

**PROCESSING AND CHARACTERIZATION OF BETEL NUT AND TEXTILE
FIBER REINFORCED HYBRID POLYPROPYLENE COMPOSITES**

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SUBMITTED BY

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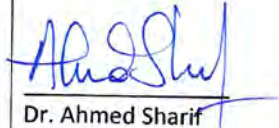
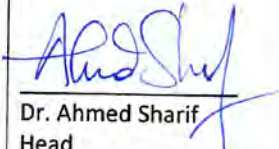
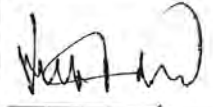
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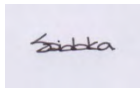
The thesis entitled 'PROCESSING AND CHARACTERIZATION OF BETEL NUT AND TEXTILE FIBER REINFORCED HYBRID POLYPROPYLENE COMPOSITES' submitted by SALMA SIDDIKA, Roll No. 0413112006 P, Session April 2013, has been accepted as satisfactory in partial fulfillment of the requirement for the degree of Master of Science in Materials and Metallurgical Engineering on 10th January, 2016.

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Salma Siddika (0413112006)

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The Author

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ABSTRACT

In the present work hybrid composites were manufactured by using hot press machine incorporating 2, 3 and 4 layers of natural (e.g. areca) and textile (e.g. nylon) fibers at constant fiber loading of 10 wt.% . Consequently areca fiber was treated by 5% NaOH. Composites were also prepared at four levels of fiber loading (5, 10, 15 and 20 wt %) with fiber ratios of (treated areca: nylon=1:1) , (treated areca: nylon=3:1) , (treated areca: nylon=2:3) , (treated areca: nylon=3:2) and (treated areca: nylon=3:2) for 20% fiber loading. The tensile, flexure and hardness tests were conducted for mechanical characterization of composites. Tensile strength, Young's modulus, %EAB, flexural strength, flexural modulus, shore hardness values enhanced with the increase of number of fiber layers. Interestingly tensile strength and % elongation at break showed a decreasing trend with the amount of fiber content in the composites. On the other hand Young's modulus and flexural properties increased up to a certain level and hardness exhibited an increasing trend with the increase of fiber content. Notably all these mechanical properties enhanced with increasing nylon fiber content except tensile strength. In addition 5% NaOH treatment of the areca fiber provided an improving trend of properties of the composites. The Fourier Transform Infrared spectroscopy was carried out of the composites to study the effect of alkali treatment which showed decrease of hemicelluloses content. Scanning Electron Micrograph imaging of tensile fracture surfaces of all the composites were carried out to examine the variation in failure modes and to investigate the interfacial adhesion and bonding between fiber and PP matrix, in this case 4 layers of fibers containing matrix showed favorable entanglement between fibers and matrix. Thermo Gravimetric analysis showed slight enhancement of thermal stability after alkali treatment. These composite can be used in manufacturing automotive parts such as various panels, shelves, trim parts and brake shoes.

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LIST OF ABBREVIATIONS

FRP	Fiber Reinforced Polymer
PP	Polypropylene
PMC	Polymer Matrix Composite
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscopy
MMC	Metal Matrix Composites
CMC	Ceramic matrix Composites
CCM	Carbon Carbon Composites
UTS	Ultimate Tensile Strength
TGA	Thermogravimetric Analysis

LIST OF NOTATIONS

σ	Ultimate tensile strength
P_{\max}	Maximum load
A	Area
R	Rate of cross head motion
Z	Rate of straining of the outer fiber
d	Depth of beam
L	Support span
b	Width of beam
D	elongation
m	slope of linear portion of stress-strain curve

CHAPTER 1

INTRODUCTION

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Introduction

With the growth in world population and overall improvement of living standard, global fiber consumption has been steadily increasing in the past few decades, which leads to a higher amount of post industrial and post consumer fibrous waste materials [1]. According to the Environmental Protection Agency (EPA), USA clothing and household textiles currently make up 5.2% of the waste in landfills. Textile waste creates pollution and wastes precious resources. Just 1 kilogram of textiles emits more than 7 kilograms of CO₂. Furthermore, the World Wildlife Fund has estimated that it wastes 8500 liters of water to raise 1 kilogram of cotton lint [2]. All these make the global textile industry one of the most polluting and waste-generating sectors in the world. Recycling clothing and textiles decreases the amount of waste disposal that are not biodegradable. It also decreases the need for energy and chemicals used in manufacturing new textiles and the pollution caused by the manufacturing processes. Using waste textile fibers in the production of composite materials has already opened a new era of recycling these into more valuable objects.

A fiber reinforced polymer (FRP) is a composite material consisting of a polymer matrix embedded with high-strength fibers. Now-a-days hybrid FRP composites are receiving considerable attention by the world's scientific community for its capability of providing designers new freedom of tailoring composites and thus achieving properties that cannot be attained in binary systems containing one type of fiber dispersed in a matrix. It has also provided a more cost-effective utilization of expensive fibers by replacing them partially with less expensive fibers. Hybrid composites provide the potential of achieving a balanced pursuit of stiffness, strength and ductility, as well as bending and membrane related mechanical properties with weight savings, reduced notch sensitivity, improved fracture toughness, longer fatigue life and excellent impact resistance [3].

In today's world increasing environmental awareness has promoted designs that are compatible with environment, non toxic to human body so eco-friendly. Natural fiber reinforced composites are inexpensive and could minimize environmental pollution due to their characteristics biodegradability [4]. Apart from this, the lignocellulosic fibers are lightweight, easily available,

renewable, non-abrasive, require less energy for processing, reduce the density of furnished products and absorbed CO₂ during their growth [3-5]. The lignocellulosic fibers can be mixed either with thermosetting or thermoplastic polymer matrix to produce composites [5]. On the other hand synthetic textile fibers provide excellent mechanical properties that are not achievable using only natural fiber. Thus incorporation of natural and waste textile fiber in one matrix to produce hybrid composite provides eco friendliness, eradicates waste disposal problem and a well-balanced combination of mechanical properties are obtained.

The physical and mechanical properties of lignocellulosic composites largely depend on the type of matrix, content and properties of the reinforcement fillers and filler–matrix interaction. However, the incompatibility between the hydrophilic natural fibers and the hydrophobic matrices leads to undesirable properties of the composites. This interfacial adhesion can be improved by the chemical modification of the fiber surface or the matrix. Apart from those the properties and interfacial characteristics of the composites are significantly influenced by the processing parameters used. Therefore, suitable processing techniques and parameters must be carefully selected in order to yield the optimum composite properties [5].

Among various natural fibers, Betel nut (Areca) is quite available in south Asia and is also cheap in context to the economic condition of Bangladesh. Thus the objective of present research is to develop hybrid composite by using lignocellulosic areca (betel nut husk fiber) fiber and waste nylon fiber reinforcements.

The purpose of this thesis is to characterize both raw and treated areca and waste nylon fiber reinforced hybrid polypropylene composite by finding out their morphological, chemical, physical and mechanical characteristics. The major steps of the research project are as follows:

- ❖ Fabrication of raw areca and waste nylon fiber reinforced hybrid polypropylene matrix composites containing same amount of fibers (e.g. 10 wt%) distributed in different number of packing layers (e.g. 2, 3 and 4 layers). In all cases ratio of the individual fiber (i.e. areca and nylon) kept constant in (1:1).
- ❖ Chemical (alkali) treatment of hydrophilic areca fibers to enhance its compatibility with hydrophobic polypropylene matrix.

- ❖ Fabrication of composite reinforced with treated areca and waste nylon fibers containing different wt% of total fiber. In this case. the number of fiber packing layers was kept constant at 4 layers and the ratio of areca and nylon was maintained at (1:1).
- ❖ Fabrication of composites reinforced with treated areca and waste nylon fibers with different ratio of areca and nylon fiber.
- ❖ Mechanical properties of composites such as Tensile strength, Young's modulus, Flexural strength and Hardness were measured.
- ❖ SEM analysis of tensile fracture surfaces of all type of composites were carried out accordingly.
- ❖ FTIR spectroscopy and TGA analysis of both raw and treated areca fiber reinforced composites were also performed.

The thesis is divided into a number of sections consisting literature review, experimental procedure, composite mechanical property characterization, their results and discussions, conclusion and references.

hopefully these composite can be used in producing mirror casing, paper weights, projector cover, voltage stabilizer cover, mail-box, helmet ,roof and various parts of car interior and truck cabins.

CHAPTER 2

LITERATURE REVIEW

LITERATURE REVIEW

What is Composite?

Composite is a multiphase material that is artificially made, as opposed to one that occurs or forms naturally. Composite materials are made from two or more constituent materials with significantly different physical or chemical properties, that when combined, produce a material with characteristics different from the individual components. The individual components remain separate and distinct within the finished structure.

Generally, a composite material is composed of reinforcement (fibers, particles, flakes, and/or fillers) embedded in a matrix (polymers, metals, or ceramics). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix [6].

Benefits of Composite

The numerous unique features of composite materials have led to the widespread adoption and use of it through worldwide in many different industries. Below are some of the most important features of composites, and the benefits they provide [7].

- ❖ Composites are incredibly lightweight, a car made from composites weighs 1/4 that of a car made from steel. As a result of which it provides high strength to weight ratio and high stiffness to weight ratio. This amazing performance results in fuel saving and makes it cost effective. This lightweight property has made composites an ineliminable candidate in aerospace industry.
- ❖ The marine industry was one of the first to adopt the use of composites because of their high resistance to various types of chemicals and they do not get corroded in marine environment.
- ❖ Fiber reinforced composites have excellent elastic properties. Bending of metals results in its yielding or dent formation. However, when composites are bent, they want to naturally

snap back into place. This feature is ideal for springs, and that is why composites are used in car leaf springs and in the limbs of archery bows.

- ❖ Certain composites, such as composite made with fiberglass, are non-conductive. This is important because often a structure requires combination of strength and non conductivity of electricity.
- ❖ Composites may be made by a wide range of processes.
- ❖ Composite Laminate patterns and ply buildup in a part can be tailored to give the required mechanical properties in various directions.
- ❖ It is easier to achieve smooth aerodynamic profiles for drag reduction by using composites. Complex double-curvature parts with a smooth surface finish can be made in one manufacturing operation.

Types of Composites

The reinforcement system in a composite material strongly determines the strengthening mechanism in a composite. It is thus convenient to classify composites according to the characteristics of the reinforcement, such as length, orientation etc and the matrix hold them in their place so there are two classification systems of composite materials. One of them is based on the matrix material (metal, ceramic and polymer) and the second is based on the material structure [3].

Classification of Composite Materials 1

(Based on reinforcing material structure)

Particulate Composites

Particulate Composites consist of a matrix reinforced by a dispersed phase in form of particles. the particle diameter is typically on the order of a few microns. In this case, the particles carry a major portion of the load. The particles are used to increase the modulus and decrease the ductility of the matrix. Particles can have virtually any shape, size or configuration. Examples of well-known particulate composites are concrete and particle board. The composite can be of following types :

1. Composites with random orientation of particles.

2. Composites with preferred orientation of particles. Dispersed phase of these materials consists of two-dimensional flat platelets (flakes), laid parallel to each other.

Particulate composites offer several advantages. They provide reinforcement to the matrix material thereby strengthening the material. The combination of reinforcement and matrix can provide very specific material properties. For example, the inclusion of conductive reinforcements in a plastic can produce plastics that are somewhat conductive. Particulate composites can often use more traditional manufacturing methods such as injection molding which reduces cost [8].

Fibrous Composites

The fiber element performs as the main load-bearing component in fiber reinforced composites. Dispersed phase in form of fibers improves strength, stiffness and fracture Toughness of the material, impeding crack growth in the directions normal to the fiber. Effect of the strength increase becomes much more significant when the fibers are arranged in a particular direction (preferred orientation) and a stress is applied along the same direction. Fibers come in three different forms: long, straight continuous fibers that are stacked parallel to each other; short chopped fibers that are randomly distributed through the composite and cloth woven fibers that give durability [9].

Short-fiber reinforced composites

Short-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers (length < 100diameter).this type of composites can be of two types:

1. Composites with random orientation of fibers.
2. Composites with preferred orientation of fibers.

Long-fiber reinforced composites.

Long-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of continuous fibers. Matrix in such materials serves only as a binder of the fibers keeping them in a desired shape and protecting them from mechanical or chemical damages.

3. Unidirectional orientation of fibers.
4. Bidirectional orientation of fibers (woven).

Laminate Composites

Laminate composites consist of layers with different anisotropic orientations or of a matrix reinforced with a dispersed phase in form of sheets. When a fiber reinforced composite consists of several layers with different fiber orientations, it is called multilayer (angle-ply) composite [6].

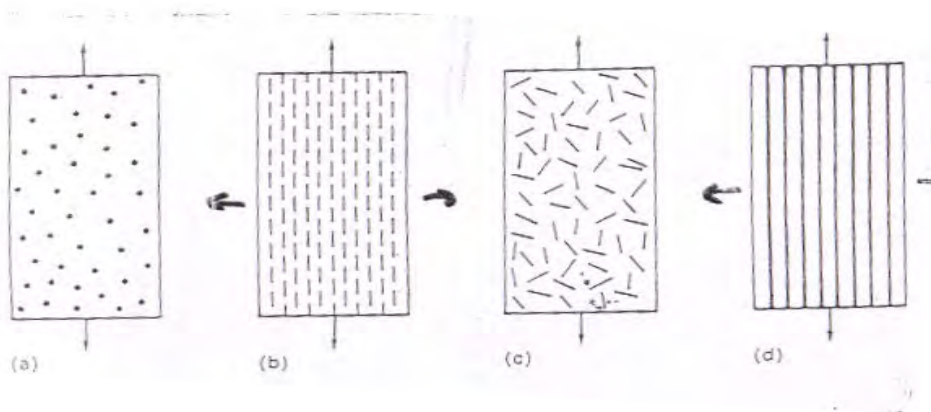


Figure 1: Types of composites based on fibers (a) Particulate & random; (b) Discontinuous fibers & unidirectional; (c) Discontinuous fibers & random; (d) Continuous fibers & unidirectional [10]

Laminate composites provide increased mechanical strength in two directions and only in one direction, perpendicular to the preferred orientations of the fibers or sheet, mechanical properties of the material are low.

Classification of Composites II

(Based on matrix material)

Metal Matrix Composites (MMC)

Metal Matrix Composite (MMC) are made by dispersing a reinforcing material (i.e. ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) into a metallic matrix [6].

The reinforcement surface can be coated to prevent a chemical reaction with the matrix. For example, carbon fibers are commonly used in aluminum matrix to synthesize composites showing low density and high strength. However, carbon reacts with aluminum to generate a brittle and water-soluble compound Al_4C_3 on the surface of the fiber. To prevent this reaction, the carbon fibers are coated with nickel or titanium boride.

The major advantages of MMC's can be listed as follows:

- ❖ High strength even at elevated temperatures
- ❖ High stiffness (modulus of elasticity)
- ❖ High thermal conductivity
- ❖ Excellent abrasion resistance
- ❖ Good creep resistance
- ❖ High wear resistance
- ❖ High thermal stability

Ceramic Matrix Composites (CMC)

Ceramic Matrix Composite (CMC) is a material consisting of a ceramic matrix combined with a ceramic (oxides, carbides) dispersed phase. Ceramic Matrix Composites are reinforced by either continuous (long) fibers or discontinuous (short) fibers [6].

The motivation to develop CMCs was to overcome the problems associated with the conventional technical ceramics like alumina, silicon carbide, aluminum nitride, silicon nitride or zirconia – they fracture easily under mechanical or thermo-mechanical loads because of cracks initiated by small defects or scratches. The crack resistance is very low like in glass. To increase the crack resistance or fracture toughness, particles (so called mono -crystalline whiskers or platelets) were embedded into the matrix.

Some of the typical properties of Ceramic Matrix Composites are:

- ❖ High mechanical strength even at high temperatures

- ❖ High thermal shock resistance
- ❖ High stiffness
- ❖ High toughness
- ❖ Low density
- ❖ High corrosion resistance even at high temperatures

Polymer Matrix Composites (PMC)

Polymer Matrix Composite (PMC) is the material consisting of a polymer (resin) matrix combined with a fibrous reinforcing dispersed phase. Polymer Matrix Composites are very popular due to their low cost and simple fabrication methods [7]. They adopt flat, gently curved or sharply sculpted contours with ease, providing manufacturers with design flexibility. In addition, composites offer the opportunity for parts consolidation and lower assembly costs. Polymer-matrix composites provide a stiff, lightweight alternative to steel, aluminum and traditional materials such as wood.

Reinforcement of polymers by strong fibrous network permits fabrication of Polymer matrix composites (PMC) characterized by the following properties:

- ❖ High tensile strength
- ❖ High stiffness
- ❖ High fracture toughness
- ❖ Good abrasion resistance
- ❖ Good puncture resistance
- ❖ Good corrosion resistance
- ❖ Low cost

But PMC has disadvantages like low thermal resistance and high coefficient of thermal expansion.

Properties of polymer matrix composites are determined by properties of the fiber and matrix and also concentration and orientation of fibers.

Polymer composites are used to make very light bicycles that are faster and easier to handle than standard ones, fishing boats that are resistant to corrosive seawater, and lightweight turbine blades that generate wind power efficiently. New commercial aircraft also contain more composites than their predecessors. A 555-passenger plane recently built by Airbus, for example, consists of 25 percent composite material, while Boeing is designing a new jumbo aircraft that is planned to be more than half polymer composites.

Carbon-Carbon Composites (CCMs)

Carbon fiber-reinforced carbon (carbon-carbon, abbreviated C/C) is a composite material consisting of carbon fiber reinforcement in a matrix of graphite. Carbon-carbon composites are used in very high temperature environments of up to 6000°F and are twenty times stronger and thirty times lighter than graphite fibers. Carbon-carbon is well-suited to structural applications at high temperatures, or where thermal shock resistance and/or a low coefficient of thermal expansion are needed. While it is less brittle than many other ceramics, it lacks impact resistance [5].

The Matrix

The constituent of fiber composites, the matrix serves two very important functions that is it holds the fibrous phase in place and under an applied force it deforms and distributes the stress to the high modulus fibrous constituent. The choice of a matrix for a structural fiber composite is limited by the requirement that it must have a greater elongation at break than the fiber.

The matrix must transmit the forces to the fibers and change shape as required to accomplish this, placing only tensile loading on the fibers. Apart from this it must protect the fibers from environment and abrasion with each other. Furthermore, during processing the matrix must encapsulate the fibrous phase without excessive shrinkage, which can place internal strain on the fibers. The matrix also helps to maintain the distribution of fibers as well as distribution of load evenly between fibers. It enhances some of the properties of the resulting material and structural component (that fiber alone is not able to impart). These properties are such as transverse strength of a lamina and Impact resistance. It also provides better finish to final product [9].

In order to accomplish this transfer of loads and also reduce the chance of failure in the matrix, adhesion to fibers or other reinforcements must be coupled with sufficient matrix shear strength to sustain these loads.

Matrix Material

The four classes of matrix materials are polymers, metals, ceramics and carbon. The properties of the four types differ substantially. These differences have profound effects on the properties of the composites using them. Characteristics of key materials in each class are described below-

Metal Matrix

The metal matrix of MMC is a monolithic material into which the reinforcement is embedded, and is completely continuous. This means that there is a path through the matrix to any point in the material, unlike two materials sandwiched together. The principal matrix materials for MMCs are aluminium and its alloys. To a lesser extent, magnesium and titanium are also used as they provide a compliant support for reinforcement. For several specialized application copper, zinc or lead matrix may be employed. In high temperature applications, cobalt and cobalt-nickel alloy matrices are common. These matrices provide higher transfer strength, toughness but more susceptible to interfacial degradation at the fiber/matrix interface and to corrosion [11].

Ceramic Matrix

The key ceramics used as CMC matrices are silicon carbide, alumina, silicon nitride, mullite, and various cements. The properties of ceramics, especially strength, are even more process sensitive than those of metals. These types of matrices can be used up to high temperature range of $>2000^{\circ}\text{C}$, provides high elastic modulus and low density. Ceramics are very flaw sensitive, resulting in a decrease in strength with increasing material volume, a phenomenon called “size effect.” As a result, there is no single value that describes the tensile strength of ceramics. In fact, because of the very brittle nature of ceramics, it is difficult to measure tensile strength and flexural strength (often called modulus of rupture).

Carbon Matrix

Carbon is a remarkable material. It includes materials ranging from lubricants to diamonds and structural fibers. The forms of carbon matrices resulting from the various carbon/carbon

manufacturing processes tend to be rather weak, brittle materials. This type of matrix provides high heat absorption capacity, chemical inertness. Some forms have very high thermal conductivities. They have exceptional frictional properties at high energy levels and low coefficient of thermal expansion [10].

Polymer Matrix

Polymer-matrix composites (PMCs) use matrices of various thermosets or thermoplastic resin. Thermoplastic soften upon heating and can be reshaped again whereas thermosets becomes cross-linked and do not soften upon heating. Problems with using polymer matrix materials are:

- ❖ Limited temperature range of using.
- ❖ Susceptibility to environmental degradation due to moisture, radiation.
- ❖ Atomic oxygen (in space)
- ❖ Low transverse strength
- ❖ High residual stress due to large mismatch in coefficients of thermal expansion of both fiber and matrix
- ❖ Polymer matrix cannot be used near or above the glass transition temperature.

Types of Polymeric Matrix

A polymeric solid material may be considered to be one that contains many chemically bonded parts or units that themselves are bonded together to form a solid. It is a chemical compound or mixture of compounds consisting of repeating structural units created through a process of polymerization. The atoms in the repeating molecules are strongly bonded (usually with covalent) and the bonds between molecules are weaker secondary bonds or similar covalent bonds.

Plastics can be divided into two classes that are:

1. Thermoplastics and
2. Thermosetting plastic (thermosets)

Depending on how they are structurally and chemically bonded.

Thermoplastics

A thermoplastic is a type of plastic made from polymer resins that becomes pliable or moldable above a specific temperature and solidifies upon cooling. When frozen, however, a thermoplastic

becomes glass-like and subject to fracture. These materials can be reheated and reformed into new shapes a number of times without significant change in their properties [11-12].

Thermoplastics are usually soft and ductile. Most thermoplastics are high-molecular-weight polymers whose chains associate through weak Vander Waals forces (polyethylene); stronger dipole-dipole interactions and hydrogen bonding (nylon); or even stacking of aromatic rings (polystyrene). Many thermoplastic materials are addition polymers; e.g., vinyl chain-growth polymers such as polypropylene.

Since most thermoplastics have weak Van Der Waals forces, the long molecular chains that make up thermoplastics clump together like lumps of entangled spaghetti. These long molecular chains give thermoplastic polymers the flexibility of candle wax [13]. Thermoplastic material provides high delamination resistance (high toughness), damage tolerance, low toxicity, low moisture absorption and high chemical resistance.

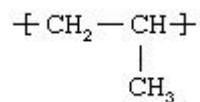
Polypropylene

Polypropylene (PP) is a thermoplastic polymer. One of the most important requirements of the polypropylene used in the manufacture of composites is that, it should be relatively pure and free of residual catalyst. It serves double duty both as a plastic and a fiber. As a plastic it is used in dishwasher safe food container and as fiber it is used in indoor and outdoor carpeting as it eradicates the problem of water absorption that occurs in case of nylon fiber [14].

Molecular structure of polypropylene

Polypropylene (PP), a polymer prepared catalytically from propylene which differs from HDPE by having an isotactic replacement of a hydrogen atom by a methyl group on alternate carbon atoms in the main chain.

