fragmented into epoxy, phenolic polyamide resin, polyester and polyethylene, polystyrene, polyamides, nylons and polypropylene, respectively.

1. Particulate composites comprise of particles submerged in matrices like alloys and ceramics. Particles are added randomly that make them isotropic. Particulate composites have strong features such as improved strength, increased operating temperature, oxidation resistance etc. Representative examples include utilizing aluminium particles in rubber, silicon carbide constituent part in aluminium, and gravel sand and cement that ensures their concreteness.

2. Flake composites are made up of flake reinforcements of materials. Typical flake materials are glass, mica, alumina and silver. Flake composites ensure benefits like high out-of-plane, flexural modulus, higher strength and low cost. Nevertheless flakes cannot be oriented and lacks stiffness.

3. Fiber composites comprise of matrices reinforced by short (discontinuous) or long (continuous) fibers. Fibers are generally anisotropic which are represented like carbon and aramides.

Polymer Matrix Composites (PMCs)

The most widely used in corporate sector are polymer matrix composites. These composites consist of a thermoplastic or thermosetting polymer reinforced with fiber (natural carbon or boron). These materials can be twisted into a variety of shapes and sizes. They provide great strength and stiffness and guard against resistance to corrosion.

Metal Matrix Composites (MMCs)

Metal matrix composites, as the name suggest, consists of a metal matrix. Examples of matrices in such composites consist of aluminium, magnesium and titanium. The distinctive fiber includes carbon and silicon carbide. Metals are mainly reinforced to ensemble the requisites of design. For example, the elastic stiffness and strength of metals can be augmented, whereas

large co-efficient of thermal expansion and electrical conductivities of metals can be abridged by the accumulation of fibers such as silicon carbide.

Ceramic Matrix Composites (CMCs)

Ceramic matrix composites comprises of ceramic matrix like alumina, calcium, alumina silicate strengthened by silicon carbide. The advantages of CMC include high strength, hardness, and high service temperature confines for ceramics, chemical inertness and low density. Naturally resistant to high temperature, ceramic materials have a propensity of becoming brittle and fracture. Composites efficaciously made with involvement of ceramic matrices are reinforced with silicon carbide fibers. These composites compromise of same high temperature tolerance of super alloys but deprived of such a high density. Composite fabrication becomes difficult because of brittle nature of ceramics. Usually most CMC production procedures require prerequisite material to be in powdered form. There are four branches of ceramics matrices like glass (easy to fabricate because of low softening temperatures, include borosilicate and alumino silicates), conventional ceramics (silicon carbide, silicon nitride, aluminium oxide and zirconium oxide are fully crystalline), cement and concreted carbon components.

Carbon-carbon composites (CCMs)

CCMs practice carbon fibers in a carbon matrix. Carbon-carbon composites are used at very high temperature atmospheres of about 3316 °C, and are twenty times robust and thirty times agiler than graphite fibers.

1.3.2 Constituents of composites

There are two main constituents of composite materials such as matrix and reinforcing agents. The purpose of matrix in a fiber-reinforced composite is transmission of stress between the fibers, to guard against adverse environment and to defend the surface of fibers from mechanical abrasion. The matrix plays a vital role in the tensile load carrying capacity of a composite structure. The binding agent or matrix involvement in the composite is of grave importance. Four major types of matrices have been testified. These are referred as polymeric, metallic, ceramic and carbon. Polymer matrices are main factor in analysis of composites used in industries. Polymer resins have been broadly divided into two categories, namely, thermosetting and thermoplastics.

Thermoset [19] is a rigid and stiff cross-linked material that does not changes shape when heated. Thermosets are stiff and doesn't stretch like elastomers and thermoplastics. Several types of polymers have been deployed as matrices for natural fiber composites. Most widely used thermoset polymers are epoxy resins and other resins like unsaturated polyester resins (as in fiber glass) vinyl ester, phenolic epoxy, Novolac and polyamide [20-22]. Unsaturated polyesters [23] are extremely resourceful in properties and applications. They are widely produced in industries as they retain many benefits as compared to other thermosetting resins including room temperature cure capability, good mechanical properties and transparency. The reinforcement of polyesters with cellulosic fibers has been extensively important. Polyester-jute [24, 25], polyester-sisal [26], polyester-coir [27], polyester-banana-cotton [28], polyester-

straw [29], polyester-pineapple leaf [30], and polyester-cotton-kapok [31], are some of the promising systems.

Thermoplastics [19] are polymers that have need of heat to make them in practise. After cooling, such materials preserve their figure. In addition to this, these polymers may be reheated and reformed, often without any significant changes in their properties. The thermoplastics which are used as matrix for natural fiber reinforced composites are as follows:

- (a) High density polyethene (HDPE)
- (b) Low density polyethene (LDPE)
- (c) Chlorinated polyethylene (CPE)
- (d) Polypropylene (PP)
- (e) Normal polystyrene (PS)
- (f) Polyvinyl chloride (PVC)
- (g) Mixtures of polymers
- (h) Recycled thermoplastics

Only those thermoplastics are functional for natural fiber reinforced composites, whose processing temperature (temperature at which fiber is incorporated into polymer matrix), does not exceed 230°C. These are, mostly, polyolefins, like polyethylene and polypropylene. Technical thermoplastics like polyamides, polyesters and polycarbonates have need of processing temperatures >250°C and are as a result not useable for such composite processing lacking fiber degradation.