Propylene is a gaseous compound obtained by the thermal cracking of ethane, propane, butane, and the naphtha fraction of petroleum. Like ethylene, it belongs to the “lower olefins,” a class of hydrocarbons whose molecules contain a single pair of carbon atoms linked by a double bond. The chemical structure of the propylene molecule is $\text{CH}_2=\text{CHCH}_3$. Under the action of polymerization catalysts, however, the double bond can be broken and thousands of propylene molecules linked together to form a chainlike polymer (a large, multiple-unit molecule). In such a molecule each propylene repeating unit has the following structure [15]:



Essentially, the molecule consists of a backbone of carbon atoms with attached hydrogen atoms; attached to every other carbon atom is a pendant methyl group (CH₃) [15].

Synthesis and Structural Isomers of Polypropylene

The first polypropylene resin was produced by Giulio Natta in Spain, although commercial production began in 1957. Polypropylene can be made from the monomer propylene by Ziegler-Natta polymerization and by metallocene catalysis polymerization as shown in "**Figure 2**" [16].

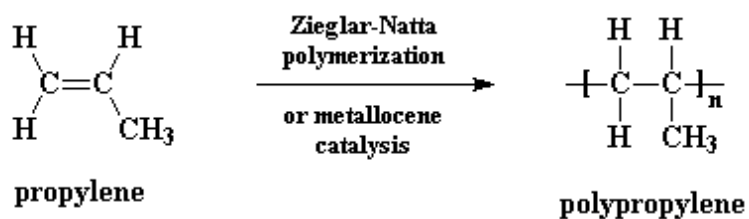


Figure 2: Synthesis of polypropylene [14]

Research is being conducted on using metallocene catalysis polymerization to synthesize polypropylene. Metallocene catalysis polymerization can do some pretty amazing things for polypropylene. The methyl groups can adopt a number of tacticities, or spatial arrangements in relation to the carbon chain, but in practice only the isotactic form (i.e., with the methyl groups arranged along the same side of the chain) is marketed in significant quantities [16].

But sometimes we use atactic polypropylene. Atactic means that the methyl groups are placed randomly on both sides of the chain "**Figure 3**"

In syndiotactic polypropylene the methyl groups are on alternate sides of the chain as shown in "Figure 3"

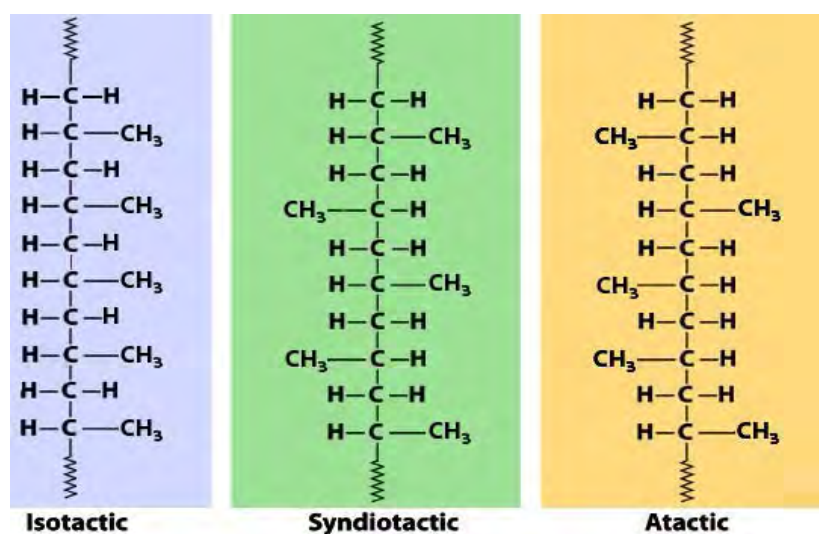


Figure 3 : Isotactic, syndiotactic and atactic polypropylene [17]

However, by using special metallocene catalysts, it's believed that we can make polymers that contain blocks of isotactic polypropylene and blocks of atactic polypropylene in the same polymer chain [18].

Properties of PP

Most commercial polypropylene is isotactic and has an intermediate level of crystallinity between that of low-density polyethylene (LDPE) and high-density polyethylene (HDPE). The crystalline and nonpolar nature of PP confers good resistance to a wide range of aqueous and polar media, including emulsifier solutions with their strong stress cracking abilities [19].

Polypropylene is normally tough and flexible, especially when copolymerized with ethylene. Polypropylene is reasonably economical, and can be made translucent when uncolored. It is often opaque or colored using pigments. Polypropylene has good resistance to fatigue.

Perfectly isotactic PP has a melting point of 171°C (340 °F). Commercial isotactic PP has a melting point that ranges from 160 to 166 °C (320 to 331 °F), depending on atactic material and

crystallinity. Syndiotactic PP with a crystallinity of 30% has a melting point of 130 °C (266 °F). Its crystallinity is around 82%.

There are three general types of polypropylene: homopolymer, random copolymer, and block copolymer.

Some typical properties of PP are given in **Table 1**.

Table 1 Properties of (polypropylene) PP [20].

Thermoplastic				
Polypropylene (PP)				
Property	Value in metric unit		Value in US unit	
Density	0.91 *10 ³	kg/m ³	56.8	lb/ft ³
Modulus of elasticity	1.36	GPa	195	ksi
Tensile strength	37	MPa	5300	psi
Elongation	350	%	350	%
Flexural strength	49	MPa	7000	psi
Thermal expansion (20 °C)	90*10 ⁻⁶	°C ⁻¹	50*10 ⁻⁶	in/(in* °F)
Thermal conductivity	0.16	W/(m*K)	1.11	BTU*in/(hr*ft ² *°F)
Glass transition temperature	-10	°C	14	°F
Maximum work temperature	150	°C	302	°F
Electric resistivity	10 ⁷ -10 ⁹	Ohm*m	10 ⁹ -10 ¹¹	Ohm*cm
Dielectric constant	2.4	-	2.4	-

Chemically the presence of the methyl groups makes polypropylene slightly more susceptible to attack by strong oxidizing agents than HDPE. The major advantages of Polypropylene's are higher temperature resistance, high corrosion resistance, high abrasion resistance, excellent tensile strength, high thermal insulating properties and high dielectric properties. Polypropylene is a translucent material. Many additives have been developed to enhance the thermal stability of polypropylene to minimize degradation during processing.

Thermosetting Plastic

A thermosetting plastic, also known as a thermoset, is polymer material that irreversibly cures. It is a prepolymer in a soft solid or viscous state that changes irreversibly into an infusible, insoluble polymer network by curing. Curing can be induced by the action of heat or suitable radiation, or both. Thermosetting Plastics are made up of lines of molecules which are heavily

cross-linked. It creates a rigid molecular structure. They may be heated the first time and shaped but they become permanently stiff and solid. They cannot be reshaped again [11].

Most thermosets consist of a network of carbon atoms covalently bonded to form a rigid solid. Sometimes nitrogen, oxygen, sulfur or other atoms are also covalently bonded into a thermoset network structure. Thermosets are generally stronger than thermoplastics due to their three dimensional network of bonds and better suited to high temperature applications.

Advantages of Thermoplastics over Thermosets

As stated earlier, thermoplastics can be repeatedly softened by heating, and hardened by cooling. Thermoplastics possess several advantages over the thermosets, one of the most important being that they do not need storing under refrigeration. They also possess improved damage tolerance, environmental resistance, fire resistance, recyclability and potential for fast processing. Primary reason for the use of thermoplastics is their cost effective processing. Increased use for thermoplastics is also due to three different reasons as stated below:

- ❖ Processing can be faster than that of thermoset composites since no curing reaction is required. Thermoplastic composites require only heating, shaping and cooling.
- ❖ The properties are attractive, in particular, high delamination resistance and damage tolerance, low moisture absorption and the excellent chemical resistance of semi-crystalline polymers.
- ❖ In the light of environmental concerns, thermoplastic composites offer other advantages also. They have low toxicity since they do not contain reactive chemicals (therefore storage life is infinite) [21].
- ❖ Using of thermoplastic provides aesthetically superior finishes and has high impact resistance.

Reinforcement of Composite Material

The role of the reinforcement in a composite material is increasing the mechanical properties of the neat resin system. Fibers or reinforcements carry load along the length of the fiber to provide strength and stiffness in one direction. Reinforcements can be oriented to provide tailored properties in the direction of the loads imparted on the end product. All of the different fibers

used in composites have different properties and so affect the properties of the composite in different ways [20].

However, individual fibers or fiber bundles can only be used on their own in a few processes such as filament winding. For most other applications, the fibers need to be arranged into some form of sheet, known as a fabric, to make handling possible. Different ways for assembling fibers into sheets and the variety of fiber orientations possible lead to there being many different types of fabrics, each of which has its own characteristics. The four main factors that govern the fiber's contribution are:

- ❖ The basic mechanical properties of the fiber itself.
- ❖ The surface interaction of fiber and resin (the 'interface').
- ❖ The amount of fiber in the composite ('Fiber Volume Fraction').
- ❖ The orientation of the fibers in the composite [20].

Natural Fiber Reinforced Polymer Composite

By natural fiber composites we mean a composite material that is reinforced with fibers, particles or platelets from natural or renewable resources. Natural fibers will play a key role in the emerging "green" economy based on energy efficiency.

Natural fibers are ecofriendly because of their renewability. They are also carbon neutral. They absorb the same amount of carbon dioxide they produce. During processing, they generate mainly organic wastes and leave residues that can be used to generate electricity or make ecological housing material. And, above all they have the superior quality of biodegradability [4].

An FAO study estimated that production of one tone of jute fiber requires 10% of the energy used for the production of one tone of synthetic fibers (since jute is cultivated mainly by small-scale farmers in traditional farming systems, the main energy input is human labor, not fossil fuels). Processing of some natural fibers can lead to high levels of water pollutants, but they consist mostly of biodegradable compounds, in contrast to the persistent chemicals, including heavy metals, released in the effluent from synthetic fiber processing. Natural fibers have good mechanical strength, low weight and low cost. Natural fibers are now -a -days are quite an attractive choice in automobile industry. In Europe, car makers are using an estimated 80, 000

tons of natural fibers a year to reinforce thermoplastic panels. India has developed composite boards made from coconut fiber that are more resistant to rotting than teak [22].

Advantages of natural fiber reinforced composites:

- ❖ Reduction in density of products.
- ❖ Acceptable specific strength, toughness and stiffness in comparison with glass fiber reinforced composites.
- ❖ Ease of shaping into complex shapes in a single manufacturing process.
- ❖ Lower energy consumption from fiber growing to finished composites.
- ❖ The manufacturing processes are relatively safe when compared with glass based reinforced composites.
- ❖ huge possibility of recycling the cuttings and wastage produced during manufacturing and moulding.
- ❖ The production of natural fibers can be started with a low capital investment and with a lower cost.

Among various natural fibers, betel nut (areca) fiber is quite available and cheap in context to the economic condition of Bangladesh.

Natural Fiber vs. Synthetic Fiber

Both natural and synthetic fibers have advantages and drawbacks. Synthetic fiber-reinforced thermoplastic composites have better mechanical properties than the natural fibers, but they are not environmentally friendly. Some advantages of natural fibers are low abrasion resistance, low density, high toughness, acceptable specific strength properties, good thermal properties, enhanced energy recovery, biodegradability and so on [4] . Natural fibers produce composites that offer advantages like renewability of the fibers, good sound abatement capability and improved fuel efficiency. On the other hand, synthetic FRPCs attracted much attention due to their high strength, better durability and moisture resistance properties. The mostly used synthetic fibers in composites are glass, carbon and aramid. Among the synthetic fibers, glass fibers are widely used due to their low-cost (compared to carbon and aramid) and better physico-mechanical properties. Besides, the greatest problem of using such materials is the convenient disposal of themselves, once they have come to the end of their useful life span [23].

Textile Waste Fiber Reinforced Composite Materials

According to the Secondary Materials and Recycled Textiles Association (SMART) and the Council for Textile Recycling, in the USA alone, more than one thousand businesses and organizations employing many tens of thousands of workers to divert some 2 million tons of textile waste from the solid waste stream [24].

Textile wastes are of two types: pre-consumer and post-consumer. Pre-consumer textile waste generally consists of by-product materials from the textile, fiber and cotton industries. Each year 750,000 tons of this waste is reused to produce raw materials for the automotive, furniture, mattress, coarse yarn, home furnishings, paper and other industries. Approximately 75 percent of the pre-consumer textile waste is recycled and reused. Post-consumer textile waste consists of any type of garments or household article, made of some manufactured textile. Approximately 1,250,000 tons of post-consumer textile waste (4.5 kg per capita) is recycled annually which represents less than 25 percent of the total post-consumer textile waste that is generated and the remaining 75 percent are disposed into the trash and end up in municipal landfills [24].

There are several disadvantages associated with the land filling of fibrous waste [2]. The textile waste products can be considered as an environmental nuisance because of its non-biodegradability. The accumulation of textile waste in landfills has harmful effects on the aesthetic qualities of cities and green zones [1].

By considering all of these aspects, in recent years, increased emphasis has been placed on reusing techniques for various fibrous waste textile products. Rising environmental concerns have directed research to the development of alternative materials which serves a dual function: elimination of wastes and production of ecologically useful new material. Industries and research organizations are now looking for applications where waste materials may represent an added-value material.

Though a variety of technologies have been developed in response to customer demands for recycled products and as alternatives to land filling [25-27], one of the most viable application of these waste materials is in the combination of polymeric matrices, producing composite materials with interesting properties for specific applications, from structural to thermal and acoustic insulations [28]. The advantages of reusing waste fibers by such techniques include

generally lower cost to process than virgin fibers and the elimination of the need for waste disposal in landfills [29].

Several studies have been carried out on utilizing different types of fibers as efficiently as possible to produce superior quality fiber-reinforced polymer composites for a wide range of applications [30]. The mechanical properties of low-density polyethylene composite consisted of waste carpet as the reinforcement element were measured [30-31]. The usefulness of cotton waste as a source of reinforcing fibers for the preparation of cost-effective and biodegradable composites has also been studied [31-32].

Areca and Waste Nylon Fiber Reinforced Hybrid Composite

Hybrid Composites

The incorporation of several different types of fibers into a single matrix has led to the development of hybrid composites. The behavior of hybrid composites is a weighed sum of the individual components in which there is a more favorable balance between the inherent advantages and disadvantages. Also, using a hybrid composite that contains two or more types of fiber, the advantages of one type of fiber could complement with what are lacking in the other. As a consequence, a balance in cost and performance can be achieved through proper material design. The properties of a hybrid composite mainly depend upon the fiber content, length of individual fibers, orientation, extent of intermingling of fibers, fiber to matrix bonding and arrangement of both the fibers [33].

Hybrid composite Materials have extensive engineering application where strength to weight ratio, low cost and ease of fabrication are required. Hybrid composites provide combination of properties such as tensile modulus, compressive strength and impact strength which cannot be realized in composite materials. In recent times hybrid composites have been established as highly efficient, high performance structural materials and their use is increasing rapidly. Hybrid composites are usually used when longitudinal as well as lateral mechanical performances are required.

There is a steady increase both in the number of applications being found for fiber reinforced plastics and, concurrently, in the variety of fiber/resin systems that are available to designers. As a result these systems provide wide range for designers to tailor the properties of such materials

by using hybrid composite to suit specific requirements. In highly specialized situations where limitations such as high cost and brittle fracture behavior are considered secondary to such qualities such as low density, high rigidity and high strength hybrid composites have found wide application. There are many situations in which, for example, a high modulus material is required but in which the catastrophic brittle failure usually associated with such a material would be unacceptable. In the case of a strut member, a high initial modulus followed by limited yielding of the material and accompanied by the smallest possible reduction of load carrying capacity is usually desirable.

Hybrid composites can be made in two separate ways either by intimately mingling the fibers shown in a common matrix, or by laminating alternate layers of each type of composite. In principle several different types of fiber can be incorporated into a hybrid system. But in practice it is likely that a combination of only two types of fiber would be of most [33].

Areca Fiber

The areca nut is the seed of the areca palm (*Areca catechu*), which grows in much of the tropical Pacific, Asia, and parts of east Africa. It is commonly referred to as betel nut, as it is often chewed wrapped in betel leaves.

The areca nut is not a true nut, but rather a fruit categorized as a drupe. It is commercially available in dried, cured and fresh forms. When the husk of the fresh fruit is green, the nut inside is soft enough to be cut with a typical knife. In the ripe fruit, the husk becomes yellow or orange and, as it dries, the fruit inside hardens to a wood-like consistency [34].

The betel nut husk is the fibrous part of the fruit, which equals to approximately 60 to 80 % of total volume and weight of betel nut.

2.6.2.1 Chemical Composition of Betel nut (Areca) Fiber

Composition of Betel Nut (Areca) fiber is as follows: [35]

Component	Weight (%)
lignin	7.20
α -cellulose	53.20
Hemicellulose	32.98
pectin	9.2-15.4
Ash	1.05
Waxy materials	0.08
Other materials	3.04

Comparison of areca fiber properties with other natural fibers

Properties of areca fiber in comparison to other fibers are shown below [36-37]

Properties	Areca fiber	Coir fiber	Banana fiber	Sisal fiber	Flax fiber	Jute fiber
Density(g/cm ³)	0.34	1.15	1.35	1.45	1.4-1.5	1.3-1.45
Diameter(mm)	0.45-0.53	0.1-0.45	0.08-0.25	0.05-0.20		0.025-0.2
Tensile Strength(MPa)	166.03	106-175	54-754	568-640	500-900	393-773
Young's Modulus(GPa)	1.381	4-6	7.7-20	9.4-15.8	50-70	13-26.5
Elongation at break [%]	23.21	17-47	10-35	3-7	3.27	1.16-1.5

Nylon Fiber

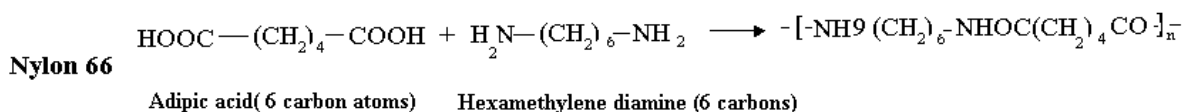
Nylon was the first truly synthetic fiber to be commercialized (1939). Nylon was developed in the 1930s by scientists at Du Pont, headed by an American chemist Wallace Hume Caruthers (1896-1937). It is a polyamide fiber, derived from a diamine and a dicarboxylic acid. Nylons are

condensation copolymers, formed by reacting difunctional monomers containing equal parts of amine and carboxylic acid, so that amides are formed at both ends of each monomer in a process analogous to polypeptide biopolymers. Nylons made from the reaction of a dicarboxylic acid with a diamine is called nylon 66 (poly hexamethylene adiamide) (e.g. PA66) and a lactam or amino acid with itself is called nylon 6 (Polycaprolactam, a cyclic nylon intermediate) (e.g. PA6) [38].

The fiber has outstanding durability and excellent physical properties. Nylons are semi-crystalline polymers. The amide group (- (-CO-NH-) -) provides hydrogen bonding between polyamide chains, giving nylon high strength at elevated temperatures, toughness at low temperatures, combined with its other properties, such as stiffness, wear and abrasion resistance, low friction coefficient and good chemical resistance. These properties have made nylons the strongest of all man-made fibers in common use. Because nylons offer good mechanical and thermal properties, they are also a very important engineering thermoplastic. For example, 35% of total nylon produced is used in the automobile industry [38].

2.6.3.1 Nylon 66:

Nylon 66 structure is so-called ABAB structure similar to polyesters and polyurethanes: the "repeating unit" consists of one of each monomer, so that they alternate in the chain. Since each monomer in this copolymer has the same reactive group on both ends, the direction of the amide bond reverses between each monomer. The raw materials for these are variable and sources used commercially are benzene (from coke production or oil refining), furfural (from oat hulls or corn cobs) or 1, 4-butadiene (from oil refining). The chemical reactions are as follows [38]:



Nylon 66 is a semi-crystalline polymer. These linear aliphatic polyamides are able to crystallize mostly because of strong intermolecular hydrogen bonds through the amide groups, and because of Vander walls forces between the methylene chains. For the form of nylon 6, the chains are parallel and the hydrogen bonding is between chains in adjacent sheets. In nylon 66, the chains

have no directionality. Research results have shown that the stable crystalline structure is the - form comprised of stacks of planar sheets of hydrogen-bonded extended chains [38].

Nylon 66 can have multiple parallel strands aligned with their neighboring peptide bonds at coordinated separations of exactly 6 and 4 carbons for considerable lengths, so the carbonyl oxygen's and amide hydrogen's can line up to form inter chain hydrogen bonds repeatedly, without interruption.

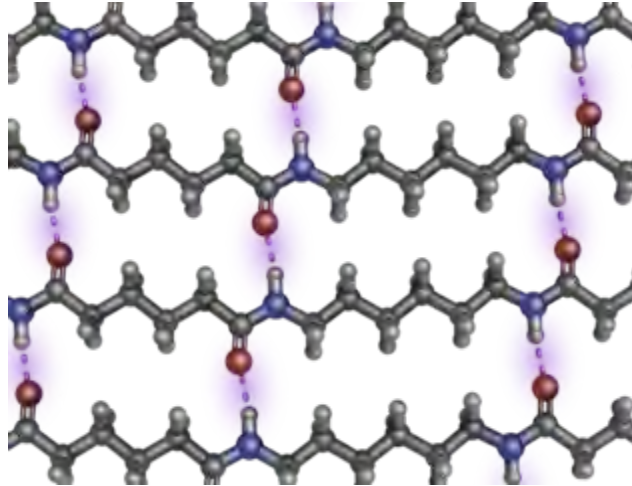


Figure 4 : Hydrogen bonding in Nylon 6, 6 [39]

Properties of Nylon 66 [38]:

- ❖ Tenacity-elongation at break ranges from 8.8g/d-18% to 4.3 g/d-45%. Its tensile strength is higher than that of wool, silk, rayon, or cotton.
- ❖ 100% elastic under 8% of extension
- ❖ Specific gravity 1.14
- ❖ Extremely chemically stable
- ❖ No mildew or bacterial effects
- ❖ 4 - 4.5% of moisture regain
- ❖ Abrasion resistant
- ❖ Lustrous- Nylon fibers have the luster of silk
- ❖ Easy to wash
- ❖ Can be pre colored or dyed in wide range of colors; dyes are applied to the molten mass of nylon or to the yarn or finished fabric.
- ❖ Resilient

- ❖ Filament yarn provides smooth, soft, long lasting fabrics
- ❖ Spun yarn lend fabrics light weight and warmth
- ❖ Pleats and creases can be heat-set at higher temperatures
- ❖ More compact molecular structure
- ❖ Better weathering properties; better sunlight resistance [36].
- ❖ High melting point (256 °C/492.8 °F)
- ❖ Superior color fastness

2.6.5 Properties of Areca and Nylon fiber:

Properties of areca and waste nylon fiber are given below [40-41] :

Properties	Areca fiber	Nylon fiber
Density(g/cm ³)	0.34	
Diameter(mm)	0.45-0.53	
Tensile Strength(MPa)	166.03	82
Young's Modulus(GPa)	1.381-1.93	2-3.6
Elongation at break [%]	23.21	50-60

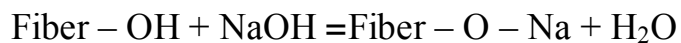
Interphase and Chemical Treatment:

The main component of natural fiber is cellulose. The elementary unit of cellulose macromolecule is anhydro-d-glucose, which contains 3 hydroxyl group (-OH). These hydroxyl form hydrogen bond inside the molecule itself (intra molecular) and between other cellulose molecules (intermolecular), as well as with other hydroxyl group from the air. Therefore all natural fibers are hydrophilic that is they absorb water.