Particle Reinforced Composite

Particles used for reinforcing ceramics and glasses include 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. They are also used to reduce the cost of the composites. Reinforcements and matrices can be common, inexpensive materials and those which are easily processed. Some of the useful properties of ceramics and glasses include high melting temperature, low density, high strength, stiffness, wear resistance and corrosion resistance. Many ceramics are good electrical and thermal insulators. Some ceramics have special properties and behave like magnetic materials, piezoelectric materials and superconductors at very low temperatures. Ceramics and glasses have one major drawback, i.e., brittleness [40]. An example of particle reinforced composites is an automobile tyre, which has carbon black particles in a matrix of polyisobutylene 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.

Fibers Reinforced Composites

Common fiber reinforced composites are made up 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.

The three most common types of reinforcing fibers include glass, carbon and Aramid fibres.

Carbon fibers

Carbon fibers [32] are used for reinforcing certain matrix materials to form composites. The physical properties of carbon fiber reinforced composite materials hinge on substantially on the nature of the matrix, the fiber alignment, the volume fraction of the fiber and matrix, and on the molding conditions. A number of types of matrix materials such as glass and ceramics, metal and plastics have assimilated as matrices for reinforcement by carbon fiber. Natural fiber reinforced composites, Carbon fiber composites, particularly those with polymer matrices, have turn out to be the foremost innovative composite materials for aerospace, automobile, sporting goods and other applications in regards to their high strength, high modulus, low density, and reasonable cost.

Glass fibers

Glass is widely used of all reinforcing fibers for polymeric (plastic) matrix composites (PMCs). The primitive advantages of glass fiber are low cost, high tensile strength, high chemical resistance and exceptional insulating properties. The two types of glass fibers mostly used in the fiber reinforced plastics commercial industries are E-glass and S-glass. Another type known as C-glass is used in chemical applications necessitating greater corrosion resistance to acids as compared to E-glass.

Kevlar fibers

Kevlar pertains to a group of highly crystalline aramid fibers having the lowest specific gravity and the highest tensile strength to weight ratio among the contemporary reinforcing fibers. They are being exercised as reinforcement in numerous marine and aerospace applications.

Boron fiber

The most prominent feature of boron fiber is that they have extremely high tensile modulus. Boron fibers provide excellent resistance to buckling, which in turn results in a high compressive strength for boron fiber reinforced composites.

The interest in natural fiber-reinforced polymer composite [45] 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 environment friendly, and are used in transportation like automobiles, railway coaches, aerospace, military applications, building and construction industries as ceiling panelling, partition boards, packaging, consumer products, etc.

Advantages of natural fiber

Natural fibers, as reinforcement, have attracted the attention of many researchers because of their benefits over other reputable materials. They are ecologically friendly, entirely biodegradable, copiously accessible, renewable and economical. Plant fibers bear insignificant weight as compared to glass, carbon and aramid fibers. The biodegradability of plant fibers can be able to subsidize to a healthy ecosystem notwithstanding the fact that their low cost and high performance accomplishes the economic interest of commercial industry. When natural fiberreinforced plastics are subjected to combustion process or landfill process, at the termination of its life cycle, the amount of carbon dioxide of the fiber released is unprejudiced with respect to its assimilation at the time of their growth [33]. Many automotive components earlier made from the glass fibers are now being produced using eco-friendly natural fibres [34]. Although natural fibers and their composites are eco-friendly and renewable, but still they are having several drawbacks which might include poor wet ability, incompatibility with some polymeric matrices and high moisture absorption [35]. Composite materials made with the incorporation of unprocessed plant fiber soon exhibit unsatisfactory mechanical properties. To subdue this, many times a surface treatment or compatibilizing agents should be incorporated in composite fabrication. The properties can be amended both by physical treatments like cold plasma treatment, corona treatment and chemical treatments such as maleic anhydride, organosilanes, isocyanates, sodium hydroxide, permanganate and peroxides [36]. Mechanical properties of natural fibers are much subordinate than those of glass fibers but their specific properties, exclusively stiffness, are analogous to the glass fibers [37].

Classification of Natural Fibers

Fibers are nothing but a category of hair-like materials that have continuous filaments or are mainly presented in discrete elongated pieces. It is a possibility that it can be fabricated into sheets for resulting in a products such as felt or paper. Fibers are of two types, namely, natural fiber and manmade or synthetic fiber. Figure 1 shows the variety/source of natural fibers.

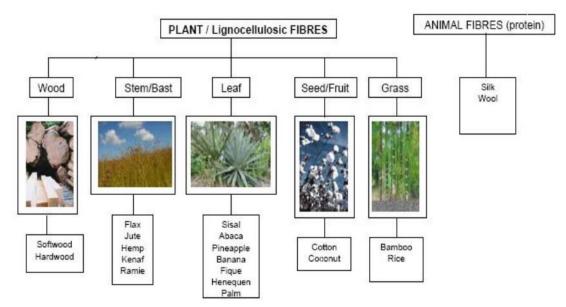


Fig 1: Various natural fibres

SOURCES OF NATURAL FIBERS

Natural fibers take account of those prepared from mineral, plant and animal sources. Natural fibers can be classified according to their source.

Vegetable fibers

Vegetable fibers are generally comprised mainly of cellulose, examples include cotton, jute, flax, ramie, sisal, and hemp [49]. Cellulose fibers aid in manufacturing of paper and cloth. This fiber can be further categorized into the following:

- > Seed fiber: Fibers gleaned from seeds or seed cases exemplified with cotton and kapok.
- ▶ Leaf fiber: Fibers gleaned from leaves, e.g., sisal and agave.
- Bast fiber or skin fiber: Fibers are gleaned from the skin or bast adjoining to the stem of their corresponding plant. These fibers have higher tensile strength on comparison with other fibers..
- Stalk fiber: These fibers are in fact the stalks of the plant. For example, straws of wheat, rice, barley, and other crops including bamboo and grass. Tree wood is also such a fiber.

The furthermost used natural fibers are cotton, flax and hemp, although sisal, jute, kenaf, and coconut are also broadly used.

Animal fibers

Animal fibers generally comprise of proteins, exemplifies with wool, angora, silk, alpaca and mohair .

- Animal hair (wool or hairs): Fiber or wool taken from animals or hairy mammals. For example, wool of sheep, hair of goat, alpaca, horse etc.
- Silk fiber: Fiber obtained from dried saliva pertaining to bugs or insects mainly for the the processing of cocoons. For instances silk obtained from silk worms.

Mineral fibers

Mineral fibers are naturally occurring fiber or to some extent altered fiber procured from minerals. These can be categorized into the following categories:

- Asbestos: The only naturally arising mineral fiber. Variations are serpentine (chrysotile) and amphiboles (amosite, crocidolite, tremolite, actinolite, and anthophyllite).
- Ceramic fibers: Glass fibers (Glass wool and Quartz), aluminium oxide, silicon carbide, and boron carbide.
- Metal fibers: Aluminium fiber

1.4 Advantages of Natural Fiber Composites

The core advantages of natural fiber composite are:

- Low specific weight, ensuing in a higher specific strength and stiffness as compared to glass fiber
- ➢ It is a renewable source, its fabrication necessitates little energy, and CO₂ is used despite the fact that oxygen is given back to the atmosphere.
- Processable with minimum investment at reasonable cost, which creates the material a thought-provoking product for low wage countries.
- > Abridged wear of tooling, robust working condition, and no skin irritation
- Thermal recycling is possible in this case whereas glass causes unruly for combustion furnaces.
- ➢ Good thermal and acoustic insulating properties

1.5 Disadvantages of natural fiber composite

Natural fibers incorporated in biocomposite are the one that decides their performance. Nevertheless, involvement of natural fibers in building materials has also some drawbacks such as low modulus elasticity, high moisture absorption, decomposition in alkaline environments or in biological attack, and variability in mechanical and physical properties. Chemical and physical properties of natural fibers are mainly reliable on cell wall polymers and their matrices. For illustration, dimensional stability, flammability, biodegradability, and degradation are accredited to acids/ bases and UV radiation that transform the biocomposites back to their basic building blocks such as carbon dioxide and water. Therefore, these problems can be rectified through improvising the basic chemistry of cell wall polymer thereby enhancing the natural fiber properties.

1.6 Applications of Natural Fiber Composites

The natural fiber composites can prove out to be cost cutting material for numerous applications including 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.

- > In storage devices: post-boxes, grain storage silos, bio-gas containers, etc.
- > In furniture: chair, table, shower, bath units, etc.
- > In electric devices such as electrical appliances, pipes, etc.
- > In everyday applications like lampshades, suitcases, helmets, etc.
- > In transportation: automobile and railway coach interior, boat, etc.
- The justification for the employment of natural fibers in the automotive industry includes 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
- > Favourable accident performance, high stability, less splintering
- ➢ Favourable ecobalance for part production
- > Favourable ecobalance during vehicle operation due to weight savings
- Occupational hazards are not there in natural fiber whereas glass fibers suffer from this during its 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

1.7 CHALLENGES

Natural fibers acquired from sustainable sources suffer a huge inconsistency ranging from medieval period of harvesting plant, climate and geographical circumstances, variation in treatment methods, and so on. Eventually, a possible variation in the quality of end-products has emerged. In particular, this resultant may have unstable properties depending on the storage and transportation conditions. Thus, the challenge is emphasized on material design where the materials must be structurally and operationally unwavering in the course of storage but degradable after utilization.

In addition, when hydrophilic natural fibers and PVA are united, then it becomes inevitable to consider their poor moisture resistance due to existence of hydroxyl groups. These natural fibers possess low mechanical strength due to their high moisture retention which explicitly affects the bonding between fibers. A great deal of work has been suggested due to the poor

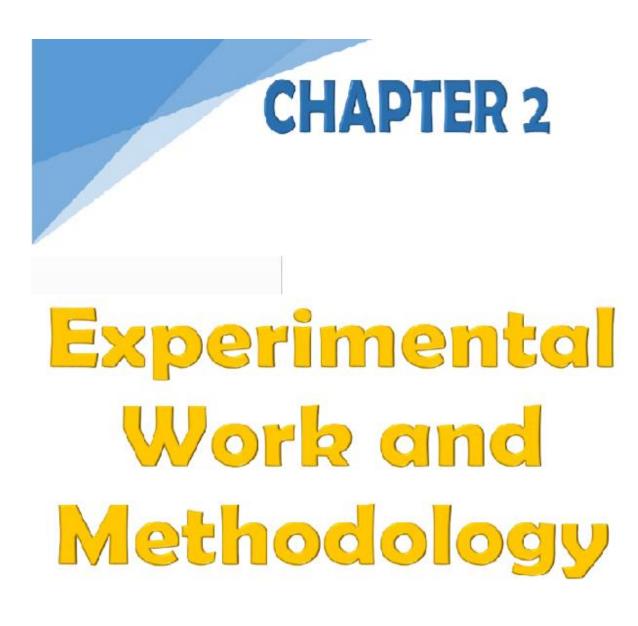
moisture resistance of PVA/natural fibers and it has been recommended, on its subjection to chemical modifications or application of certain coatings, to diminish their moisture uptake. As an effort continues to modify the composites' possessions, it is imperative to evaluate their aftermath on the surroundings because of involvement of chemical reagents.