Natural fiber due to its hydrophilic nature, leads to an adhesion problem with hydrophobic nature of the polymer matrix. Hydrophilic character of natural fibers is incompatible with hydrophobic polymer matrix and has a tendency to form aggregates [4]. Furthermore, it exhibit poor resistant to moisture, which lead to high water absorption, subsequently resulting in poor mechanical

properties and dimensional stability of the natural fiber reinforced composites. Therefore, chemical modifications of either natural fiber, polymer matrix or both materials are the alternative solutions to overcome these challenge. The chemical modification is attempted to improve hydrophobic nature of natural fiber, interfacial bonding between matrix and fiber, surface roughness and wet ability, and also decrease moisture absorption which leads to the enhancement of mechanical properties of the natural fiber reinforced composites.

Alkaline treatment or mercerization is one of the most used chemical treatments of natural fibers when used to reinforce thermoplastics and thermo sets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites [42]. Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide:



Thus, alkaline processing directly influences the cellulosic fibril, the degree of polymerization and the extraction of lignin and hemi cellulosic compounds. It is reported that alkaline treatment has two effects on the fiber:

1. It increases surface roughness resulting in better mechanical interlocking;
2. It increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites.

Consequently, alkaline treatment has a lasting effect on the mechanical behavior of fibers, especially on fiber strength and stiffness.

Apart from alkali treatment various type of chemical treatment has carried out by other researchers. In one study abaca fiber was treated with benzene diazonium salt [43], Jute fibers were chemically treated with Copper sulfate (CuSO₄) and sodium carbonate (Na₂CO₃) for rot retardant (RR) treatment, diammonium hydrogen phosphate (NH₄H₂PO₄) and lissapol for fire retardant (FR) treatment [44], flax was treated with propyl-trimethoxysilane, phenylisocyanate and maleic acid anhydride modified polypropylene (MAA-PP) [45]. Coir was chemically treated

with stearic acid as compatibilizer [46], and in one case jute fiber was chemically modified by using saline coupling agent [47].

Review of Previous Researchers:

Composite materials with thermoplastic matrices and a reinforcement of natural fiber was always regarded as an important structural application source of biodegradable and renewable material. A large number of natural fibers like coir, jute, abaca, areca, flax, sisal has studied as an incorporation in thermoplastic polymeric matrices most commonly polypropylene.

Mir, Samia Sultana, et al. studied coir fiber reinforced polypropylene composites. In order to increase the compatibility between the coir fiber and polypropylene matrix, raw coir fiber was chemically treated with basic chromium sulfate and sodium bicarbonate salt in acidic media [48]. In another research Rahman, MdRezaur, et al. studied the mechanical properties of polypropylene composites reinforced with chemically treated abaca where it was treated with benzene diazonium salt in order to improve in the mechanical properties of the abaca-PP composites [43]. Both raw and treated abaca samples were utilized for the fabrication of the composites. Jute fiber was also studied in quite a lot of researches. Researchers Rana, A. K., A. Mandal, and S. Bandyopadhyay. studied polypropylene composites reinforced with jute fiber and using compatibilizer and impact modifier [49]. SweetyShahinur et al studied physico mechanical properties of jute fiber reinforced MAgPP green composites and the effect of chemical retardent in improving its properties [44]. Van de Velde, Kathleen, and Paul Kiekens studied the effect of both fiber and matrix modification in obtaining the optimal mechanical characteristics of flax polypropylene composites [45]. Recently areca fibers are using as a new reinforcing materials by many researchers. Srinivasa, C. V., et al. evaluate physical, flexural and impact properties of composites reinforced by areca fibers with urea formaldehyde, melamine urea formaldehyde and epoxy resins by means of compression molding technique [34].

Not only polypropylene matrices but also a large number of thermoset matrices has already been used as matrix materials. In another study Enriquez, Joseph King Eos Dawn V., et al. studied coir reinforced high density polyethylene composites. In these research stearic acid was used as the compatibilizer [46]. Doan, Thi Thu Loan. studied jute fiber reinforced epoxy composites and also tried to compare their property with composite prepared using polypropylene matrix [50]. In

another study Rokbi, Mansour, et al. studied effect of chemical treatments on flexural properties of natural fiber reinforced polyester composites [51]. Jayamani, Elammaran, et al. Investigated effect of Fiber Surface Treatment by sodium hydroxide on Mechanical, Acoustical and Thermal Properties of Betelnut Fiber reinforced Polyester Composites [52].

Hybrid composites are recently getting considerable attention from worlds scientific community. Using both natural fiber as reinforcing materials provide a biodegradable material with a favourable balance of properties. Siddika, Salma, et al. studied the physico-mechanical properties of coir-jute fiber reinforced hybrid polypropylene composites .They also tried to improve their properties by chemical treatment [37]. Hatta, Mohd, Mohamed Nasrul, and Nairul Akmar Akashah. also studied coir jute fiber reinforced composites but change the matrix material by polystyrene [47]. Reddy, G. RAMACHANDRA, A. M. Kumar, and K. V. P. Chakradhar studied performance of Hybrid Betel Nut (Areca Catechu) Short Fiber/Sansevieria Cylindrica (Agaveceae) reinforced Epoxy Composites [35].

Reinforcing with a natural and synthetic fiber provides a favourable balance of both of their properties .Though natural fibers are extremely acceptable for their characteristic renewability and biodegradability ,synthetic fiber are quite exceptional in terms of their extraordinary mechanical properties. Joseph, Seena, et al. compared the mechanical properties of phenol formaldehyde composite reinforced with banana fibers and glass fibers [53]. In another study H.P.S Abdul Khalil .et al. studied oil palm fiber/glass hybrid reinforced polyester composites [54] . Researchers also studied flax/glass ,carbon/glass,jute /glass and basalt/glass reinforced hybrid composites [55-58].Schroder, N., et al. studied the effect of more than two fiber reinforcement by incorporating glass beads and alpha, omega-oligo (butylmethacrylate) diol in epoxy matrix [59].

Premalal, Hattotuwa GB, Hanafi Ismail, and A. Baharin. studied Unmodified and ground talc and rice husk (RHP) reinforced polypropylene composite [4]. Effects of rice husk filler on the mechanical and thermal properties of liquid natural rubber compatibilized high-density polyethylene/natural rubber blends were investigated by Jamil, MohdSuzeren, Ishak Ahmad, and Ibrahim Abdullah [60]. Now-a -days nanostructured fillers are also been used as the way of reinforcement. In one study Luyt, A. S., et al. evaluated mechanical and thermal properties of nanostructured wollastonite filler reinforced polypropylene composites [61]. MAM Mohd, et

al.studied mechanical and morphological properties of treated tropical wood sawdust polypropylene composites [62].

Properties of waste fiber-reinforced PP composites (FRPCs) have studied by many researchers. Much of the research has concentrated on determining the physical and mechanical properties of waste fiber reinforced composites and the influence of different parameters on their mechanical behaviour. The results show that a wide range of different properties and performance may be designed by altering various production parameters, such as thickness of the fibers used, relationship between fiber/matrix ratio, polymeric film used and number of layers.

Kim and Moonb *et al.* studied the mechanical properties of polypropylene (PP)/waste cotton fiber composites [63]. The effect of the melt index of PP on the mechanical properties of the composites was also investigated. Composites containing cotton fabric and cotton textile waste as reinforcements and phenol phormaldehyde resin as matrix has been studied by Krsteva and Srebrenkoska *et al* [64]. For the composites, the mechanical and thermal properties were analyzed and compared to those of commonly used continuous fiber reinforced composites based on cotton fabric and phenolic resin.

According to the study of Tasdemir and Kokac *et al* Silk and cotton waste and recycled PA6 polymer was mixed and a composite structure was obtained .The mixture was made by twin screw extruder [65]. The usefulness of cotton waste as a source of reinforcing fibers for the preparation of cost-effective and biodegradable composites has also been investigated [66]. Biodegradable polyester (bionolle 3020) was melt-compounded together with cotton fibers. Maleic anhydride-grafted bionolle (bionolle-g-MA) was used as a compatibilizer an intensive mixer in the presence of dicumyl peroxide as initiator.

2.8 Feasibility of Preparing Areca and Waste Nylon Fiber Reinforced Hybrid Polypropylene Composites :

Asia continues to be the center of the worlds nylon fiber manufacturing industry. In 2013, it accounted for two-thirds of the worlds nylon fiber output. As production has continued to increase in China, most other Asian countries, such as Japan, Taiwan and India, have experienced no growth to slow declines. Output in Other Asia, which includes smaller producing countries such as Malaysia, Pakistan and Sri Lanka, increased at double-digit rates in 2010–2012

largely as a result of the start-up of new capacity in Vietnam. Overall Asian output is forecast to increase at an average annual rate of 2–3%, matching projected regional demand growth [67].

Nylon fibers consumed in textile applications remained the dominant end use in 2013. Nylon fiber demand in both textile and industrial yarn applications accounted for nearly 80% of total demand globally.

It is estimated that 4–5 billion pounds (1.8–2.3 million metric tons) of carpet are dumped into U.S. landfills each year. Nylon carpets and rugs are the largest end use for nylon fibers. End scraps and off-spec runs of nylon fibers have been recycled for many years, but it has only been during the last fifteen years or so that postconsumer nylon carpets have been collected and reclaimed. Independent recycling companies have established regional collection and processing centers to recycle postconsumer carpeting back to fiber, which can then be used as stuffing or batting for industrial uses and as filler in composite applications. There are currently about fifty local carpet collector companies in the United States that collect, sort and find buyers for used carpeting [67].

Aside from the carpet industry, molding resin compounders have also discovered that the lower prices of the regenerated nylon resins allow nylon to compete on a cost basis in some lower-cost resin applications, particularly in the automotive industry. This in turn is expanding the market for compounded nylon products and creating a growing demand for regenerated nylon resins. Improved extrusion systems have also helped by making it even easier for compounders and molders to incorporate a wide range of regenerated resin grades in mixed-stream extrusions.

World demand for nylon fibers is forecast to grow only slowly, at 2% per year, averaged annually. In China, growth is forecast to be only about 3.2% per year [68].

According to a study conducted by Economics of arecanut cultivation in Karnataka, India in 2001 77.80 HA areas of Bangladesh were used for areca cultivation and the total production was 47 tonnes and at 2009 The total area of areca cultivation enhanced to 79 HA and total production increased to 56 tonnes So the productivity increase was 604 Kg/HA to 709 Kg/HA [69].

According to today's costing nylon 66 filament waste costs US \$1.8-2.5 / Kilogram [70]. So around 140-190 BDT/Kg, whereas in local markets betel nut fruit costs around 400 BDT/Kg. If we want to prepare a composite sample weighing 1 kg, then according to the experimental result

20% (areca: nylon=1:1) reinforced composite provides best results. So requirements of fiber will be 20% of 1 kg that is 200 gm. (1:1) ratio means 100 gm areca and 100 gm nylon fiber. 1 areca nut fruit is around 6 gm in weight where recoverable fiber from husk is around 1.5 gm. So 100 gm areca fiber requires $(6 \times 100 / 1.5) = 400$ gm areca nut fruit. So taking the cost as 400 BDT for 1 kg, the amount requires $(400 \times 400 / 1000)$ i.e 160 BDT. According to the price of nylon fiber 100 gm costs 1.4-1.9 tk. 1 kg PP sheets costs around 120 BDT so 800 gm means 96 BDT . So 1 kg composite preparation will cost in terms of raw materials $(96 + 160 + 1.4)$ BDT=257.4 BDT .

2.8 Probable Application of Areca and Waste Nylon Fiber Reinforced Hybrid Composites:

Manufacturing companies are in always in constant search of new materials of lower costs and profit margins. Natural fibers are low-cost fibers with high specific properties and low density. Performance characteristics that predestine polymer use in automotive applications include corrosion resistance, low density, good impact toughness and chemical resistance.

In 1986, a study has been published where it is reported that coir/polyester composites have been used to produce mirror casing, paper weights, projector cover, voltage stabilizer cover, mail-box, helmet and roof [71]. The property of areca fiber in many cases are close to coir fiber properties. So this composite will hopefully can be used in those applications.

A composite of woven jute fabric (burlap) and soybean oil based resin system has been successfully used to develop an I-shaped beam using vacuum assisted resin transfer molding (VARTM) method so this composite can also be used in beam manufacturing [72].

The widespread application of natural fiber is in automotive industry. Plant fibers are mainly used in the part of car interior and truck cabins. The use of plant fiber based automotive parts such as various panels, shelves, trim parts and brake shoes are attractive for automotive industries worldwide because of its reduction in weight about 10%, energy production of 80% and cost reduction of 5%. For those various part manufacturing this composite can also be considered [73].

The major car manufacturers like Volkswagen, BMW, Mercedes, Ford and Opel now use natural fiber composites in applications .BMW has been using natural materials since the early 1990's in the 3, 5 and 7 series models with up to 24 kg of renewable materials being utilized. In 2001,

BMW used 4 000 tonnes of natural fibers in the 3 series alone. The combination here is a 80% flax with 20% sisal blend for increased strength and impact resistance. The main application is in the interior door linings and paneling. Wood fibers are also used to enclose the rear side of seat backrests and cotton fibers are utilized as a sound proofing material [73].

Recently, Volvo has started to use soya based foam linings in their seats along with natural fibers. They have also produced a cellulose based cargo floor tray which resulted in improved noise reduction.

CHAPTER 3

EXPERIMENTAL PROCEDURE

EXPERIMENTAL PROCEDURE

Fiber Extraction and Collection of Polypropylene Resin

Commercial grade polypropylene (PP) sheets were used in this study. Polypropylene sheets were collected from the local market. The PP was white in color having a melting point of 160°C. Waste nylon fibers were collected from a local textile industry and they were chopped in the size of 3-4 mm.



Figure 5 : Commercial polypropylene (PP) sheet

Extraction of areca nut fiber

The dried areca empty fruit was soaked in water for about seven days. This soaking process loosens the fiber allowing it to be removed from the fruit easily. This process is called retting. The fibers were removed from the fruit. After drying in the room temperature, the fibers were further separated into an individual state using hand.



Figure 6 : Betel nut fruit



Figure 7 : Extracted areca nut fiber



Figure 8 : Commercial nylon fiber

chemical Treatments

Alkali Treatment

Alkali treatment of areca fibers was carried out to enhance its compatibility with the polypropylene matrix. Fibers were treated with 5% NaOH.

Firstly 5% NaOH solution was prepared into a beaker. Solution volume was taken as 15 times of the volume of areca fiber. Then the dried extracted fiber were submerged into the solution and stirred properly, so that all fibers were wetted by the solution. Then the beaker containing the solution with fiber was taken to the oven for heating. It was maintained at 60°C for about 24 hours.

After that the beaker was taken out from the oven and then neutralized by acid solution. The fiber was washed with clean water. They were then heated at 100°C for about two hours inside an oven for the complete evaporation of moisture and then removed from the oven.

Composite Manufacturing

At the first stage hybrid composites of polypropylene matrix reinforced with 10 wt% of fiber was prepared by reinforcing the mixture of areca and nylon fibers with a ratio of (1:1) in 2,3 and 4 different layers. In another experiment areca fibers were alkali treated to improve its compatibility with polypropylene matrix and composites were prepared in second stage by changing the fiber wt% in four different values 10, 15, 20, 25 and in all cases fibers were introduced in 4 different layers keeping the treated areca and nylon fiber ratio constant as(1:1).At the last stage impact of changing individual fiber percentages were studied by changing areca: nylon ratio as (1:3), (3:1), (2:3) and (3:2).In all cases the total fiber percentage was kept constant in 20wt%.

The following procedure was followed for the manufacturing of Hybrid composites of polypropylene matrix reinforced with areca and waste nylon fibers in a 150×150×5 mm die using a hot press machine having maximum load of 35 kN and maximum temperature of 300 °C:

- ❖ At first fibers were weighed according to the required weight % needed. Then both fibers were cut into 3-5 mm size in length. Then to remove adhered moisture, fibers were dried in an oven at 80°C for 20 minutes before preparing each composite.
- ❖ Mold surface was cleaned very carefully and mold releasing agent was sprayed over the mould surface properly for the easy removal of the composite after the production procedure.
- ❖ In 4 layered fibers reinforced composite, properly weighed fiber percentage was divided into four equal halves and the PP sheets were divided into five equal plies. After that placing of one ply of PP sheets and one fiber layer one above another inside the die, the composite lamina was arranged into the die. Both the fibers were premixed in order to ensure better distribution of both fibers into the matrix.
- ❖ Then the lower part of the die containing arranged lamina was covered with the upper part of the die according to the indication provided in both part of the die.
- ❖ Then the die "**Figure 9**" was placed in a hot pressing machine.
- ❖ The fiber-matrix mixture was allowed to press at 30kN pressure. The temperature was initially raised to 160°C and hold there for around 12-15 minutes, after that the temperature was raised to (180-185)°C depending on the thickness required.
- ❖ The die was cooled to room temperature, pressure was released and the specimen was carefully withdrawn from the die.
- ❖ The same procedure was applied for preparing composites containing 2 and 3 layers of fiber and also the composites containing different percentages of fibers.

Since the compression temperature was higher than the melting point of PP (160⁰C), the matrix melted however the fibers (melting point > 220⁰C) remained intact.

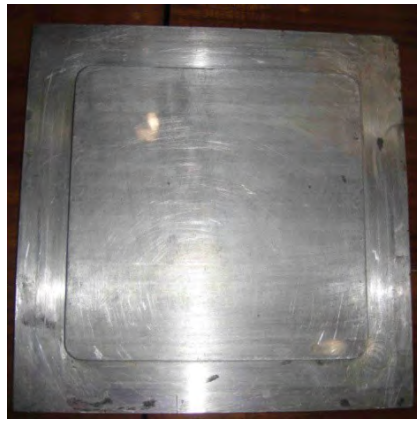


Figure 9 : Die used for composite manufacturing

Tensile Test

Tensile tests were conducted according to ASTM D 638-01 [74] using an Instron UTM machine (system Id 3369J8567, Maximum capacity 50KN) . Each test was continued until tensile failure. Tensile properties as determined by the test method helps to know the stress of the composite specimen, their fracture characteristics under tensile load, load strain behavior bearing properties of fiber and the matrix materials.

Specimen preparation

Test specimens of required shape were cut from the composite part. The specimen dimension was given according to the ASTM D 638-01 [74] as following:

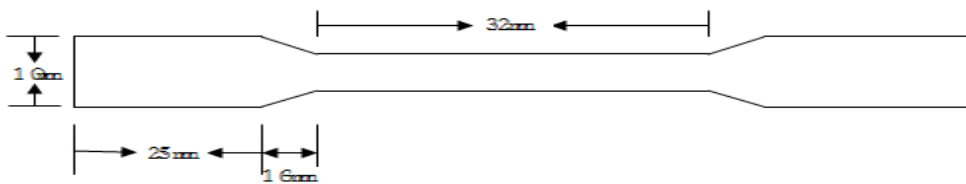


Figure 10 : Tensile test specimen dimension



Figure 11 : Photograph of a Tensile test specimen

The following steps were followed to perform tensile test -

- a) Dimensions of the specimen were taken
- b) The test machine was calibrated
- c) Cross head speed was taken 4 mm/min
- d) The ultimate tensile strength (UTS) was calculated from the following formula-
UTS = maximum load / initial area (MPa)

or

$$\sigma = P_{\max} / A \dots \dots \dots (1)$$

- e) The modulus was calculated by taking the slope of the stress-strain curve.

Three Point Flexural Test

The three-point bend test is generally performed on composites. It says that, “the depth of support span shall be 16 times the depth of beam. Specimen width shall not exceed one fourth of the support span for specimens greater than 3.2 mm in depth”.

Total length of the specimen is taken as {SP (length of support span= 16d) + (20-30 mm)}

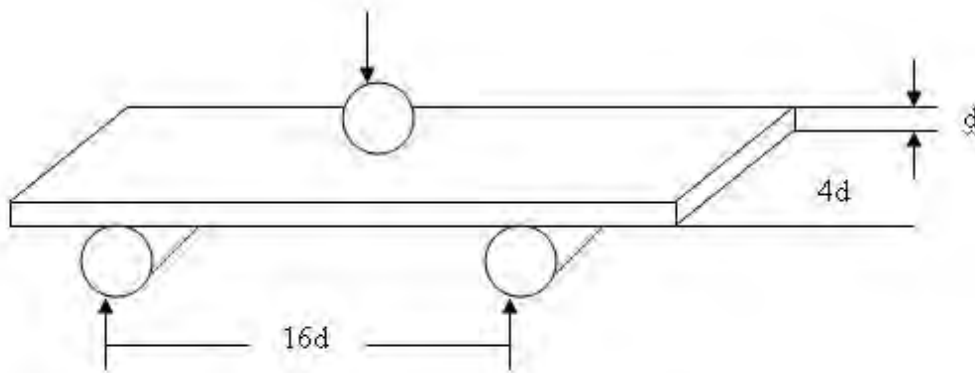


Figure 12 : Three point Flexure test specimen dimension.



Figure 13 : Photograph of a Flexure test specimen.

Flexure test steps and calculation

Test specimens of required shape were cut from the composite part. The specimen dimension was given according to the ASTM D 790-00 [75]. Then all the dimensions were measured. After that the rate of cross head motion was calculated using the following formula,

$$R = 0.05 * L^2 / 6d \dots\dots\dots (2)$$

Where, R = rate of cross head motion mm/min

d = depth of beam, mm

The loading nose and supports were aligned in such a way so that the axis of the cylindrical surfaces was parallel and the loading nose was midway between the supports. The load was applied to the specimen at the specified cross head motion and simultaneously load deflection data was taken.

The test was terminated when rupture occurred at the outer surface of the test specimen. The flexural stress was calculated by means of the following equation:

$$\sigma_f = \frac{3PL}{2bd^2} \quad \text{.....}$$

(3)

Where

σ_f = stress in the outer fibers at mid point, MPa

P = load, N

L = support span, mm

b = width of beam, mm

d = depth of beam, mm

Flexural Modulus,

$$E = \frac{PL^3}{4bd^3m} \quad \text{.....} \quad (4)$$

Where, m = the gradient (i.e., slope) of the initial straight-line portion of the load deflection curve

Flexural strain is calculated according to the equation

$$\epsilon = \frac{6Dd}{2PL*100} \quad \text{.....} \quad (5)$$

Where, D = elongation, mm

Hardness Test

Hardness may be defined as a material's resistance to permanent indentation. The hardness of the

composite was measured using a shore hardness testing machine. Shore Durometer is one of

several measures of the hardness of a material. Shore Durometer is typically used as a measure of hardness in polymers, elastomers and rubbers. There are several scales of Durometer, used for materials with different properties. The A scale is for softer plastics, while the D scale is for harder ones. Hardness property of composite was measured using shore A scale.

Field Emission Scanning Electron Microscopy (FESEM)

The scanning electron microscopy is a versatile, non-destructive technique that reveals detailed information about the morphology and the composition of natural and manufactured materials. Morphological analysis of the fiber is carried out using SEM. This is the most widely used of the surface analytical techniques. High resolution SEM has proved an invaluable tool for studying surface topography and failure analysis. The technique enables qualitative three dimensional (3-D) imaging of surface features, however, it does not easily lend itself to quantitative surface roughness characterization. This can be overcome by complementing SEM investigations with atomic force microscopy. In SEM, a highly focused scanning electron beam bombards the surface causing large numbers of secondary electrons to be generated, the intensity of which is governed by surface topography. The method is suitable for all materials, but non conducting materials must be given a thin conductive coating of sputtered gold, which can alter or mask the true surface morphology.