1.8 Objective of the present work

The aim of the present work is to fabricate natural fibre based polymeric composites at different proportions of polymer and to study the tensile strength, hardness and tear resistance etc. In the present project work, an attempt was also made to increase the density and hardness of the water cured cylindrical samples.

1.9 Work Plan

A thorough literature survey and related publication analysis was done to identify the subject matter and chalk out the strategy in detail for the preparation of mould for samples. Experiment process commenced in very conducive environment and all the planned processes were completed step by step. After the preparation of test specimen, UTM machine was scrutinised whether all the parameters are in the permissible variation limit or not. Calibration of the machine was also verified and validated on the date of testing. Tensile strength of the samples were determined and recorded in the file. Subsequently, tear resistance study as per provision of ASTM D412 was done in detailed to characterize the various samples followed by other studies like water absorption etc.



2.1 MATERIALS USED

2.1.1 Polyvinyl alcohol

Polyvinyl alcohol (PVA) is a translucent, water-soluble synthetic resin engaged predominantly in textile and paper treatment industries. PVA is different from other polymers as it is not synthesized from its single-unit precursor molecules known as monomer(s). Instead, PVA [54] is prepared by dissolving another polymer, polyvinyl acetate (PVAc), in methanol and treating it with catalyst such as sodium hydroxide. The chemical structure of repeating unit, i.e., 'vinyl alcohol' is presented in Fig. 1 as below.

Fig. 2: General structure of polyvinyl alcohol

When the reaction is complete, the resultant product is highly water soluble and it is insoluble in all other organic solvents. The partial elimination of the acetate groups from PVAc yields resins which are less soluble in water and more soluble in certain organic solvents.



Fig. 3: Polyvinyl alcohol

PVA has got its applications in sizing agents that imparts greater strength for textile yarns and increases paper resistance to guard against oils and greases. It is used in the form of component for adhesives and emulsifiers, as a water-soluble protective film, and as a starting material for the preparation of other resins.

Functional uses

Polyvinyl alcohol has numerous applications in the food industries playing the role of binding and coating agent. It can be used as a film coating agent to guard against moisture. Polyvinyl alcohol protects all the active constituents from moisture, oxygen and other environmental hazardous components, while concurrently disguising their taste and odour. It provides easy management of finished product. The viscosity feature of polyvinyl alcohol allows for its usage as coating agents in tablets and capsules. Polyvinyl alcohol is used in high moisture foods so that taste, texture and all qualities of the foods persists. The use levels of polyvinyl alcohol are tabulated in the following table as reported in the literature.

Use levels of Polyvinyl alcohol				
Food Category	Proposed Food-Use	Use Levels for Polyvinyl alcohol (%)		
Dairy-based desserts	Ice cream and frozen yogurt with inclusions	0.2		
Confectionery	Multi-component chocolate Bars	1.5		
Cereals and cereal products	Ready to eat breakfast cereals with dried fruits	0.5		
Food supplements	Food supplement tablets	1.8		
Ready to eat savories	Nut and fruit mixtures	1.5		

The foodstuffs in which polyvinyl alcohol is proposed to be added because of their extensive qualities like neutral pH and storage at either low temperature or at room temperature conditions that would not result in cessation of PVA.

2.1.2 Glycerol

Glycerol is colourless, viscous, sweet-tasting liquid pertaining to the class of alcohol with molecular formula HOCH₂CHOHCH₂OH. Until 1948 glycerol was acquired in the form of byproduct in manufacturing soaps from animal and vegetable fats and oils, but industrial syntheses is nowadays centred on propylene or sugar. The term glycerin (or glycerine), familiarised in 1811 by French chemist Michel-Eugène Chevreul, is generally incorporated in commercial materials containing more than 95 percent glycerol."

Glycerol [51] has abundant uses. It is an elementary ingredient in gums and resins used to make several contemporary protective coatings like automotive enamels and exterior house paints. Glycerin on reaction with nitric and sulfuric acid results in the explosive nitroglycerin (or nitroglycerine). Glycerol is also a component of mono- and diglyceride emulsifiers, which are quite often used for the purpose of softening agents in baked goods, plasticizers in shortening, and stabilizers in ice cream. Its wide-ranging uses in the pharmaceutical and toilet goods fields take account of skin lotions, mouthwashes, cough medicines, drug solvents, serums, vaccines, and suppositories. Another noteworthy use is for instance a protective medium for freezing red blood cells, sperm cells, eye corneas, and other living tissues.

2.1.3 NATURAL FIBRES

(1) Bagasse fiber-reinforcement

Sugarcane bagasse is a fibrous residue of cane stalks left over after the crushing and extraction of juice from the sugar cane and is broadly produced in high magnitudes for agro based industries. Bagasse [52] is generally bulky, gray-yellow to pale green in colour, and quite non uniform in particle size. The sugar cane residue bagasse is a renewable agricultural material that consists of two distinct cellular constituents. The first is a thick walled, relatively long, fibrous fraction derived from the rind and fibro-vascular bundles dispersed throughout the interior of the stalk. The second is a pith fraction derived from the thin walled cells of the ground tissue. The main chemical constituents of bagasse are:

• Cellulose and hemicelluloses are present in the form of hollow cellulose in bagasse which contributes to about 70 % of the total chemical constituents present in bagasse.