A Field Emission Microscope consists of a metallic sample in the form of a sharp tip and a conducting fluorescent screen enclosed in ultrahigh vacuum. The tip radius used is typically of the order of 100nm. It is composed of a metal of high melting point, such as tungsten. The sample is held at a large negative potential (1-10 kV) relative to the fluorescent screen. This gives the electric field near the tip apex to be the order of 10^{10} V/m which is high enough for field emission of electrons to take place.

The interfacial bonding between the fibers and PP matrix in manufactured composites and tensile fracture surfaces of the same composites were examined using a Scanning Electron Microscope JEOL FESEM analyzer of model no JSM-7600F. The micrographs are presented in the Results and Discussion section.

FT-IR Spectroscopy

FTIR is an effective analytical tool for identification of unknowns, sample screening and profiling samples. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, by the sample material versus wavelength. As chemical bonds absorb infrared energy at specific frequencies (or wavelengths), basic structure of compounds can be determined by the spectral locations of their IR absorptions.

In the present study infra-red spectra of areca waste nylon fiber reinforced hybrid PP composites were recorded on a SHIMADZU spectrophotometer with co-addition of 32 scans. Firstly some powdered samples were collected by scratching of the composite by a knife. Then potassium bromide (KBr) was mixed at a ratio of (KBr : Sample = 100:1) with them in a mortar pestle. The mixture was then taken in a dice of specific dimensions (13 mm dia). The pellet was formed by pressing (mechanical presser at 8 tons of pressure) and was placed on the sample holder and the spectroscopy was carried out.

TGA Analysis:

Thermo gravimetric analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature programmed in a control atmosphere. It provides important data towards understanding the structure-property relationships and controlling the technology for molecular design with the aim of industrial production of different polymeric materials, especially fiber reinforced composites. Moreover, it is a useful technique to determine the thermal stability of the materials. Additionally, it permits to quantify the amount of possibly damaging, deteriorating volatiles, such as the moisture uptake during a hydrothermal treatment, which can cause deterioration in the composites.

In our study TGA was carried out in an universal V4.2E TA instruments (TGA Q50 V6.4) at a temperature range of 25-800°C, with a constant heating rate of 10°C/sec.

CHAPTER 4

RESULT AND DISCUSSION

Result and discussion

4.1 FTIR Spectroscopic Analysis of Areca and Waste Nylon Fiber Reinforced Hybrid Polypropylene (PP) Composite

The FTIR spectrum of PP (polypropylene) is shown below in "Figure 14". In the spectrum the major peaks are found at 2922.6 cm^{-1} , 1458 cm^{-1} and 1377.3 cm^{-1} corresponds respectively to the sp^3 C-H stretch, CH_2 (methylene) bend and CH_3 (methyl) bend. Also other important peaks are at 1169.1 cm^{-1} corresponds to C-C stretching, CH_3 wagging and 998 cm^{-1} corresponds to C-C stretching, CH_2 rocking, CH_3 rocking.

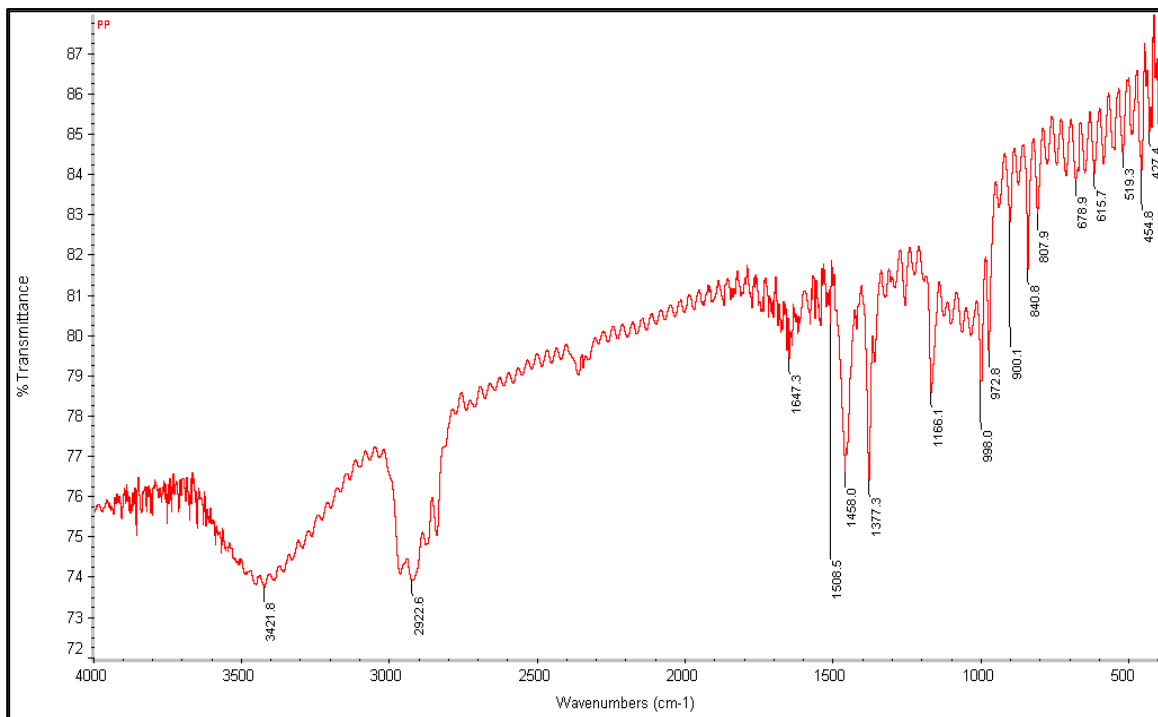


Figure 14 : FTIR Spectrum of PP (polypropylene) [76]

The FTIR spectrum of the untreated areca fibers and waste nylon fibers between around 450 cm^{-1} and 4000 cm^{-1} are shown in "Figure 15 and Figure 16". The characteristic infrared absorption frequencies that dominates in the FTIR spectrum of areca fiber are at 3421.72 cm^{-1} which indicates the hydroxyl group and bonded OH stretching, 1045.42 cm^{-1} which indicates C-O stretching vibrations. The peak at 1737.86 cm^{-1} represents ester and other crosslinks between cellulose and lignin or cellulose and hemicelluloses [37]. Furthermore, the peak of C=C

stretching of the aromatic ring of the guaiacyl unit is found at 1643.35 cm^{-1} [37]. The peaks observed between $1100\text{-}1600\text{ cm}^{-1}$ showed the presence of hemicellulose in the fiber. The peak at 1379.1 cm^{-1} is referred to alcohol group of cellulose. The peak at 1512.19 cm^{-1} corresponds to the lignin and lignocelluloses (aromatic skeletal). Absorption peak corresponding to C=O stretching around 2927.94 cm^{-1} , aromatic C–H in-plane deformation (typical of S units) plus secondary alcohols plus C=O stretching around $\sim 1253.73\text{ cm}^{-1}$ was also found in this study [77].

The characteristic infrared absorption frequencies of the waste nylon fiber includes peak at 1739.79 cm^{-1} which represents C=O stretching, 3425.58 cm^{-1} corresponds to amine N-H stretching. The peaks at $3500\text{-}3700\text{ cm}^{-1}$ represents amide N-H stretching. The peak around 725.23 cm^{-1} corresponds to N-H wagging and the peak around 1315.45 and 1456.26 cm^{-1} corresponds amide III band and N-H deformation [78].

All these characteristic peaks are quite conspicuous at the FTIR of untreated areca and waste nylon fiber reinforced PP composite "**Figure 17**" also the peak in the range of $\sim 2900\text{-}2880\text{ cm}^{-1}$ is due to C-H vibration of –(CH₂)– group of PP [79].

FTIR spectra analyses of the treated areca and waste nylon fiber reinforced PP composite is shown in "**Figure 18**". Although the occurrence of majority peaks has not changed, it is noted that the absorption peak around 1737 cm^{-1} , corresponding to the C=O stretching of hemicellulose in the untreated fiber, has lost its intensity in the spectrum of the alkali treated fiber, indicating the elimination of hemicellulose phases by alkali treatment. Furthermore, C=C stretching of the aromatic ring of the guaiacyl unit at around 1640 cm^{-1} and lignin and lignocelluloses peak at 1514.12 cm^{-1} also showed lower intensity in the treated fiber reinforced composite sample. In both the samples the band at around 2930 cm^{-1} is due to the C-H stretching of aromatic methoxyl groups and methyl and methylene groups of the side chains [37].

A strong sodium hydroxide (NaOH) treatment might remove lignin, hemicelluloses and other alkali soluble compounds from the surface of the fibers to increase the numbers of reactive hydroxyl groups on the fiber surface available for chemical bonding. Thus the alkali treatment could cause an increase of the fiber surface free energy. Moreover, the alkali treatment could make the fiber surface 'clean' due to removal of waxes, hemicelluloses, pectin and part of lignin. The removal of these substances enhanced the surface roughness. Therefore, the mechanical

interlocking at the interface could be improved. In general ,alkali treatment increases the strength of natural fiber composite [37].

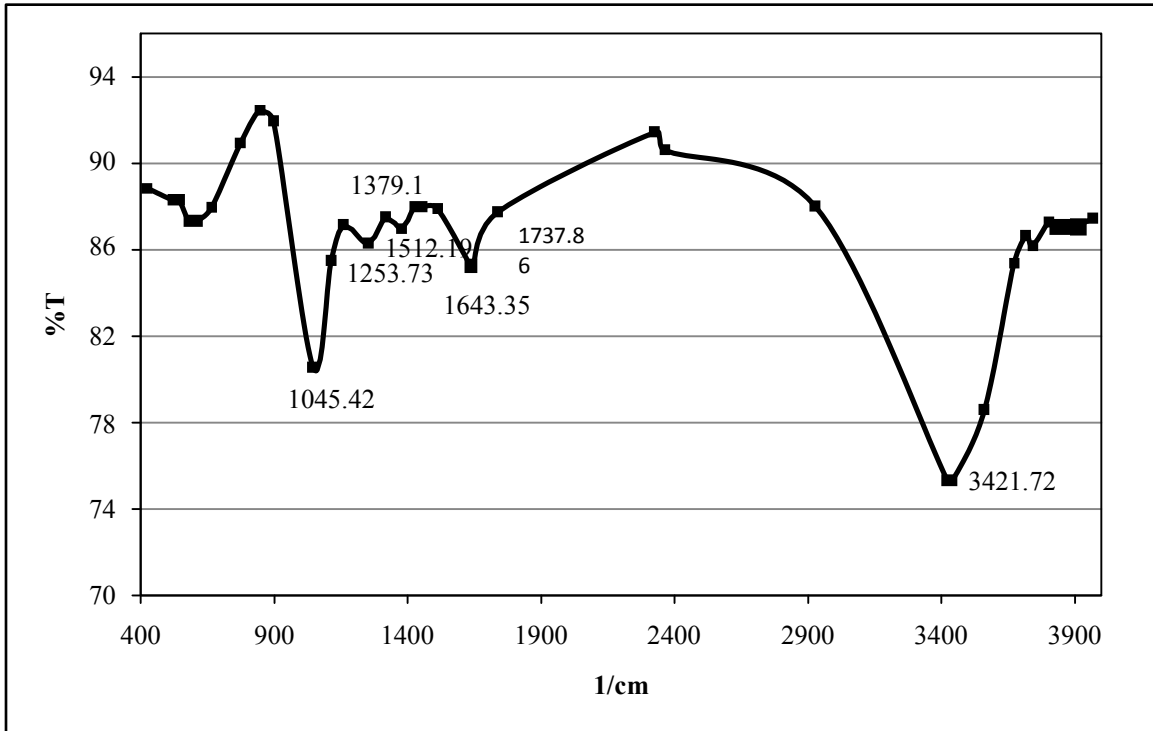


Figure 15 : FTIR Spectrum of areca nut Fiber

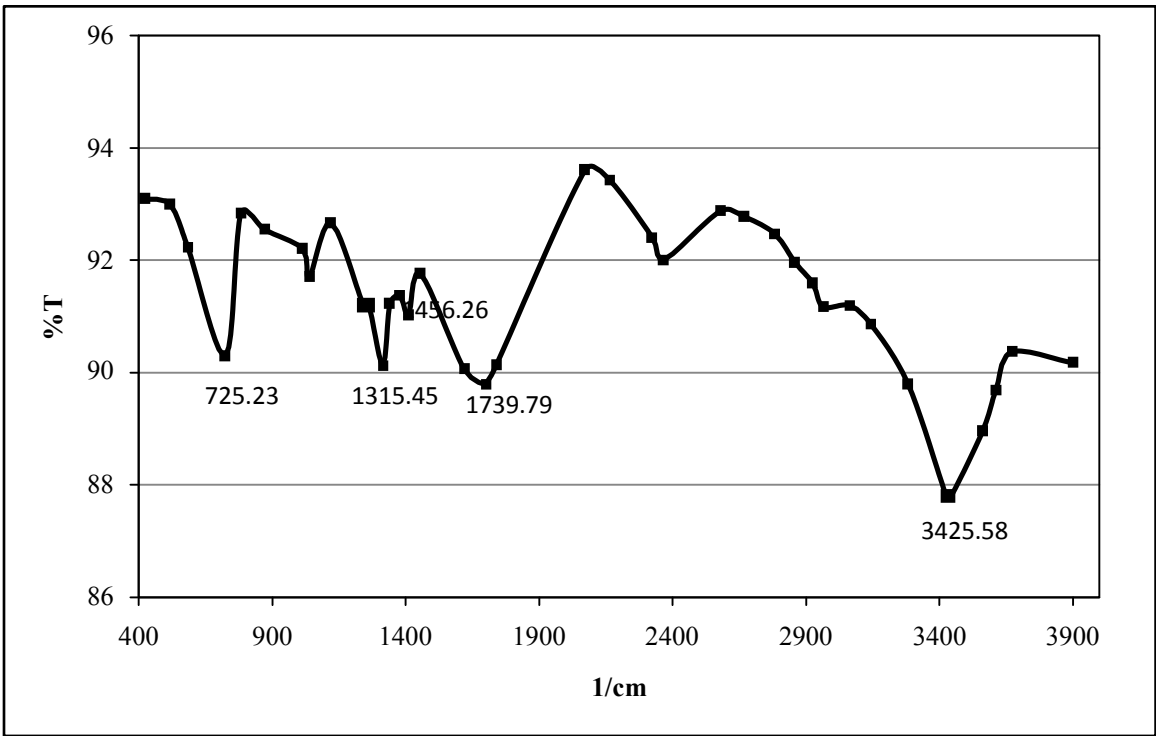


Figure 16: FTIR Spectrum of nylon fiber

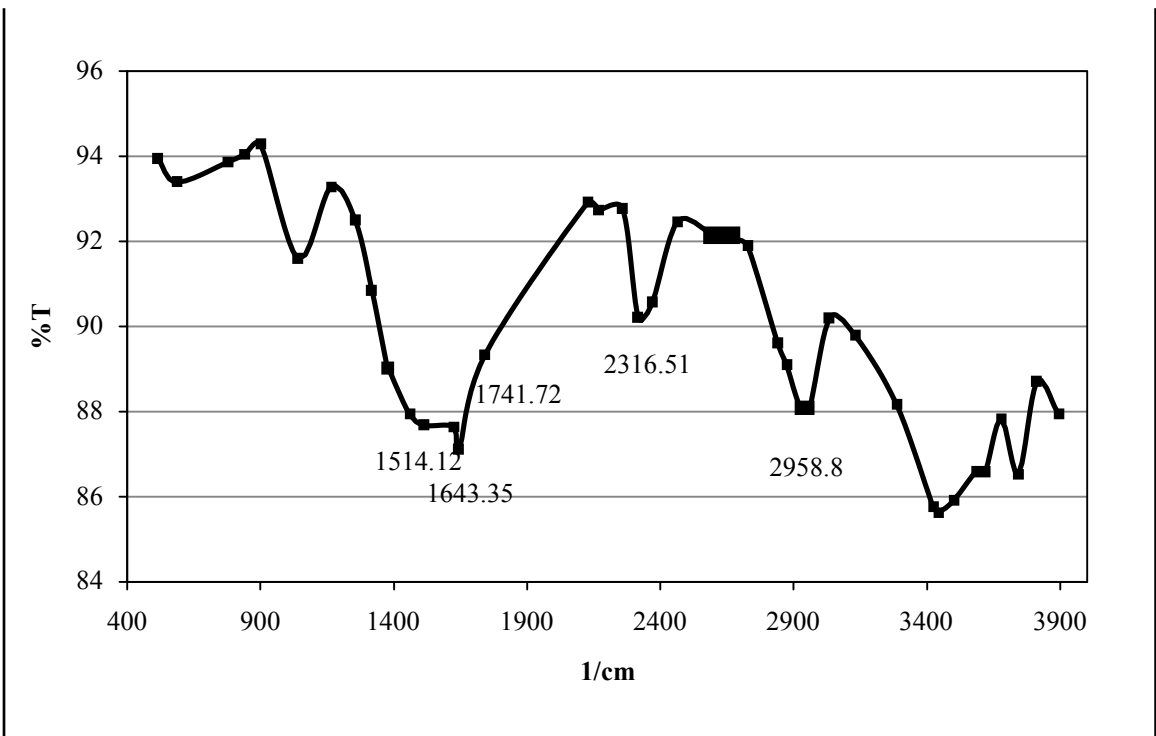


Figure 17 : FTIR Spectrum of 3L10 wt % areca and waste nylon fiber reinforced PP composite

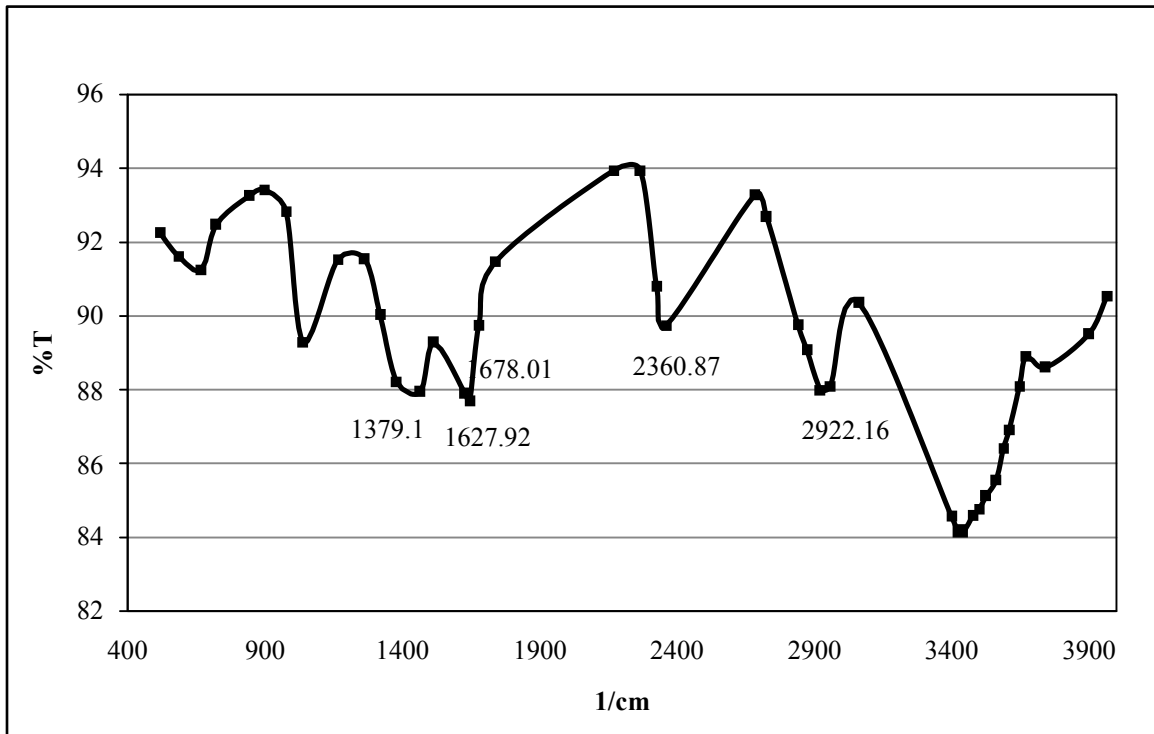


Figure 18: FTIR Spectrum of 3L 10 wt% treated areca and waste nylon fiber reinforced PP composite

4.2 Mechanical Properties

4.2.1 Tensile Test Results

Tensile properties (Tensile strength, Young's modulus and % elongation at break) of the 2 layered (2L), 3 layered (3L) and 4 layered (4L) 10 wt% areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composite samples were measured. The tensile strength and tensile modulus of areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composites are shown in “**Figure 19 and Figure 20**”.

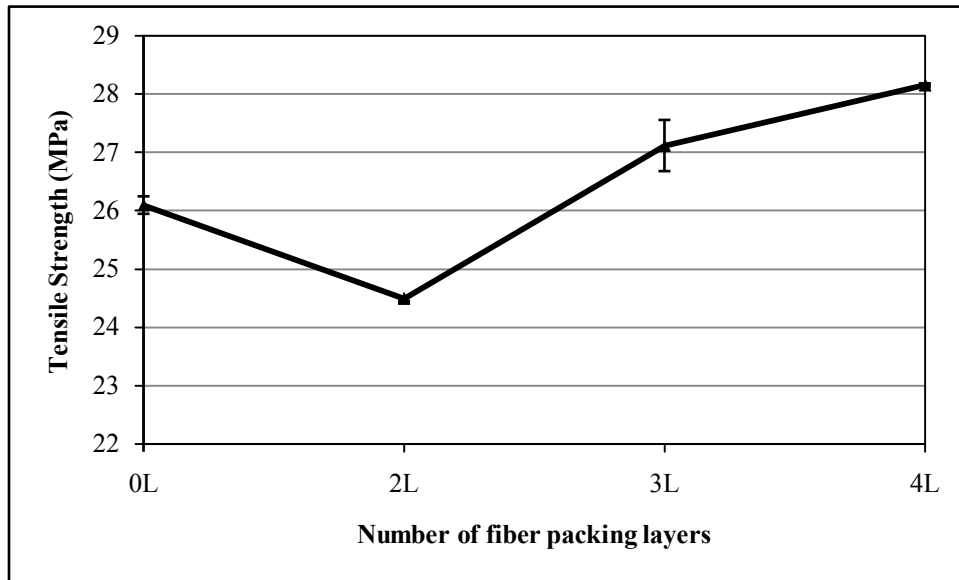


Figure 19: Variation of Tensile Strength with number of fiber packing layers

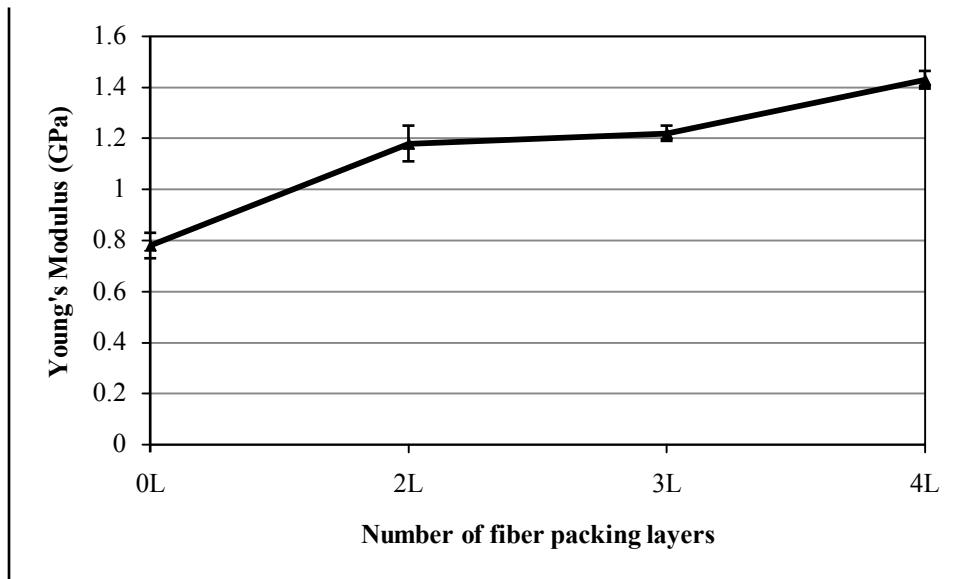


Figure 20 : Variation of Young's Modulus with number of fiber packing layers

According to the experimental results the tensile strength increased with the increase in number of fiber layers. As the number of fiber layers increased, the fibers became uniformly dispersed into the matrix. Thus the interaction between the fibers were limited which constrained the formation of voids [80-81]. This consequently increased the tensile strength. The same trend was also observed by other researchers [81-82]. It was also observed that the Young's modulus

increased with an increase in number of fiber layers. High stiffness was related to the fine dispersion of individual fiber which creates good materials contact [80-84].

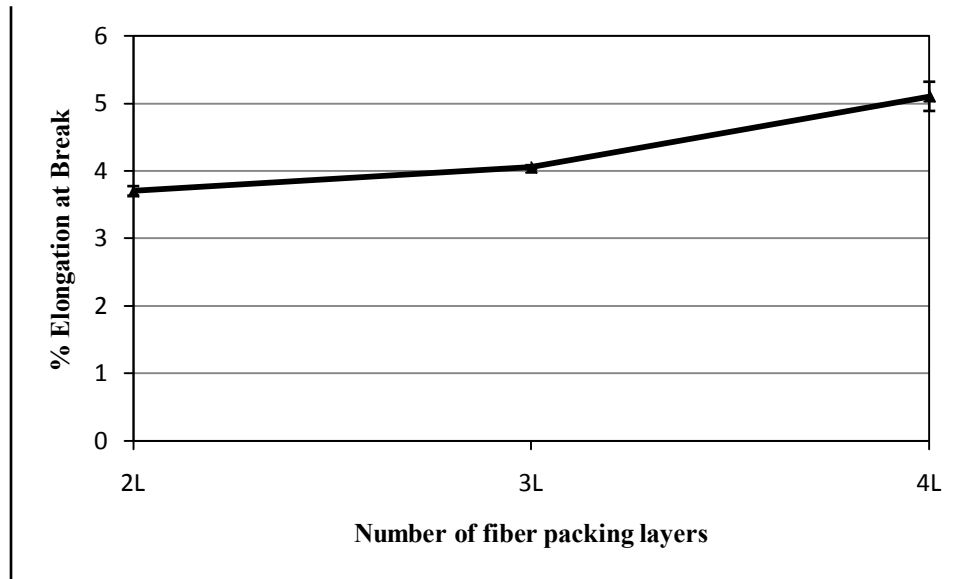


Figure 21 : Variation of % Elongation at Break with number of fiber packing layers.

Percentage elongation at break increased with the enhancement of number of fiber layers. This was due to the uniform dispersion of fibers that restricted the interference of fibers in the mobility or deformability of the matrix, which resulted in the enhancement of elongation at break of the composite [82-83].

Among the 2L, 3L and 4L composites 4L composite showed better mechanical properties. Thus for further studies tensile properties (Young's modulus, tensile strength and elongation at break) of the 4L (10, 15, 20, 25) wt.% NaOH treated areca and waste nylon fiber (areca: nylon=1:1) reinforced composite samples were measured with the help of stress/strain curves.

The tensile strength and Young's modulus of 10, 15, 20, 25 wt.% treated areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composites are shown in “**Figure 22 and Figure 23**”.

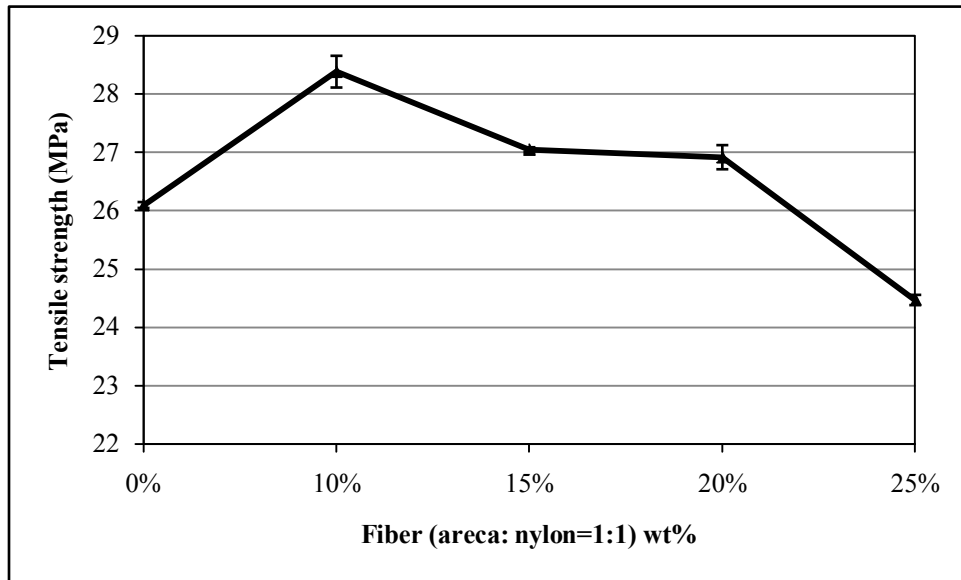


Figure 22 : Variation of Tensile Strength with fiber wt%

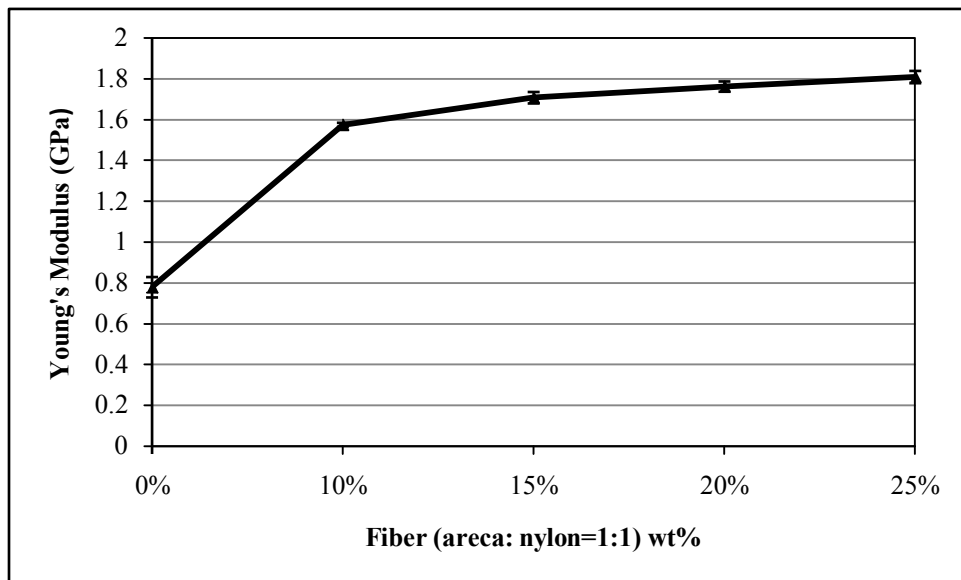


Figure 23 : Variation of Young's Modulus with fiber wt%

From the given tables and curves it's quite conspicuous that the treated areca fiber reinforced composites showed better mechanical properties than untreated fiber reinforced one. Untreated fiber reinforced composites provided poor mechanical properties because of the weak interfacial bonding between cellulose based hydrophilic filler (areca) and hydrophobic matrix.

Mercerization (alkali treatment) removed the impurities, waxy materials, lignin as well as hemicellulose and resulted in an increment of cellulose exposed on the fiber surface and thereby increased the number of possible reaction sites. It also enhanced the interfacial bonding by mechanical interlocking due to increase in surface roughness. In addition mercerization improved the fiber wetting by fibrillation, which increased the effective surface area available for the contact with the wet polymer matrix [50].

During alkaline treatment, the inter-fibrillar region was likely to be less dense and less rigid, that made the fibrils rearranged themselves more along the direction of tensile loading. When fibers were stretched, such arrangements among the fibrils would result in better load sharing and hence in higher stress development in the fiber [50,85].

Also alkali treatment caused loss of crystalline structure of cellulose molecular chains in a microfibril partly, as a result of which the alignment of the microfibrils was destroyed and the overall crystallinity was reduced. In addition, removal of certain elements caused easy deformation of the cellular networks. The extensive hydrogen bonding network might be broken and the ordered structural arrangement of cellulose might also lose [51].

Though an increase in the tensile strength of the hybrid composites were observed with the incorporation of treated areca fiber in place of untreated one but with the increase of total fiber loading in composites tensile strength decreased [43,87]. An increase in the filler content increased the micro spaces between the filler and the matrix, which weaken the filler–matrix interfacial adhesion. As a result, the values of tensile strength showed a decreasing trend with increasing filler content in the composite. Also increase in fiber content up to a certain level may lead to an increase in fiber–fiber interactions and may also cause difficulties in dispersion resulting in fiber agglomeration [49]. The over presence of fiber in the material contributes in initiating the crack which can cause non-uniform stress transfer.

It was also observed that Young's modulus increased with an increase in fiber loading [48,53]. This was because with an increase in fiber content, the brittleness of the composite increased and stress/strain curves became steeper. Also incorporation of fibers in matrix creates partially separated micro spaces which obstruct stress propagation between the fiber and the matrix [14]. As the fiber loading increased, the degree of obstruction increased, which in turn increased the

stiffness. Furthermore incorporation of fiber into the polymer matrix reduced the matrix mobility which in turn enhances the stiffness property [48].

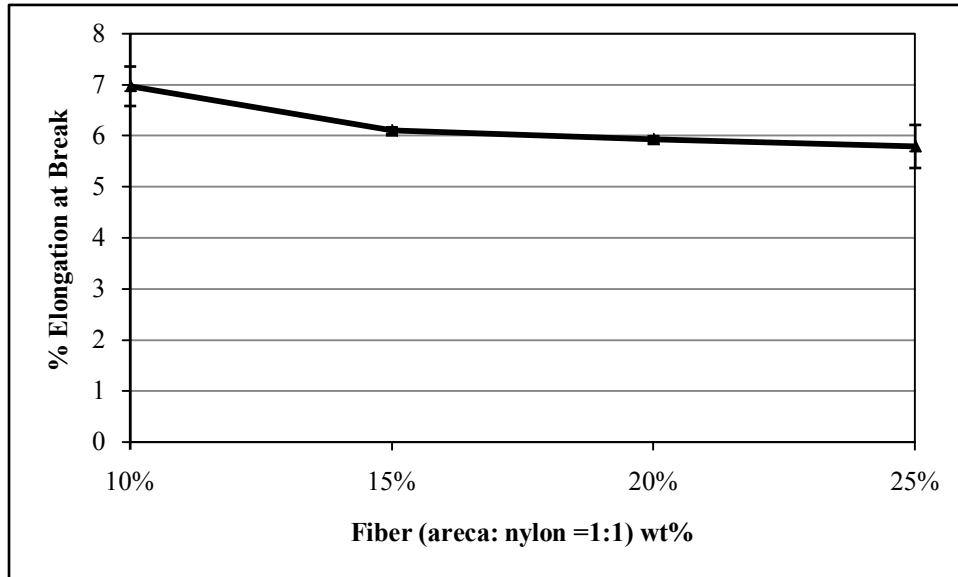


Figure 24 : Variation of % Elongation at Break with fiber wt%

The tensile strength and Young's modulus of different ratio of treated areca and waste nylon 20 wt.% fiber (areca: nylon=3:1, 3:2, 2:3, 1:3) reinforced hybrid polypropylene composites are shown in “**Figure 25 and Figure 26**”.

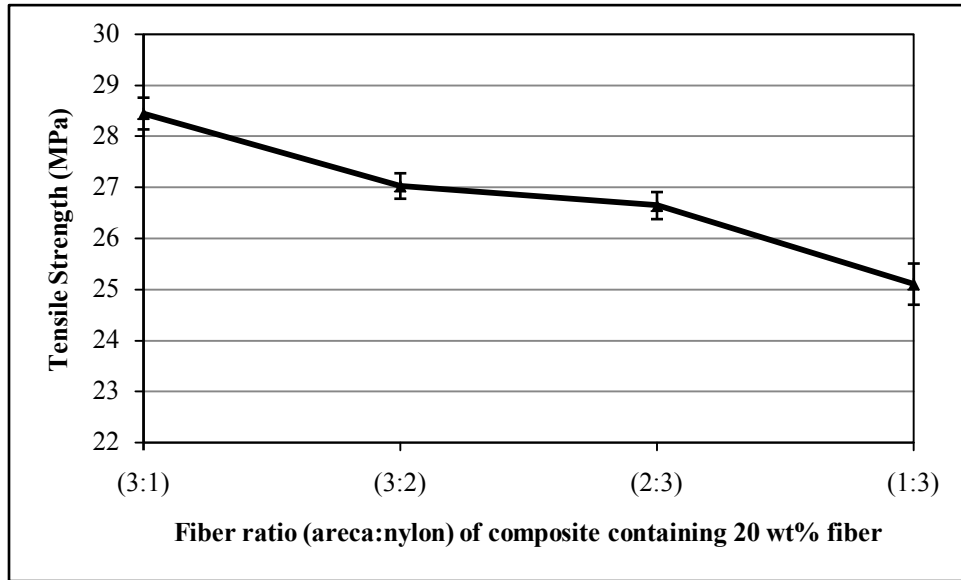


Figure 25 : Variation of Tensile Strength with different fiber ratio

It was found that maximum amount of areca fiber reinforced composite showed maximum tensile strength. With the enhancement of the amount of nylon fiber tensile strength decreased. The properties of hybrid composites were mainly dependent on the properties of individual reinforcing fibers, orientation, and arrangement of fibers, the extent of intermingling of the fibers and also the fiber-matrix adhesion. In this case, the increase in the strength of the hybrid composites with the incorporation of areca fiber was mainly due to the high tensile strength of areca fiber (166.03 MPa) compared to the low strength nature of nylon fiber (82.7 MPa) [54].

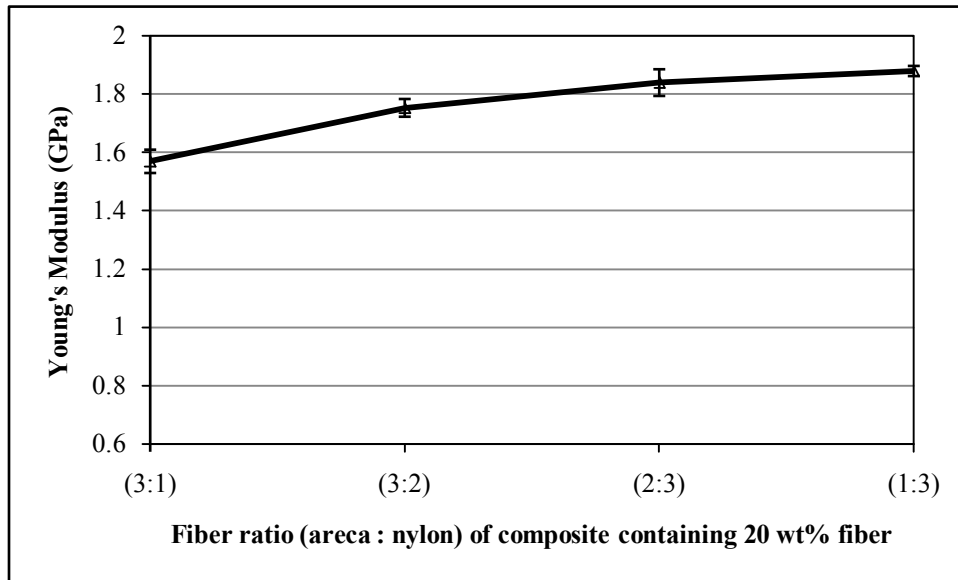


Figure 26: Variation of Young's Modulus with different fiber ratio

The incorporation of nylon fiber into areca and waste nylon fiber reinforced PP composite increased the modulus values of the hybrid composites as seen in "Figure 26". The enhancement in the stiffness of these composites is attributed to the higher modulus of nylon fiber (2.93-3.3GPa) than that of the areca fiber (1.38-1.93 GPa) [54,77]. Among the hybrid composites, a maximum loading of nylon fiber in composites having 75wt% fiber content gave the highest value of tensile modulus. It was observed that with an increase in the weight fraction of nylon fiber from 25 to 75%, the tensile modulus of hybrid composites increased steadily from 1.59 to 1.88 GPa. As the weight fraction of nylon fiber increased, the load bearing capability of the hybrid composites also increased and resulted in improved stiffness.

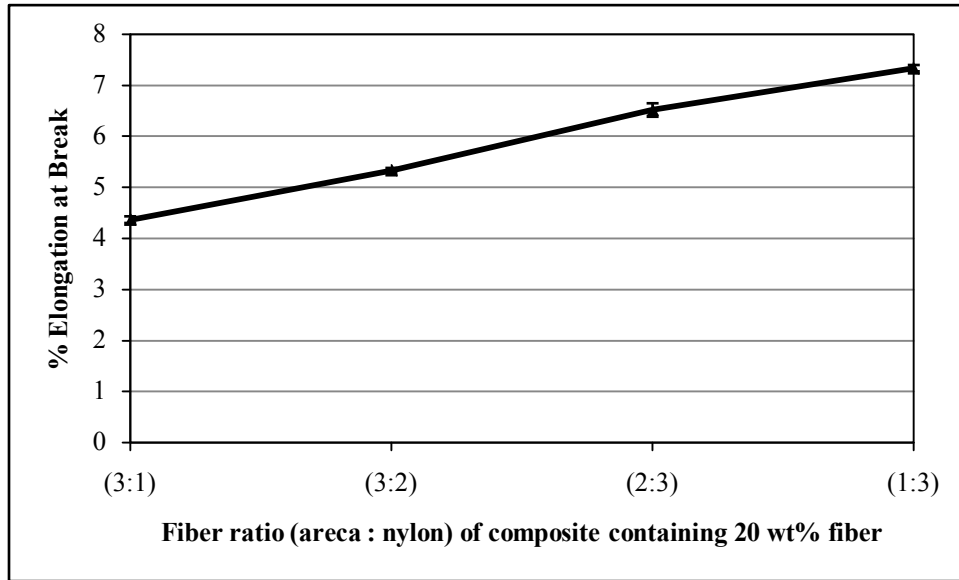


Figure 27 : Variation of % Elongation at break with different fiber ratio

In hybrid composites, the properties of the composites are mainly dependent on the modulus and percentage of elongation at break of the individual fibers. "Figure 27" shows the variation of elongation at break with both areca and nylon fiber loading. The value of elongation at break showed an improvement with an increase of nylon fiber content in hybrid composites. Among the hybrid composites, the incorporation of 75wt% nylon fiber exhibited the highest elongation at break of composites, This phenomenon was due to the fact that nylon fiber individually has high % elongation at break in comparison to areca fiber [77].

4.1.2 Flexure Test Results:

Flexural properties (flexural strength and flexural modulus) of the 2 layered (2L), 3 layered (3L) and 4 layered (4L) 10 wt.% areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composite samples were measured with the help of flexural load/deflection curves. The flexural strength and flexural modulus of areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composites are shown in "Figure 28 and Figure 29".

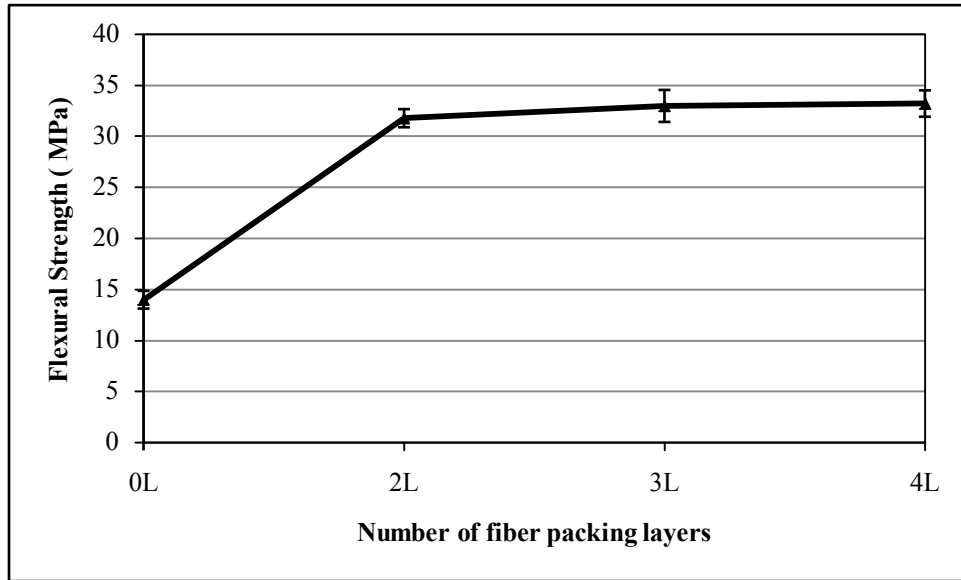


Figure 28 : Variation of Flexural Strength with number of fiber packing layers

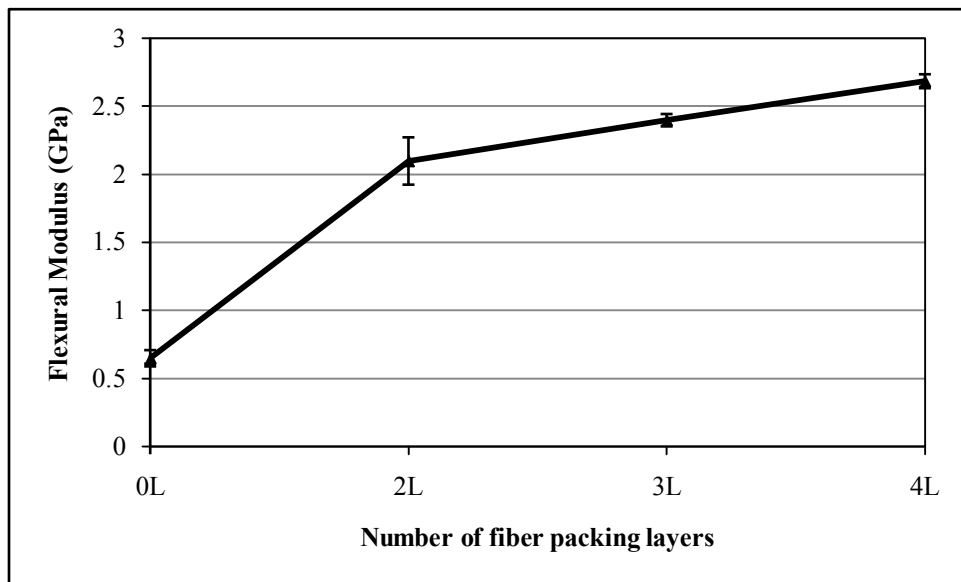


Figure 29 : Variation of Flexural Modulus with number of fiber packing layers

From the experimental observation it was found that the flexural strength increased with an increase in number of fiber layers. This might be due to the lower amount of clustering of fibers which resulted due to the non-uniform dispersion of fibers with decreasing number of fiber layers. Clustering affects the effective load transfer and reduces the flexural strength [81].