• Lignin acts as a binder for the cellulose fibres and also behaves as an energy storage system. Bagasse consists of water, fibre and small quantities of solids in solution in the following proportions. Water 46-57 % (mean 50%), fibre 43-53 % (mean 47%), and solids in solution (sugar) 2-6 % (mean 3%). It is a composition within certain limits as variable and depends in the varieties, their maturity, the harvest technique and efficiency of milling.



Fig. 4: Bagasse Fibre

(2) Rice husk

Rice husk is widely used as substitute for agricultural wastes in voluminous rice cultivating countries of world. Globally, rice paddy production has approximately found out to be 600 million tons per year. Out of this massive production 20% of it pertains to rice paddy husk which in turns results in an yearly gross delivery is around 120 million tonnes. Residual part after burning of rice husk [50] in ambient atmosphere is known as rice husk ash. For every single 1000 kgs of paddy milled, about 220 kgs (22 %) of husk is obtained.



Fig. 5: Rice husk

Besides its usage as fuel in power plants, rice husk are also been used as source raw material in the development and synthesis of new phases and compounds. Rice husk are tremendously used as fuel in boilers for generating process steam. Direct combustion and/or by gasification is employed for the production of heat energy. Virtually rice husk are available in abundance for free. Now, rice husk has also found its pathway for alternative fuel in household.

(3) Wheat bran

Wheat bran [53], a by-product of flour milling, is composed of the pericarp and the outermost tissues of the seed including the aleurone layer. It constitute almost 10% of the total weight of wheat milled for flour. On a moisture-free basis it contain about 70% carbohydrates, comprising, in approximate amount, 43% hemicellulose, 35% cellulose, 14% starch, and 8% sugars.



Fig. 6: Wheat bran

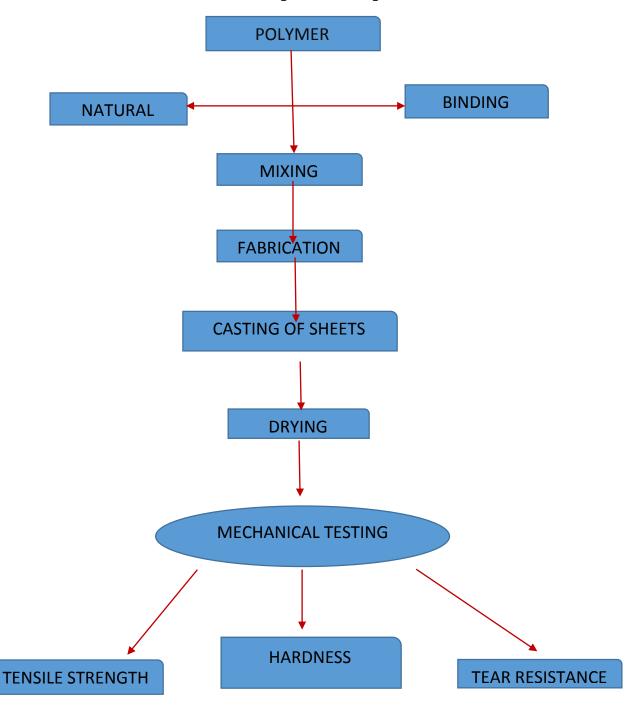
(4) Saw dust

Saw dust is a waste product which comprises of small constituent part extracted during the process of sawing wood [54]. The dimensions of sawdust particles are prominently determined by the sort of cutting tool, the cutting speed, and the rate of feed employed. Hydrolysates incorporate sawdust as a raw material for its production. It is quite often used in the manufacturing of wood meal in the form of filler and fuel. It can present a threat in manufacturing industries, particularly in terms of its flammability. Sawdust is the main component of particleboard.



Fig. 7: Saw dust

Flow chart for experimental procedure



2.2 EXPERIMENTAL METHODS

The various materials required for the sample preparation include polyvinyl alcohol, glycerol, urea, sodium hydroxide and various natural materials such as rice husk, saw dust and sugarcane bagasse. Polyvinyl alcohol is usually in the form of powder or granule form which is converted to hot liquid during fabrication by the addition of hot water. Glycerol was added to the hot molten PVA which will then be used as a binding agent.

A. Sample preparation

Waste recycled materials have been used as raw materials during the experimental work. The resources such as rice husk, wheat bran, saw dust and bagasse are utilized in this work because of their remarkable qualities like bio-degradability, higher strength to weight ratio and also due to their flexible nature. Straw is hay gleaned from a neighbouring farm and is made into use after drying it evenly to a constant temperature of about 35 °C. The foremost reason to favour straw is because of its hardness, strength and flexibility. Sawdust is used in the form of powder to avoid lumps as it binds very hard to its matrix phase. Bagasse is the desiccated form of sugarcane. The sugarcane is basically chopped and dried to a consistent giddel

temperature of about 38 °C and then it is powdered evenly for the resin. Natural fibers are used as reinforcement phase and polyvinyl alcohol as matrix phase in the bio-composite formation. The samples are prepared in the form of sheets of thickness 3 mm.

B. Preparation of mould

Steel sheet and foam board is used for the preparation of mould. Steel sheet is given preference over glass because of their more stress bearing capacity. A border was erected using the foam board around the steel sheet to prevent the PVA to stream out. The thickness of the border was taken to be 6 mm approximately, so that a 3 mm sheet of bio-composite using the materials was formed.



Fig.8: Layout and setup of mould

C. Preparation of the resin

A beaker containing 500 ml of water is taken and heated to the temperature of 100°C. Then, 70 g of polyvinyl alcohol is added to the boiling water and stirred constantly using the glass rod.