Enhancement of number of fiber layers provides uniform dispersion of fibers as well as effective load distribution. Also properly arranged high modulus fiber causes the enhancement of flexural modulus.

The flexural strength and flexural modulus of 10,15,20,25wt% treated areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composites are shown in “**Figure 30 and Figure 31**”.

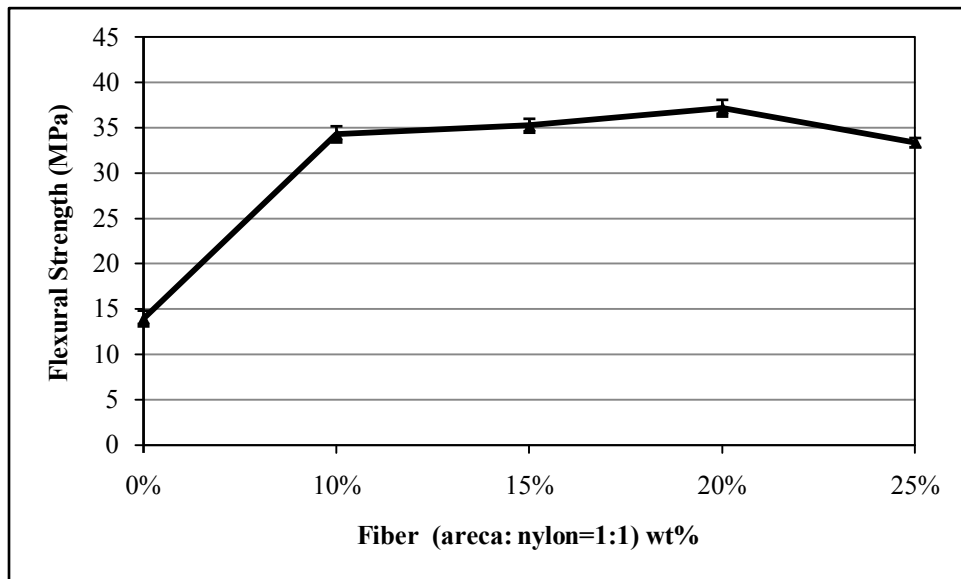


Figure 30 : Variation of Flexural Strength with fiber wt%

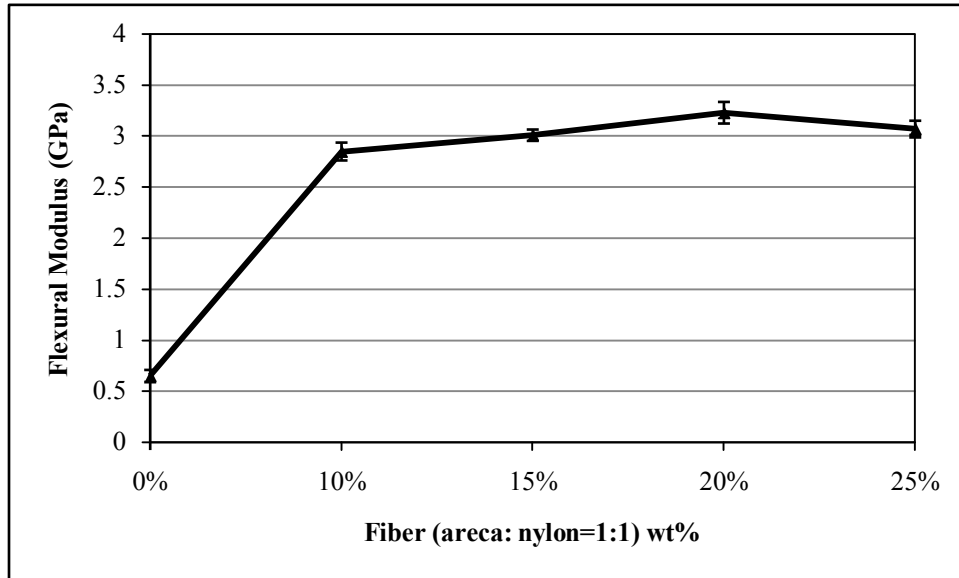


Figure 31: Variation of Flexural Modulus with fiber wt%

According to the values provided in the curves flexural strength and flexural modulus value of treated areca reinforced composite was better than that untreated areca reinforced composite. As a consequence of alkali treatment the interfacial interaction between the fiber and matrix were enhanced, which increased the effective available surface area of contact with the matrix, and the possibility of load transfer between the matrix and the reinforcing fibers which resulted into the increase of flexural strength and as well as modulus [51].

It was also found flexural strength and flexural modulus increased with an increase in fiber loading which was in agreement with the findings by other researcher [48,53] up to a certain fiber loading. This might be due to the favorable entanglement of the polymer chain with the filler which effectively enhanced the filler matrix adhesion with increasing filler content [53]. Also the flexural modulus increased with filler loading. Since both areca and nylon is high modulus material, higher fiber concentration demands higher stress for the same deformation and increased fiber-filler adhesion provides for increased stress transfer from the matrix to the filler. Thus this result can be attributed to the incorporation of rigid areca into the soft PP matrix.

However, the flexural properties of hybrid composites decline after 20wt% fiber content. The decrease in the flexural properties at higher fiber loadings (>20 wt%) was due to the increase in

fiber-to-fiber interactions and dispersion problem which resulted in low mechanical properties of composites.

The flexural strength and flexural modulus of different ratio of treated areca and waste nylon 20 wt.% fiber (areca: nylon=3:1,3:2,2:3,1:3) reinforced hybrid polypropylene composites are shown in “Figure 32 and Figure 33”.

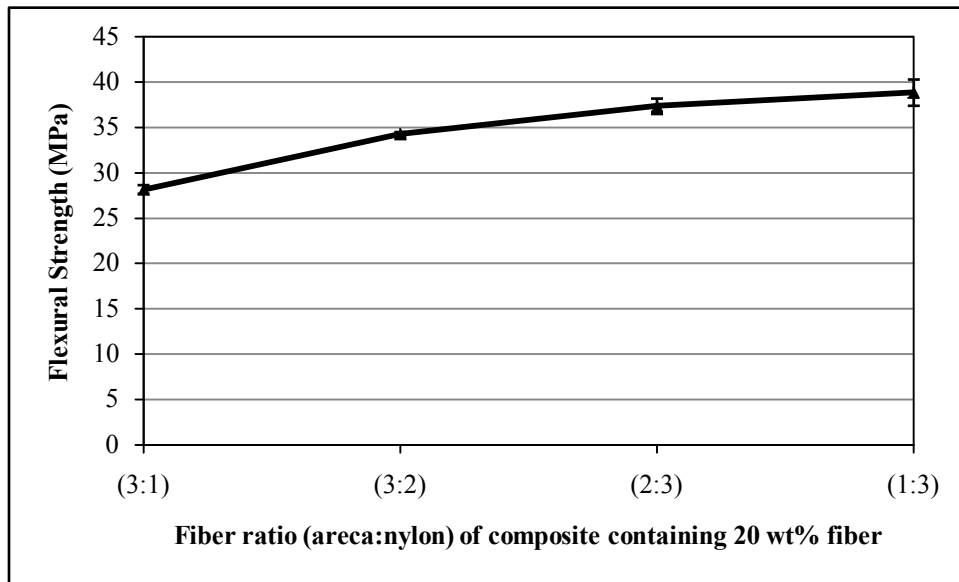


Figure 32 : Variation of Flexural Strength with different fiber ratio

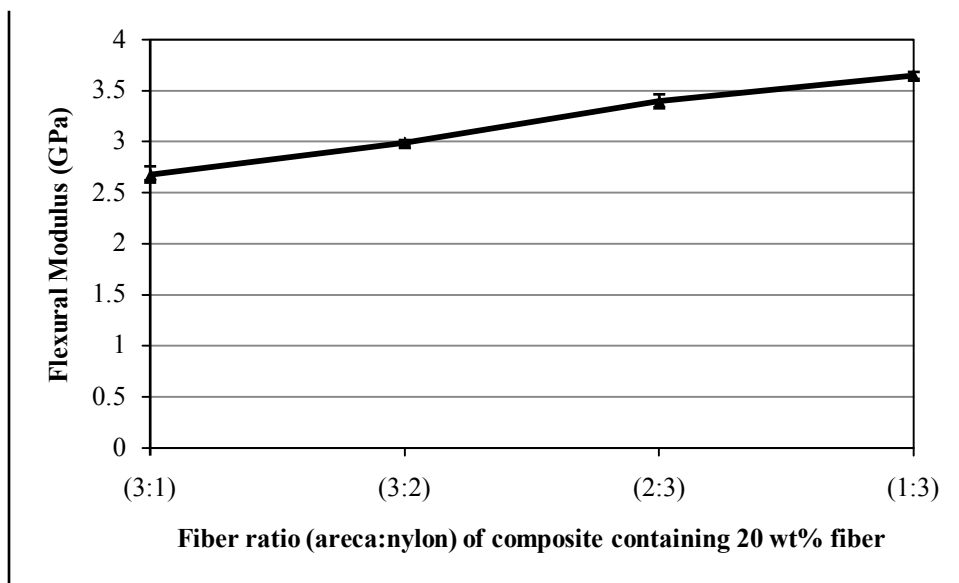


Figure 33 : Variation of Flexural Modulus with different fiber ratio

It was observed that the flexural strength and modulus of the hybrid composites had similar trends as the tensile properties where the flexural behavior increased with the addition of nylon fiber. In flexural loading, the composites samples were subjected to compression, tension and shear stresses. In a three-point flexure test, failure occurred due to bending and shearing. The increased flexural strength of the hybrid composites with the loading of nylon fiber was mainly due to the increased resistance to shearing of the composites as a result of the inclusion of rigid nylon fiber [87].

With the enhancement of nylon fiber percentage from 25 to 75% the flexural modulus of areca and waste nylon fiber reinforced PP hybrid composites increased by about 36%. This suggests that the flexural modulus of the composites depends largely on the amount of nylon fiber which could be due to the high modulus of nylon fiber. The highest flexural modulus was observed in hybrid composites with 75% weight fraction of nylon fiber. These were due to the presence of sufficient amount of fiber that provided the effective stress transfer between the fiber and the matrix and also due to the inherent property of nylon fiber [54].

4.2.4 Hardness Test Results:

Hardness property (Shore hardness A) of the 2 layered (2L), 3 layered (3L) and 4 layered (4L) 10 wt% areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composite samples were measured using shore hardness device. The shore hardness A of areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composites is shown in “**Figure 34**”.

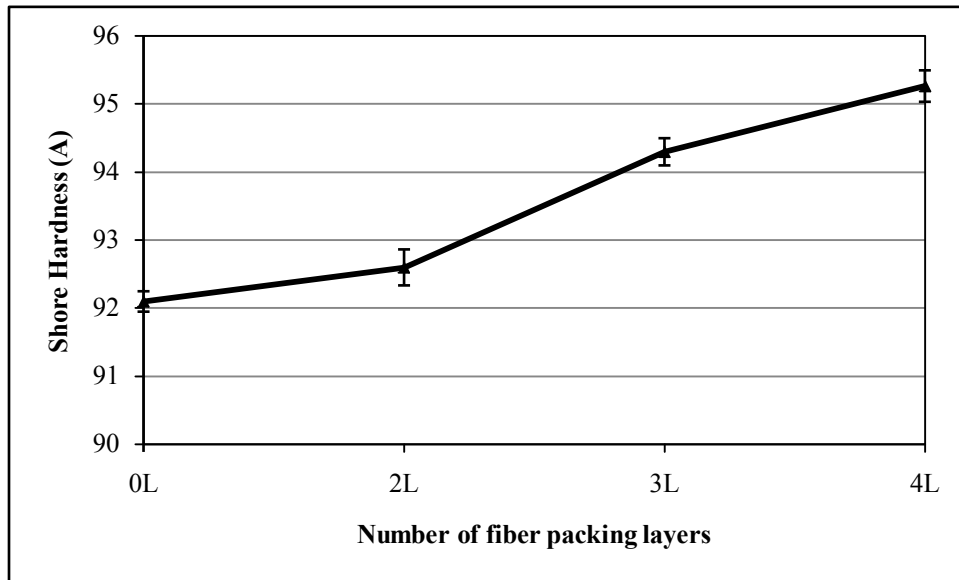


Figure 34 : Variation of Shore Hardness (A) with number of fiber packing layers

Hardness of a composite depends on the uniform distribution of the filler into the matrix [41]. Hardness is enhanced when the flexibility of matrix is lowered. The presence of a rigid fiber material into the pp matrix results into more rigid composites. Better dispersion of the filler into the matrix with minimization of voids between the matrix and the filler enhanced hardness. With the enhancement of number of fiber layer better dispersion of filler materials has obtained so hardness of areca waste nylon fiber reinforced hybrid PP composites has showed a slight increasing trend with an increase in the number of fiber layers [60].

The hardness values of 10,15,20,25 wt% treated areca and waste nylon fiber (areca: nylon=1:1) reinforced hybrid polypropylene composites is shown in “**Figure 35**”.

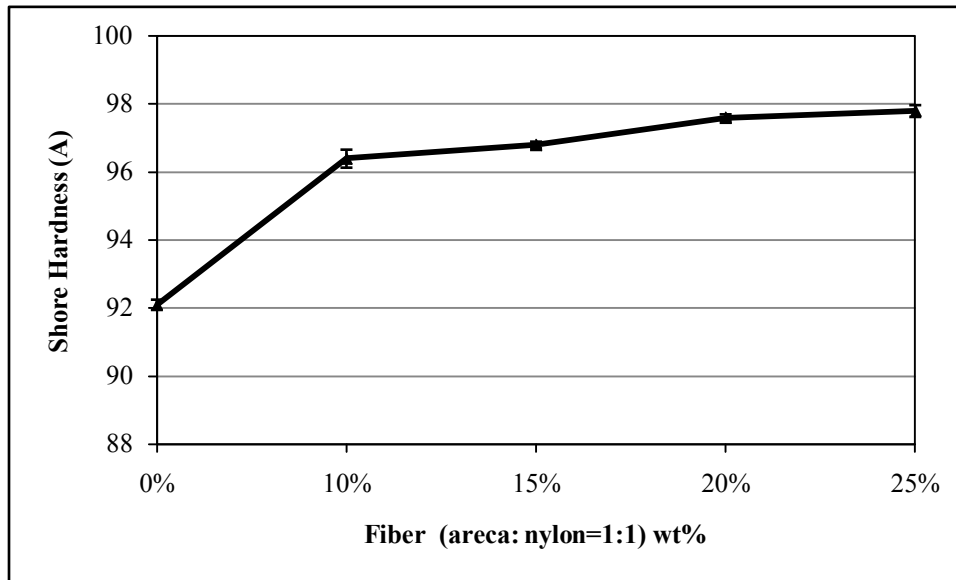


Figure 35 : Variation of Shore Hardness (A) with Fiber Wt%

Comparing results of raw and treated areca fiber reinforced composite it's quite evident that hardness value increased with the incorporation of alkali treated fiber though the wt. % of fiber is kept constant. Actually the alkaline treatment used on the fiber results collapse of the cellular structure due to the removal of cementing material. which leads to a better packing of cellulose chains and results into the reduction of void [51] and as well as better adhesion between the matrix and the filler. In this case, the polymeric matrix phase and the solid filler phase would be pressed together and touch each other more tightly. Thus the interface can transfer pressure more effectively. This might have resulted in an enhancement of hardness.

As shown in “**Figure 35**” with the enhancement of fiber wt% hardness value shows slight increasing trend. Actually incorporation of fiber into the PP matrix has reduced the flexibility of the matrix resulting in more rigid composites. Due to the increase of stiffness of respective composite the hardness of areca -waste nylon fiber reinforced hybrid PP composites showed a slight increasing trend with an increase in the fiber content [41].

The hardness value of different ratio of treated areca and waste nylon 20 wt% fiber (areca: nylon=3:1, 3:2, 2:3, 1:3) reinforced hybrid polypropylene composites is shown in “**Figure 36**”.

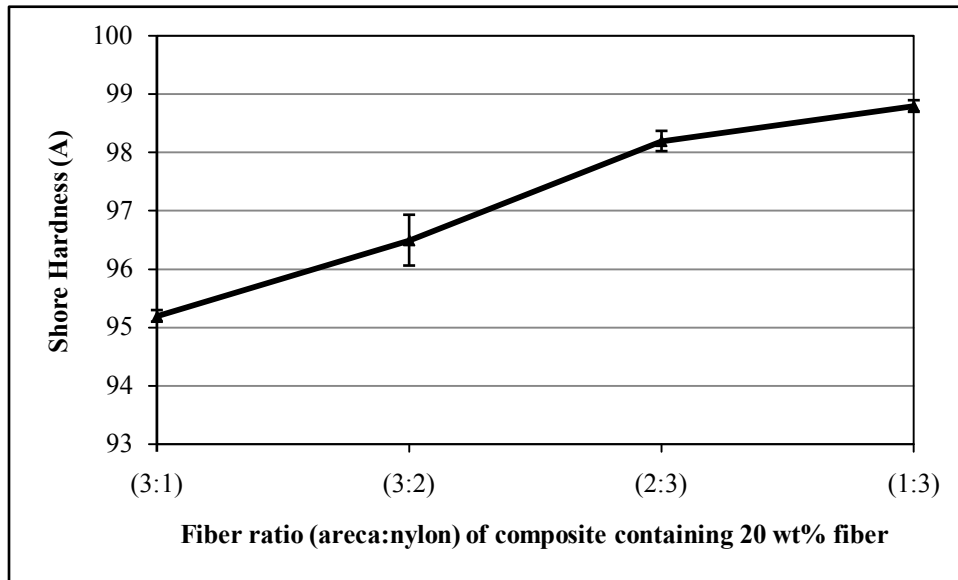


Figure 36 : Variation of Shore Hardness (A) with different fiber ratio

Though the enhancement of nylon fiber percentage has shown the enhancement of hardness values, the change wasn't quite conspicuous. Enhancement of nylon fiber percentage increased the hardness value by only 1.3%.

Surface morphology:

The SEM micrograph of untreated and sodium hydroxide (NaOH) treated betel nut fibers were shown in "Figure 37 and Figure 38". From these micrographs it was quite obvious that the alkaline treatment of fiber had effectively cleaned the fiber surface and had improved the fiber surface roughness. Also the removal of impurity resulted in separation of fiber strands.

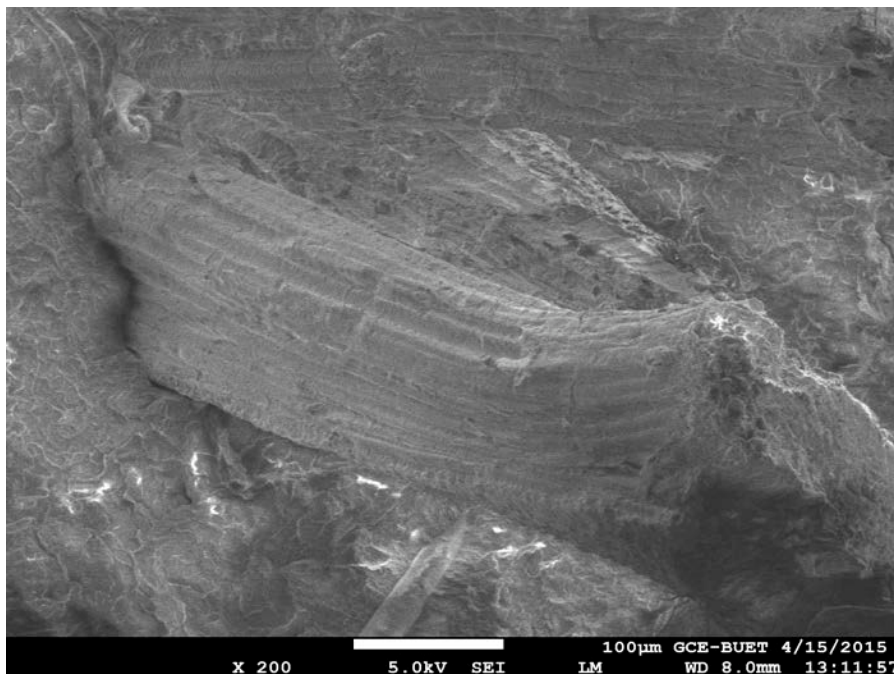


Figure 37 : SEM micrograph of untreated areca fiber

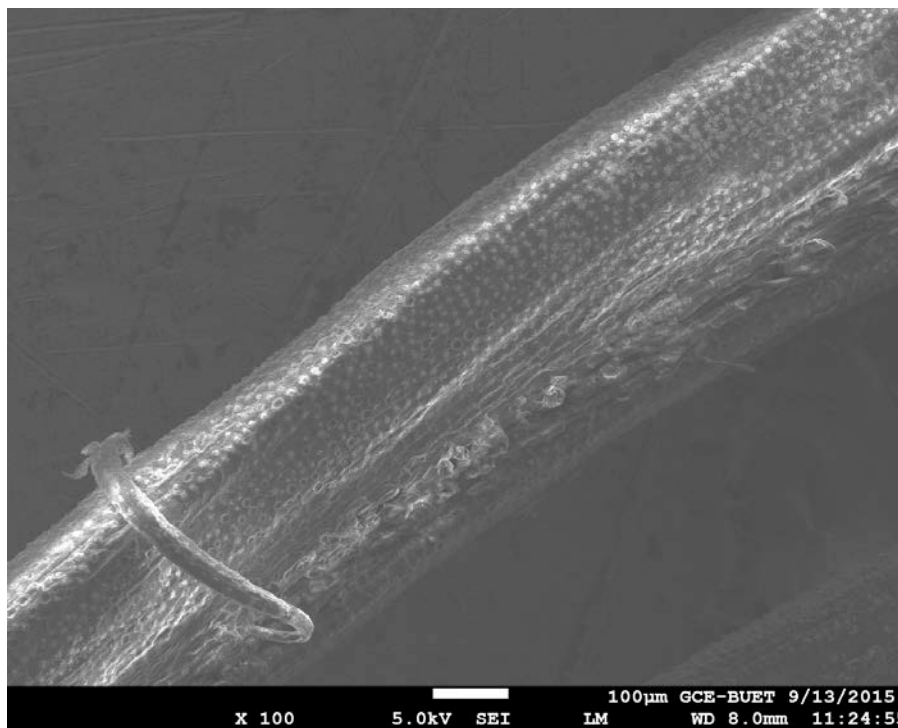


Figure 38 : SEM micrograph of treated areca fiber

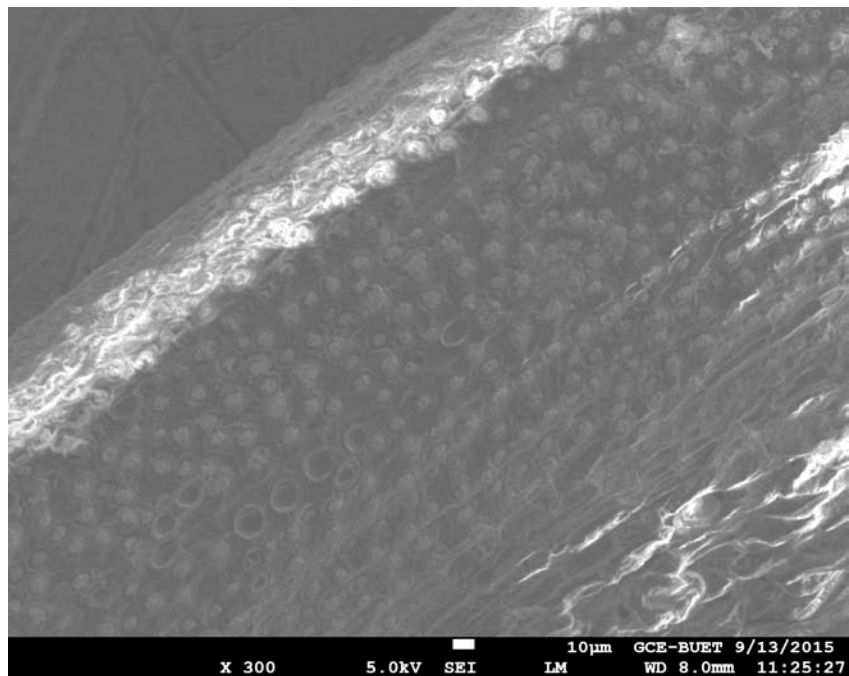


Figure 39 : SEM micrograph of treated areca fiber showing pores on to the surface of the fiber

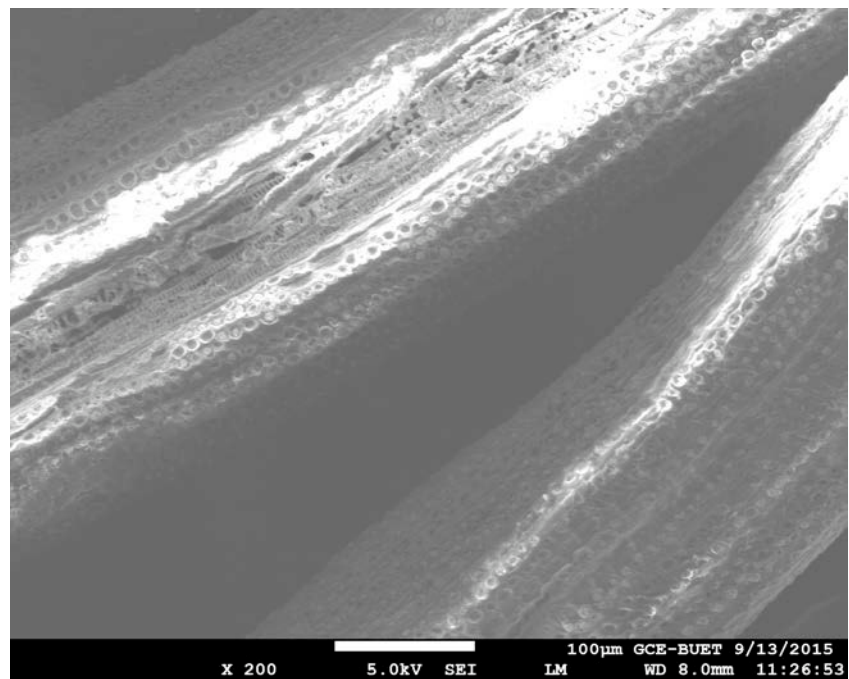


Figure 40 : SEM micrograph of treated areca fiber showing fibrillation

Alkali treatment causes removal of waxy layers from the surface of areca fiber which results in pit like pores on to the fiber surface [43]. It can be seen from the figure that there are deep pores and also the presence of rough surface is quite evident. As a result of which more possible anchoring points for mechanical interlocking is provided. The presence of this waxy layer prohibits the effective interfacial bonding between the natural fiber and polymer matrix. So removal of this low molecular weight components provide better wetting of the fiber surface with the matrix resin. From the second figure formation of elongated structure and fibrillated microfibrills are quite evident which provides more exposed area and better reaction sites [43].

The tensile fracture surface morphologies of 2L,3L,4L areca and waste nylon (areca: nylon=1:1) fiber reinforced hybrid polypropylene composites are shown in" **Figure 41,Figure 42 and Figure 43**" respectively. From The morphology of the fracture surface the phase information can be understood. It reflects the reasons why the mechanical properties of the composites fabricated with more number of fiber layers is better than others. The SEM micrograph of the 2L areca and waste nylon fiber reinforced composites shows weak interfacial bonding of fiber and matrix as well as agglomeration of the fibers in polypropylene matrix. In the SEM images of 3L fiber reinforced PP composites a number of pullout traces of fiber are present on the other hand 4L fiber reinforced PP composites show better dispersion of the filler into the matrix as clearly seen in the micrograph. Clustering of fiber suggest non uniform dispersion of fibers in matrix [89]. Fiber pull-out traces and the agglomeration of the fibers in the matrix is substantially reduced in 4L fiber reinforced one. The outcome of the better dispersion of fiber in the matrix is reflected by the improvement of the mechanical properties of the 4L fiber reinforced composites.

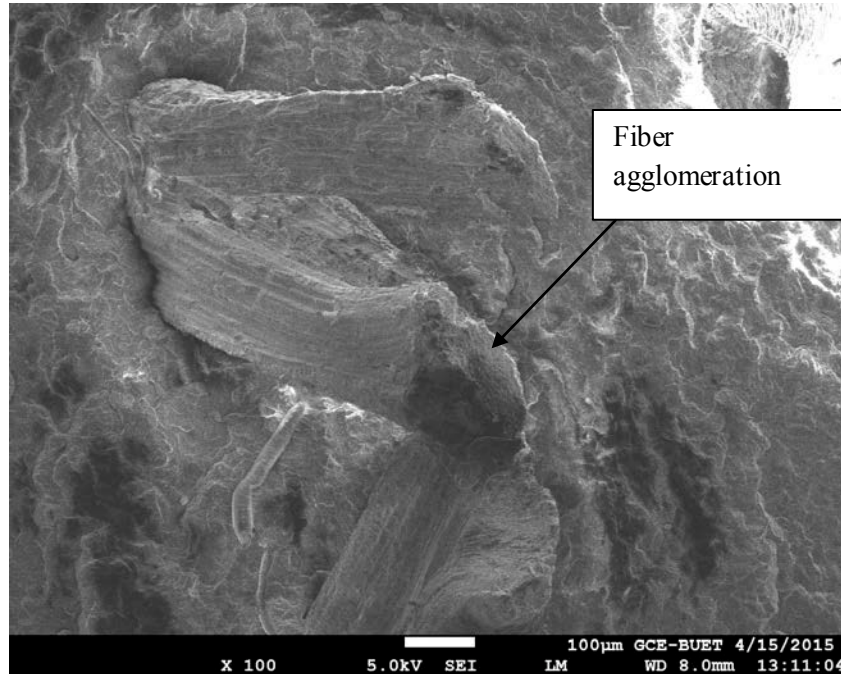


Figure 41 : SEM micrograph of Tensile fracture surface of 2L 10 wt% areca waste nylon fiber(areca: nylon=1:1)fiber reinforced PP composite.

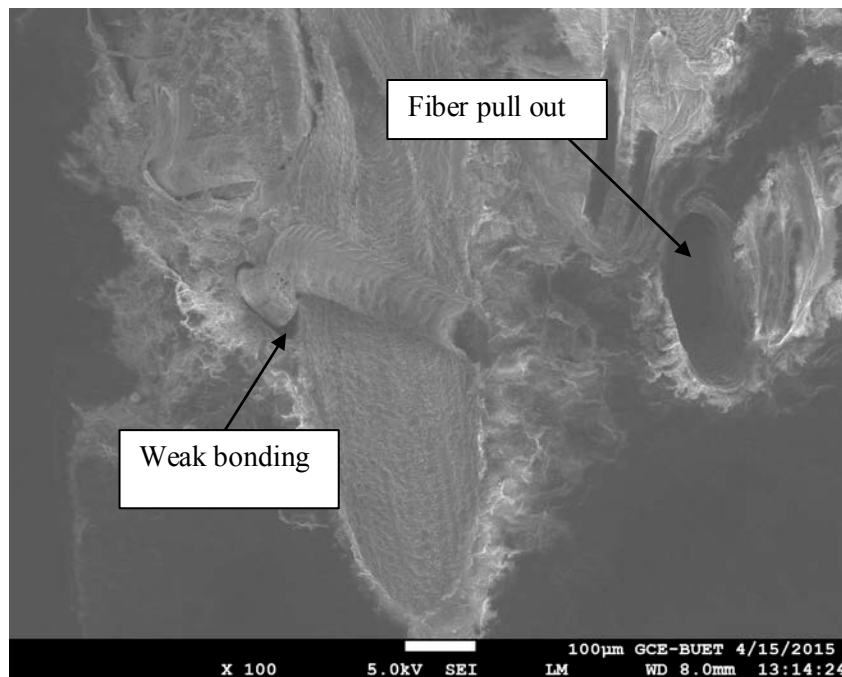


Figure 42 : SEM micrograph of Tensile fracture surface of 3L 10 wt% areca waste nylon fiber (areca: nylon=1:1) fiber reinforced PP composite.

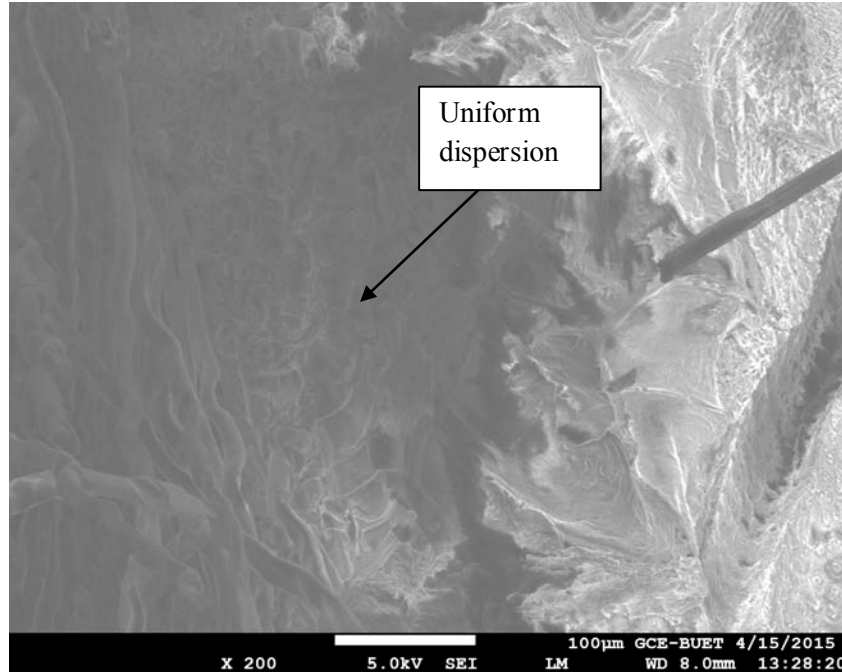


Figure 43 : SEM micrograph of Tensile fracture surface of 4L 10 wt% areca waste nylon fiber (areca: nylon=1:1) fiber reinforced PP composite.

The tensile fracture surface morphologies of 4L treated areca and waste nylon (areca: nylon=1:1) 10, 15, 20, 25 wt.% fiber reinforced hybrid polypropylene composites are shown in " **Figure 44, Figure 45, Figure 46 and Figure 47**" respectively.

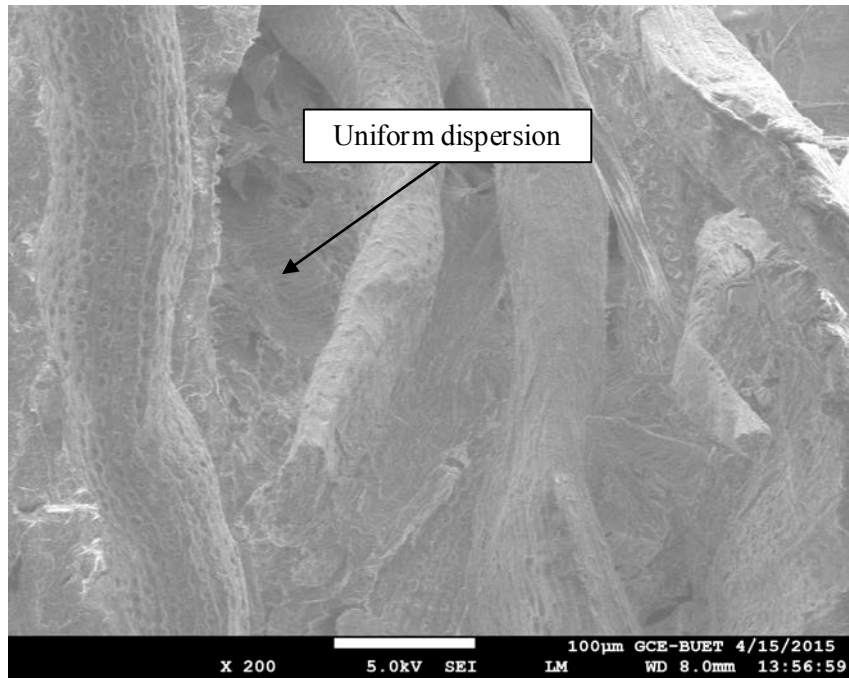


Figure 44 : SEM micrograph of Tensile fracture surface of 4L 10 wt% areca waste nylon fiber (areca : nylon=1:1) fiber reinforced PP composite.

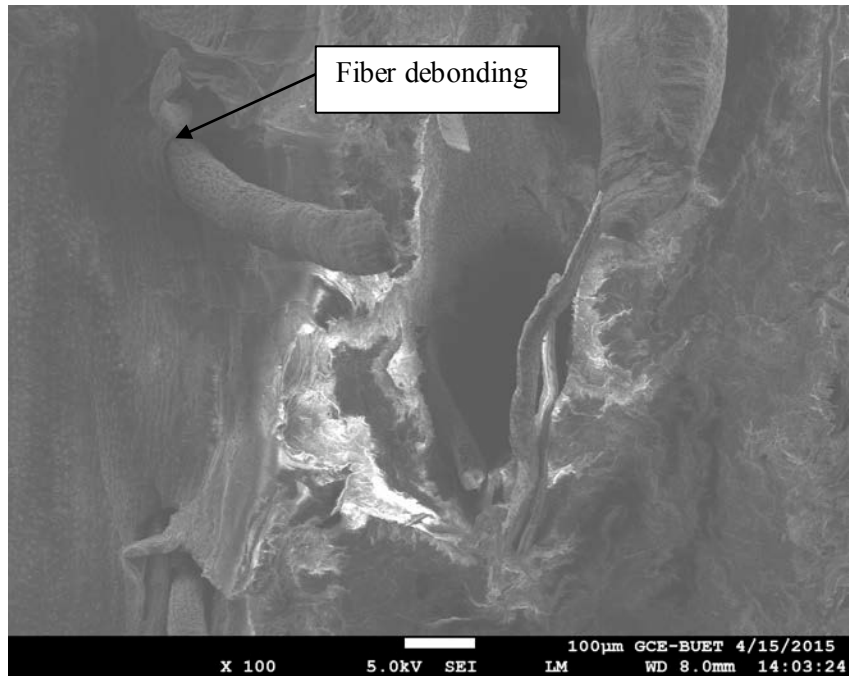


Figure 45 : SEM micrograph of Tensile fracture surface of 4L 15 wt% areca waste nylon fiber (areca : nylon=1:1) fiber reinforced PP composite.

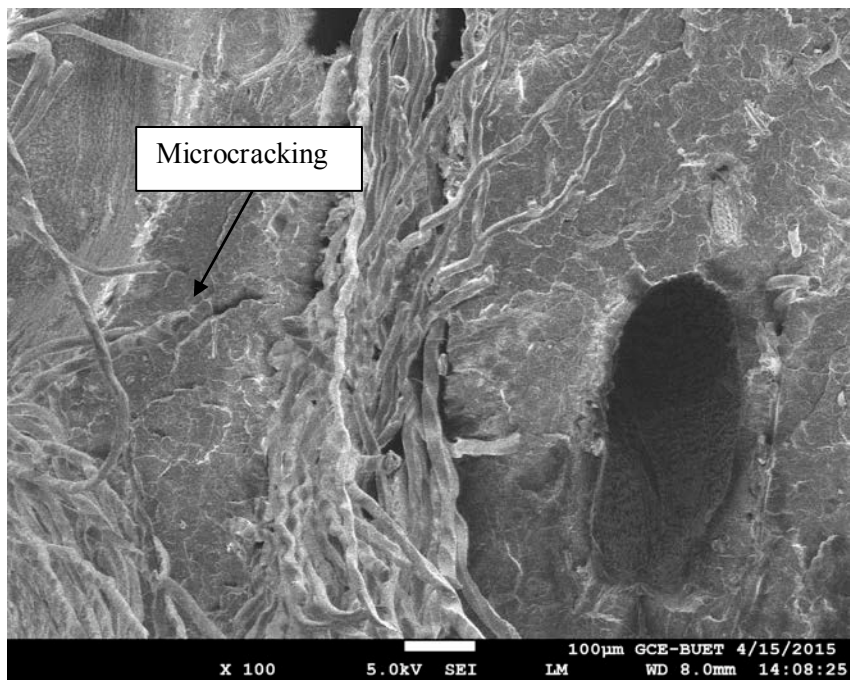


Figure 46 : SEM micrograph of Tensile fracture surface of 4L 20 wt% areca waste nylon fiber (areca : nylon=1:1) fiber reinforced PP composite.

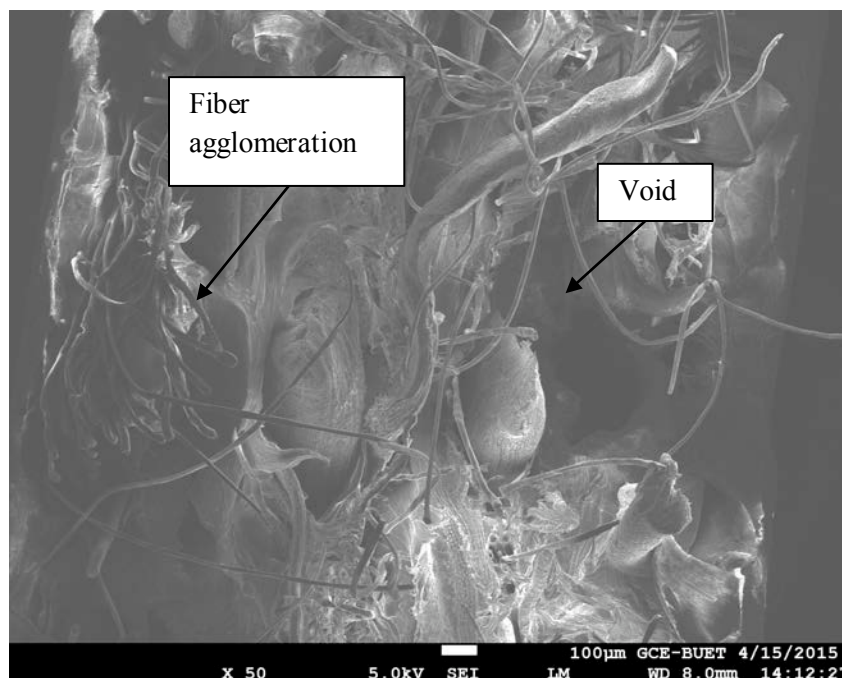


Figure 47 : SEM micrograph of Tensile fracture surface of 4L 25 wt% areca waste nylon fiber (areca : nylon=1:1) fiber reinforced PP composite.

Comparison of SEM micrograph of untreated and treated fiber reinforced composites showed some gaps between the fiber and matrix in untreated composites revealing weak interfacial bonding whereas sodium hydroxide treated fiber composites showed improved fiber matrix adhesion.

Mechanical interlocking involves diffusion of polymer matrix into porous fiber surface. The polymer matrix will flow into the porous fiber surface and the embedded polymer will solidify in the pores and become inextractable [52]. As shown in " **Figure 44**", short betel areca fibers are arrayed with random distribution in the polypropylene matrix composites. As clearly seen in the micrograph both fiber pull-out traces and the agglomeration of fiber in the matrix have substantially reduced providing evidence of better dispersion of fiber in matrix in alkali treated fiber reinforced composites [89].

The surface morphology of 15, 20 and 25 wt.% fiber reinforced tensile fractured surfaces reflected the reasons why mechanical properties of composites got worsened with the increase in fiber wt%. From those micrographs it was quite obvious that the number of fiber agglomeration, fiber pullout and the presence of micro voids enhanced with the fiber percentage enhancement

[90]. Also comparison of 10 and 20 wt% fiber reinforced composite revealed the presence of matrix cracking in the later one which was not found in the former one. This was also one of the reasons of better tensile strength of the 10 wt% fiber reinforced composites.

The tensile fracture surface morphologies of 4L treated 20 wt% areca and waste nylon fiber reinforced hybrid polypropylene composites where the fibers are reinforced in various ratio (areca: nylon= 3:1, 1:3, 2:3, 3:2) are shown in " **Figure 48, Figure 49, Figure 50 and Figure 51**" respectively.

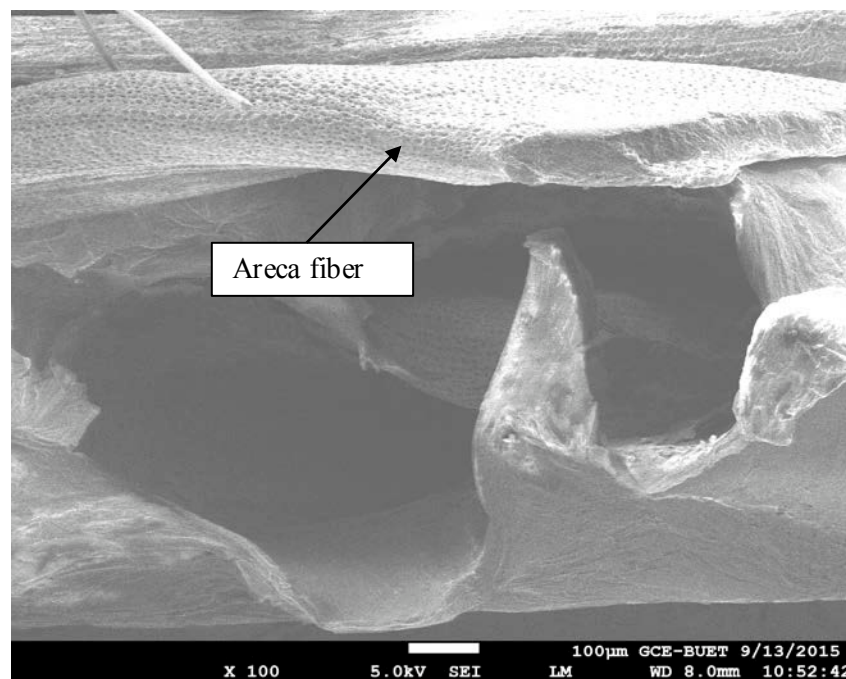


Figure 48: SEM micrograph of Tensile fracture surface of 4L 20 wt% areca waste nylon fiber (areca : nylon=3:1) fiber reinforced PP composite.