D. Addition of waste recycling materials

Sawdust and rice husk, already present in the ground powder form, were added directly to the molten PVA gel without any further grinding but Sugarcane bagasse was grounded properly until it turned into a nice powder form before its addition.

E. Casting of sheets

The prepared resins along with the powdered fibers were poured over the properly lubricated mould that ensures the smooth extraction of the sheet. Then, over the fiber the second layer of the resin PVA is poured and the process is continued. Thus, the mould is completely filled with the resin and the matrix and is left open under the sunlight to dry.

F. Drying

The mould was allowed to dry under the sunlight for the period of 2 days. An extensive drying of the mould should be avoided as it may lead to the sticking of the mould to the surface and it will then become hard to remove after drying. Also, an extensive drying leads to the formation of the voids.

G. Extraction of sheets

After drying the bio-composite sheet made of recycling materials was removed from the mould. The thickness of the resin and fiber mixture gets reduced roughly by about 50% due to the evaporation of the water from the mixture during the drying process. The sheet is then first sliced at the border and removed slowly with care so that there should not be any wear and tear of the sheet.



Fig 8: Bio-composite sheet with different types of fibres

2.3 DETERMINATION OF MECHANICAL PROPERTIES

2.3.1 Hardness

Vickers hardness tester (LECO, LM 248AT) as shown in Fig. 9, was used to find the hardness values of all the dry and wet samples using 20 gf load for a dwell time of 15 sec. The measurements for each sample were taken at different positions in order to get good results.



Fig. 10: Micro indentation hardness tester

2.3.2 Tensile strength

A universal testing machine (UTM) also known as a universal tester, materials testing machine or materials test frame is used to test the tensile stress and compressive strength of the samples. This machine can perform many standard tensile and compression tests on materials, components, and structures.



Fig. 11: Universal testing machine

2.3.3 Tear resistance

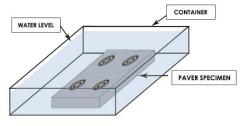
Tear resistance is the measurement of a sample's ability to resist tearing. Tear resistance testing can be found in plastic film, paper and textile industries. Tear resistance can be impacted considerably by the speed of the test, e.g., test speed used to generate the tear. Tear propagation resistance for the purpose of acceptance testing is common with materials such as paper and rubber. Tear resistance in textiles involves the load required to propagate a single "rip-tongue" type of tear.



Fig. 12: Tear resistance machine

2.3.4 Water absorption test:

For water absorption tests, samples of each composite were oven dried before weighing and the weight recorded was reported as the initial weight of the composites. The samples were then placed in distilled water maintained at room temperature (25 °C) and at the time intervals of 24h, the samples were removed from the water, cleaned using a dry cloth and weight recorded.



Water Absorption Test

The amount of water absorbed by the composites (in percentage) was calculated using the following equation:

 $W(\%) = W_t - W_o / W_o \times 100$

Where, W is the percent water absorption, W_o and W_t are the oven dry weight, and the weight of the specimen after time t, respectively.

2.3.5 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) is mainly used for microanalysis and failure examination of solid inorganic materials. Scanning electron microscopy is executed at high magnifications, it results in a high-resolution image and accurately do the analysis of minute objects.

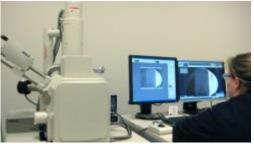


Fig 14: scanning electron microscopy



Results and Discussion

This section deals with effect of different fibrous fillers on the mechanical and absorption properties of Polyvinyl alcohol. Samples were formed in triplicate for this study. Morphological studies were also carried to study the dispersion of fillers.

Properties	Sample 1	Sample 2	Sample 3	Average
Tensile Strength (N/mm ²)	0.34666	0.79819	0.67875	0.607
Hardness (Shore A)	56	67	77	66.66
Tear Resistance (N/mm)	21.24	25.86	31.04	26.04

Table 1: Various	Test Results of	Wheat Bra	n Fiber	Samples (all samples are of
same dimension)				

 Table 2: Various Test Results of Rice Husk Fiber Samples (all samples are of same dimension)

Properties	Sample 1	Sample 2	Sample 3	Average
Tensile Strength (N/mm ²)	14.60	6.72	7.78	9.52
Hardness (Shore A)	47	51	56	51.33
Tear Resistance (N/mm)	31.55	34.68	36.72	34.31

Properties	Sample 1	Sample 2	Sample 3	Average
Tensile Strength (N/mm ²)	46.69	68.28	52.12	55.69
Hardness (Shore A)	54	67	72	64.33
Tear Resistance (N/mm)	102.7	115.7	119.4	112.6

 Table 3: Various Test Results of Saw Dust Fiber Samples (all samples are of same dimension)

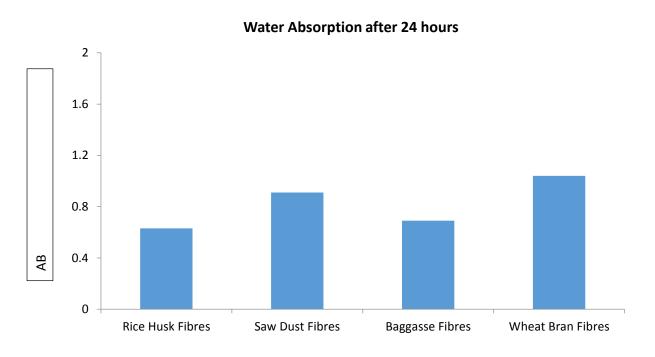
 Table 4: Various Test Results of Baggasse Fiber Samples (all samples are of same dimension)

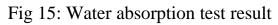
Properties	Sample 1	Sample 2	Sample 3	Average
Tensile Strength (N/mm ²)	3.03	5.16	1.90	3.36
Hardness (Shore A)	45	56	71	57.33
Tear Resistance (N/mm)	68.60	78.95	82.89	76.81

	Rice Husk Fiber	Saw Dust Fiber	BaggasseFiber	Wheat Bran Fiber
Specimen	Absorption(%)	Absorption(%)	Absorption(%)	Absorption(%)
1	0.58	0.84	0.63	0.97
2	0.65	0.89	0.69	1.04
3	0.67	1.02	0.75	1.11
Average	0.63	0.91	0.69	1.04

Table 5: Absorption Results (24-Hours)

Bar Chart Showing Percent Water Absorption of the Samples





3.1 TENSILE TEST

Tensile strength of composites containing various fibrous fillers were studied. It was found that there is a significant increase in the tensile strength of the saw dust reinforced composites while there is no significant increase in other samples. The reason for the significant increase in saw dust reinforced composites is attributed to better dispersion of filler in PVA matrix while with other fillers, this was not the case.

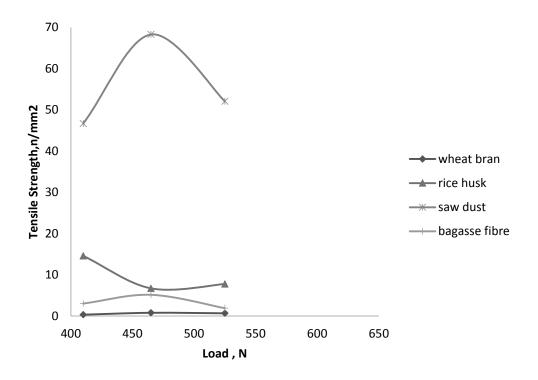


Fig 16 : Tensile strength variation of different types of fibre

In order to associate the results for tensile strength variation for these four types of fibers , an average tensile strength of rice husk, saw dust, baggasse and wheat bran fibers are testified in figure 17 given below . It has been perceived from the figure that average tensile strength of sawdust fiber is highest among all these types of fiber.

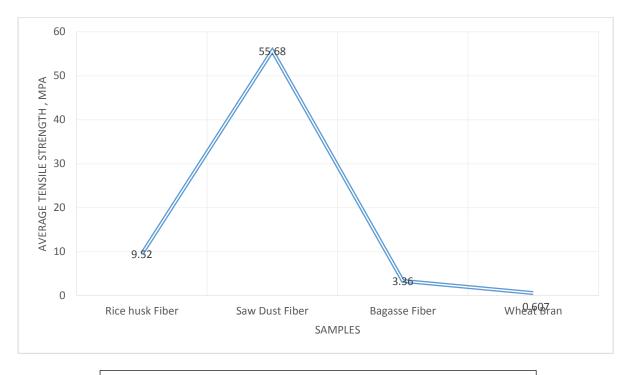


Fig 17 :Average tensile strength variation of different types of fibers

3.2Tear Resistance Test

Tear is caused due to any damage when under tension. The saw dust fiber has shown an exceptional tearing resistance over all types of fiber as shown in the fig. 18. Again this increase in tear strength of saw dust containing composites is attributed to better dispersion of filler.

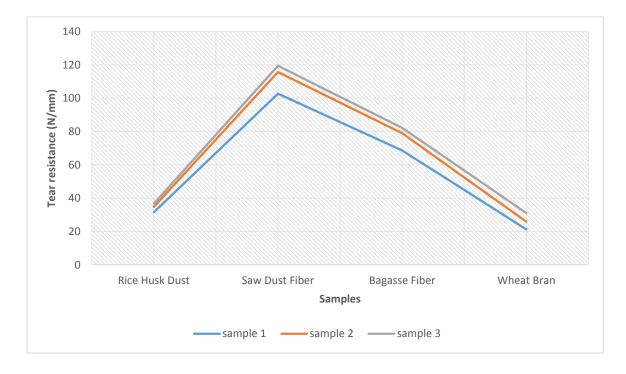
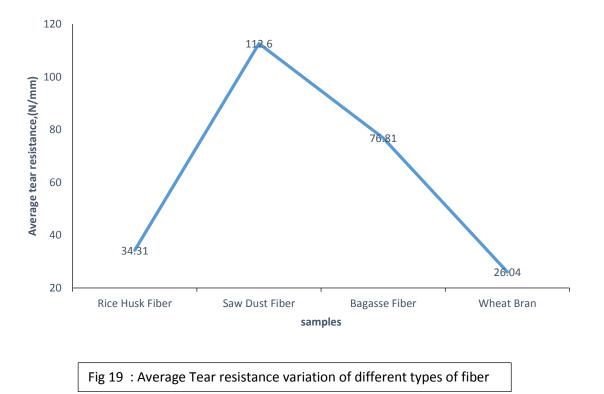


Fig 18 :Tear Resistance variation of different types of fiber

A comparison of average tearing resistance for different types of fiber has been shown in fig 19. The saw dust fiber has shown the highest tearing resistance among all types of fibers.



3.3 Shore Hardness Test: The figure shown hardness status of different fiber (rice husk, baggasse, saw dust and wheat bran fibers) which were added to the resin. For comparison among all the types of fiber, it has been reported that the wheat bran fiber has shown maximum average hardness of 77A as shown below.

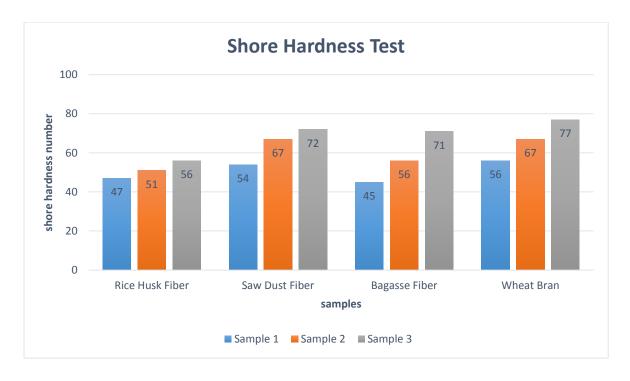


Fig 20 : Shore hardness variation of different types of fiber

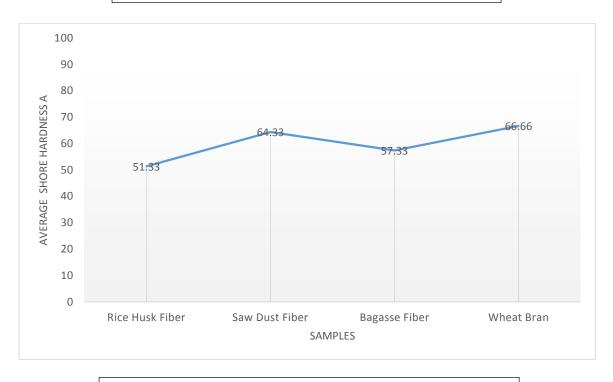


Fig 21 : Average shore hardness variation of different types of fiber

Morphological studies

Microstructure features were studied using Scanning Electron Microscope. The SEM microstructure of all samples is shown below. It provides an excellent examination of surface morphology of different natural fibre reinforced composites .It has been shown that surface morphology of all the fibre is different in terms of level of roughness and smoothness .Therefore analysis of fibre morphology could provide vital information on the level of interfacial adhesion that exist between fibre and matrix.

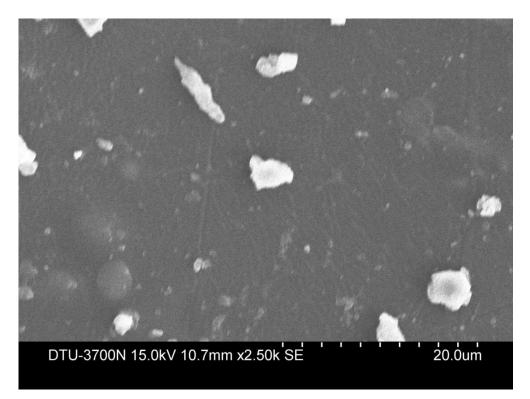


Fig 22: Saw dust

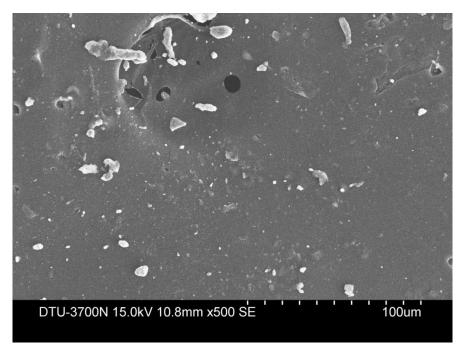


Fig 23: Bagasse fibre

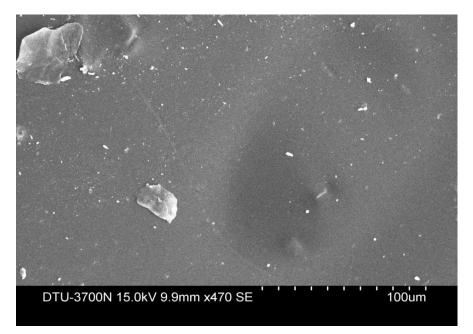


Fig 24: Rice husk

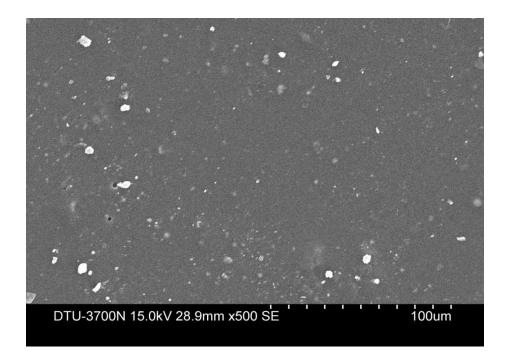


Fig 25: wheat bran



Conclusions

CONCLUSION:

Natural fiber reinforced polymer composites comprises of properties such as low density, cost effective, and reduced solidity in comparison with synthetic composite yields, which results in benefits in commercial applications like automotive industry, buildings, and constructions. Involvement of natural fibers as reinforcement for polymeric composites has shown drastic positive changes on the mechanical characteristics of polymers. A successions of mechanical tests were implemented to scrutinise the external morphology, chemical composition, crystalline structure, tensile strength, tear resistance, hardness and water absorption of different types of bio-composite materials.it has been concluded from the results that: the composition containing sawdust as filler has highest tensile strength compared to other fillers. Saw dust fiber has presented excellent tearing resistance over all types of fiber. As the loading is increased in each test the tearing resistance also increases for the saw dust fiber composite. The wheat bran fiber bio-composite reported highest average hardenss of 77 shore A. Such composites have turn out to show commendable properties and hence evolved as a new generation for composites. Considering their broad, universal and profitable characteristics, they have outnumbered their area of application including consumer, agriculture and biomedical.



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