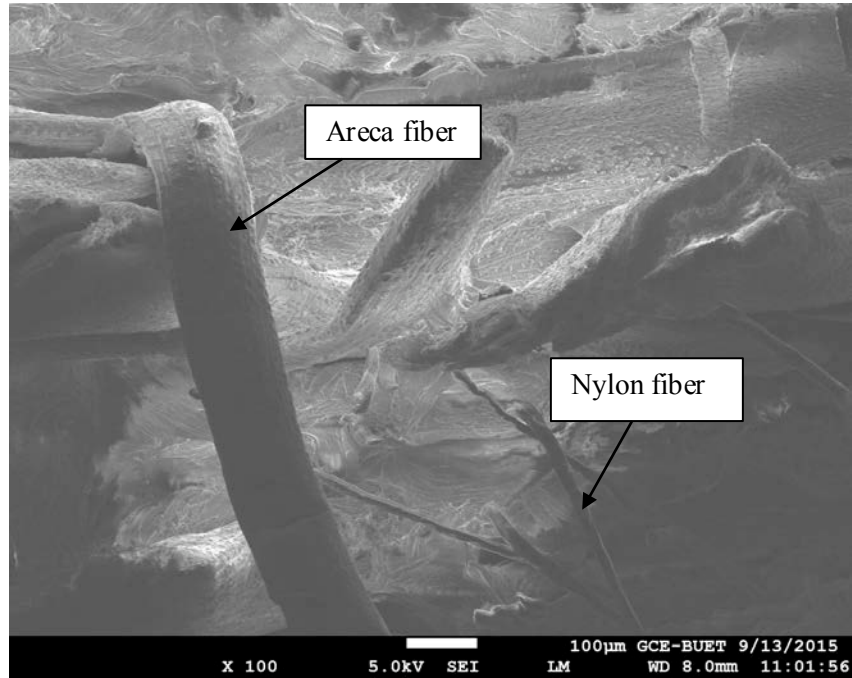


Figure 49 : SEM micrograph of tensile fracture surface of 4L 20wt% areca waste nylon fiber (areca : nylon=3:2) fiber reinforced PP composite.

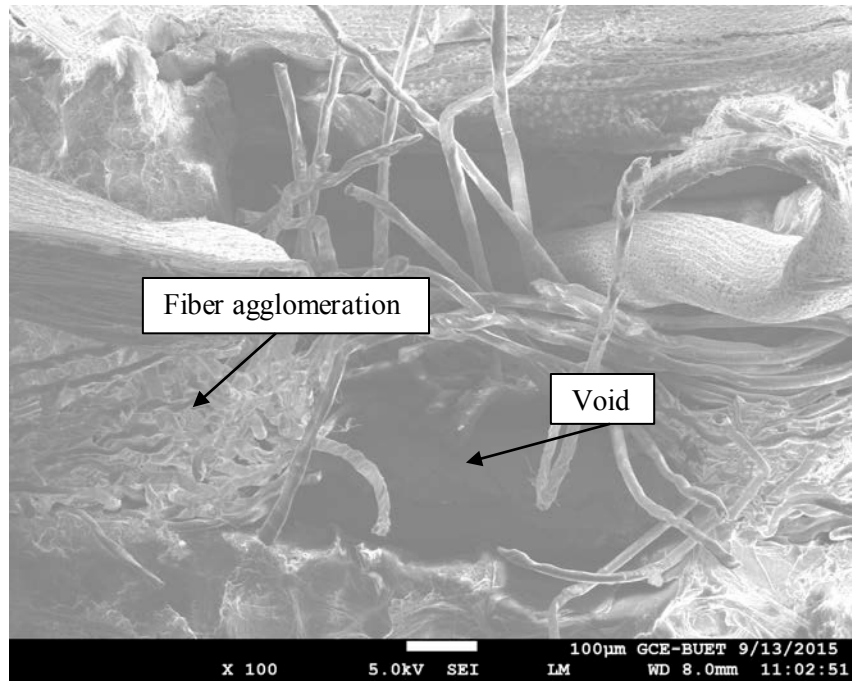


Figure 50 : SEM micrograph of tensile fracture surface of 4L 20wt% areca waste nylon fiber (areca: nylon=2:3) fiber reinforced PP composite.

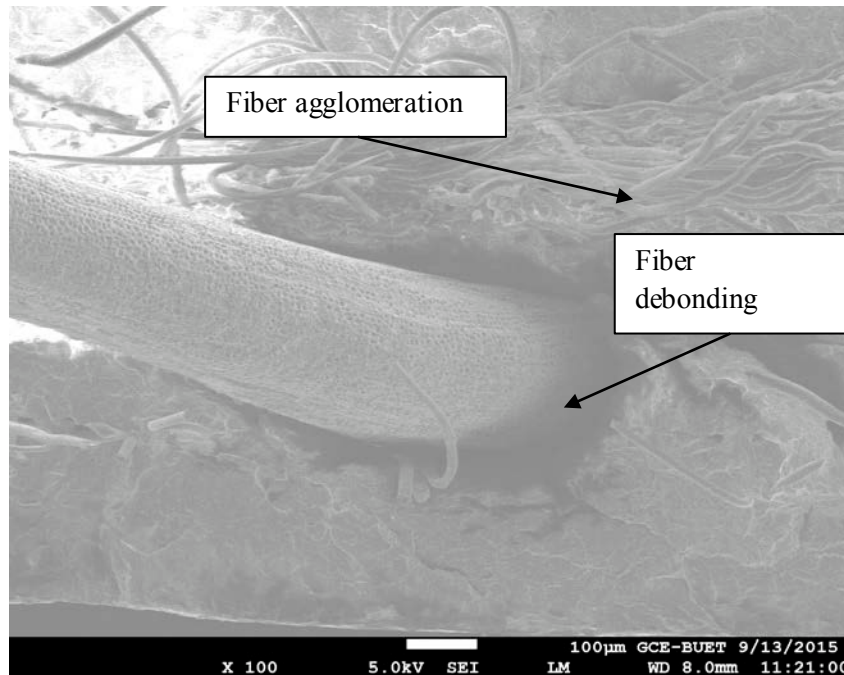


Figure 51 : SEM micrograph of tensile fracture surface of 4L 20wt% areca waste nylon fiber (areca : nylon=1:3) fiber reinforced PP composite.

From those micrographs the presence of fiber matrix debonding and fiber pullout all were quite evident. Fiber agglomeration was also found in all those micrographs ,however it was quite huge in the composite in which percentage of nylon fiber was high.

Thermo gravimetric Analysis

The TGA curves of 3 layered (3L) and 4 layered (4L) 10 wt% areca and waste nylon fiber reinforced polypropylene (PP) composites are shown in "**Figure 52 and Figure 53**".In both cases they obtained TGA curves represents one-stage decomposition and therefore represents the thermal stability limit of the composite.

The starting of weight loss temperature has been calculated by using extrapolation technique because it's a reproducible temperature calculation and it is specified to be used by ASTM [91].

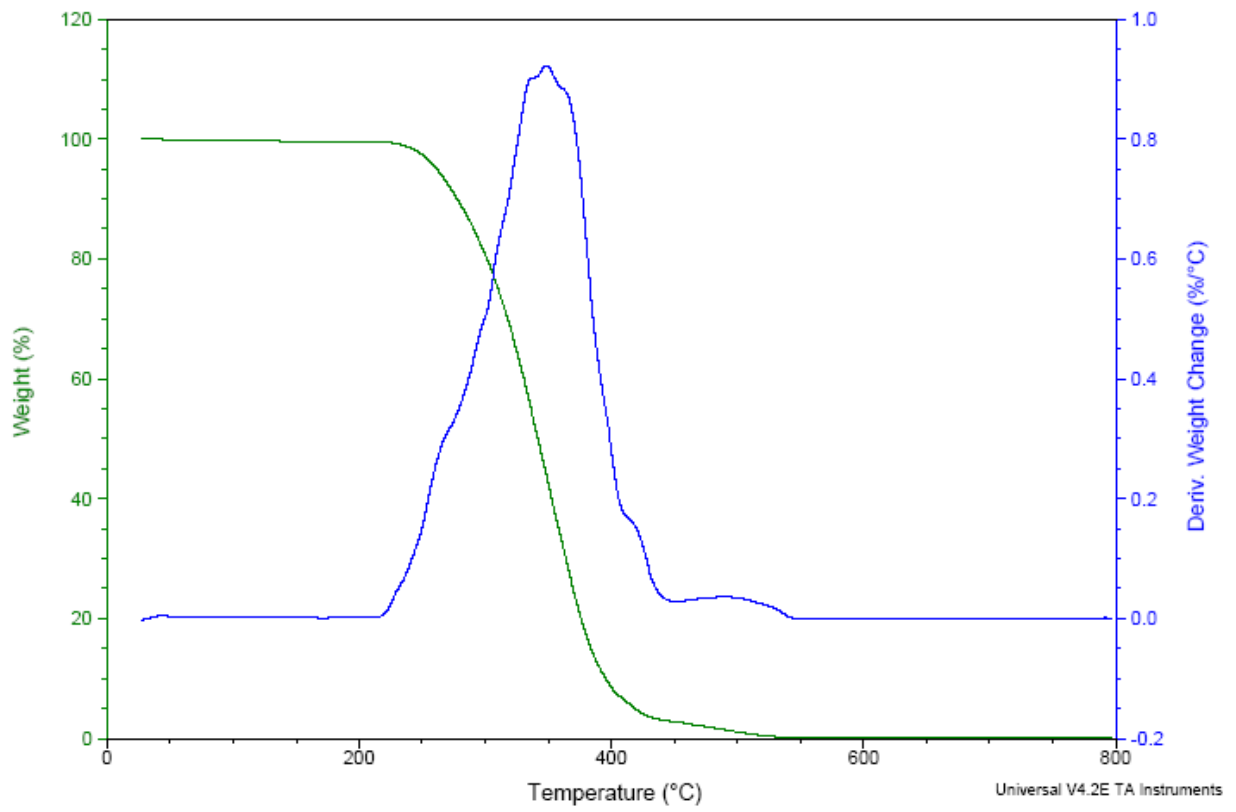


Figure 52 : TGA curve of 3L 10 wt% untreated areca waste nylon fiber(areca : nylon=1:1) reinforced PP composite

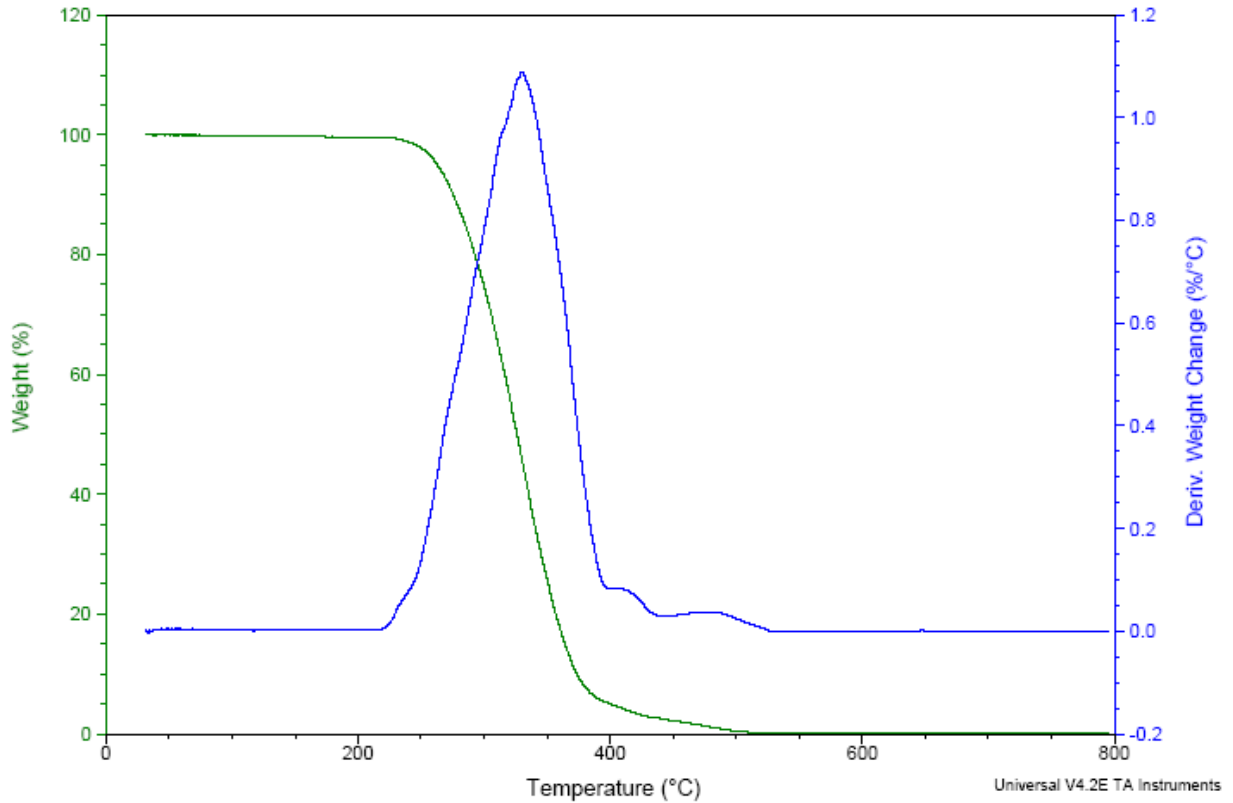


Figure 53 : TGA curve of 4L 10 wt% untreated areca waste nylon fiber (areca : nylon=1:1) reinforced PP composite

From "Figure 52" it can be seen that in case of 3 layered fibers reinforced composite thermal degradation starts at around 280.07 °C and the decomposition starting temperature does not differ much in case of 4 layered fiber reinforced composite without slight enhancement. From this it can be concluded that the composites have lower thermal stability than pure polypropylene which starts to decompose in around 350 °C [43] and this occurs due to the presence of less thermally stable fibers. However, around 2.5% residual products remain in 4 layered fiber reinforced composite, the amount is slightly higher in case of 3 layered fiber reinforced composites [43,92].

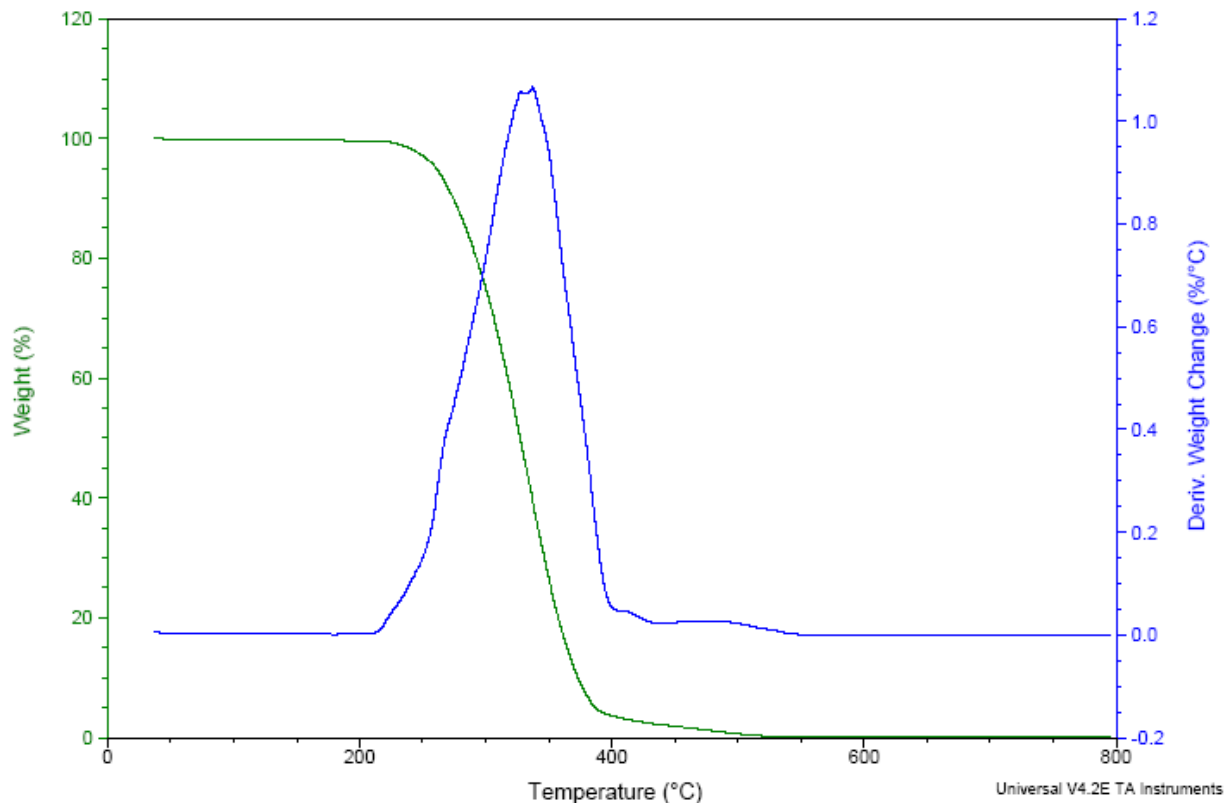


Figure 54 : : TGA curve of 4L 10 wt% treated areca waste nylon fiber (areca : nylon=1:1) reinforced PP composite

For the manufacturing of composites, the thermal stability of the fiber is very important. In our study alkali treatment has shown little enhancement of thermal stability of the composite. For 4 layered composite reinforced with treated areca fiber decomposition starts at 289.08°C.

In DTG curve of all these composites no peak was observed below 200°C. The major peak at about 320°C is due to the thermal degradation of cellulose which is the main component of areca fiber. Also synthetic waste fiber nylon 66 degrades at around 342°C [93].

The thermal degradation of hemicelluloses occurred at around 200-260°C. Between 150°C and 300°C, cleavage of α - and β -aryl-alkyl-ether linkages of lignin occurred. At around 300°C, aliphatic side chains started to split off from the aromatic ring. Finally, the carbon-carbon linkage between lignin structural units was cleaved at 370–400°C. The glycosidic linkage of cellulose degrades at around 260°C [94]. The peak of degradation at around 473°C was due to the degradation of polypropylene [95].

It is clearly seen that slightly higher peak of hemicelluloses in case of untreated fiber reinforced composite at around 260°C has got vanished in case of treated reinforced composite due to the considerable removal of hemicelluloses during alkaline treatment. The third peak has shown slight increase in weight loss than the untreated fiber reinforced composite. Also the peak of lignin at 370-400°C has also got lowered in case of treated fiber reinforced composite.

On the other hand, the TGA curves of the treated fiber reinforced composite shifted to slightly higher temperature, these demonstrated the slightly higher thermal stability of treated fiber. The residual char left at 400°C increased considerably from 7% to 10.5% in the cases of treated fiber reinforced composite. It is proposed that besides removing hemicellulose, NaOH treatment can give rise to a lignin-cellulose complex, therefore the NaOH treated fiber becomes more stable and this has reflected in the increased amount of residual char.

CHAPTER 5

CONCLUSION

CONCLUSION

- In the present research hybrid composites were fabricated with hot press technique incorporating 2, 3 and 4 layers of natural (e.g. areca) and textile (e.g. nylon) fibers at constant fiber loading of 10 wt% in polypropylene matrix. The tensile strength of the hybrid composites increased with increasing number of fiber layers. However 5% alkali treated fiber reinforced composites showed higher tensile strength than raw fiber reinforced composites. In 5% alkali treated fiber containing composites, the enhancement of compatibility of the fiber with PP resulted into better stress transfer from the matrix to the fiber and thus increase in tensile strength in comparison to raw ones occurred. Tensile strength also showed decreasing trend with enhancement of fiber wt%. Higher percentages of areca containing composite displayed better tensile properties than the higher nylon containing one.
- % Elongation at break for fiber reinforced composite increased with increasing number of fiber layers, however 5% NaOH treated fiber reinforced composites showed better value than that of the raw fiber reinforced composite with same volume fraction of fiber. % Elongation at break decreased with the enhancement of fiber wt%. Composite containing higher percentages of nylons showed better elongation at break than that of the other systems.
- It was also observed that Young's modulus increased with the increase of number of fiber layers and fiber volume fraction. 5% NaOH treated fiber reinforced composite exhibited higher Young's modulus than the raw one. Amount of nylon fiber enhancement resulted in higher Young's modulus value.
- Flexural strength and flexural modulus increased with the increase in number of fiber layers. The flexural strength and flexural modulus of 5% alkali treated fiber reinforced composite was higher than that of the raw one. Though these properties increased with the fiber wt% up to a certain level, then it decreased again. Higher amount of nylon fiber containing composites demonstrated better flexural properties.

- SEM was carried out to reveal the adhesion between the fiber and the matrix and also the tensile fracture surfaces of different composites. FTIR was carried out to study the effect of alkali treatment which displayed the removal of hemicelluloses.
- In conclusion it can be said that areca and waste nylon can give a better prospect in improvement of hybrid composite. Changing their ratio resulted in different combination of properties that could not be obtained using one fiber alone. Above all chemical modification of fiber surface can also help to obtain better set of properties.
- The widespread application of natural fiber is in automotive industry. Plant fibers are mainly used in the part of car interior and truck cabins. Hopefully this composite will be able to used in producing various automotive parts such as various panels, shelves, trim parts and brake shoes.

FUTURE WORK

- Development of new composite products from the easily renewable natural materials has a strong potential to deliver novel biodegradable and/or readily recyclable materials suitable for the automotive, packaging industry and all other applications. Hybrid composite reinforced with natural and synthetic fibers provide biodegradable products, easy way of recycling in conjunction with balanced pursuit of properties.
- In our recent study only fiber surface modification was carried out by alkali treatment. Apart from alkali treatment large numbers of other chemical treatments like addition of coupling agent are available which can be used for fiber surface modification.
- To enhance compatibility matrix modification can also be carried out. In this research work composite was prepared by reinforcing different number of fiber layers but fiber orientation was remain unchanged. To obtain different combination of properties fiber orientation can be changed.

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Table 2 Tensile test results of areca waste nylon fiber-PP composites reinforced with different number of fiber layer

Sample	Sample No	Tensile Strength (MPa)	Average Tensile Strength (MPa)	Young's Modulus (GPa)	Average Young's Modulus (GPa)
2 Layered 10 wt.% (areca: Nylon=1:1) fiber reinforced PP composites (2L)	1	24.53	24.49	1.10	1.18
	2	24.46		1.23	
	3	24.49		1.21	
3 Layered 10 wt.% (areca: Nylon=1:1) fiber reinforced PP composites (3L)	1	26.65	27.12	1.19	1.22
	2	27.52		1.25	
	3	27.19		1.22	
4 Layered 10 wt.% (areca: Nylon=1:1) fiber reinforced PP composites (4L)	1	28.13	28.16	1.39	1.43
	2	28.15		1.45	
	3	28.19		1.45	

Table 3 Elongation at break result of areca waste nylon fiber-PP composites reinforced with different number of fiber layers

Sample	Sample No	% Elongation at Break	Average % Elongation at Break
2 Layered 10 wt.% (areca: Nylon=1:1) fiber reinforced PP composites (2L)	1	3.62	3.7
	2	3.75	
	3	3.73	

3 Layered 10 wt.% (areca: Nylon=1:1) fiber reinforced PP composites (3L)	1	4.02	4.05
	2	4.08	
	3	4.05	
4 Layered 10 wt.% (areca: Nylon=1:1) fiber reinforced PP composites (4L)	1	5.35	5.1
	2	4.98	
	3	4.97	

Table 4 Tensile test results of areca waste nylon fiber-PP composites reinforced with different fiber wt.

Sample	Sample No	Tensile Strength (MPa)	Average Tensile Strength (MPa)	Young's Modulus (GPa)	Average Young's Modulus (GPa)
4 Layered 10 wt.% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	28.075	28.39	1.584	1.58
	2	28.52		1.579	
	3	28.57		1.565	
4 Layered 15 wt.% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	27.01	27.05	1.736	1.71
	2	27.09		1.71	
	3	27.05		1.68	
4 Layered 20 wt.% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	26.98	26.92	1.79	1.76
	2	27.09		1.74	
	3	26.69		1.76	
4 Layered 25 wt.% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	24.563	25.47	1.81	1.81
	2	24.462		1.78	
	3	24.389		1.84	

Table 5 Elongation at break result of areca waste nylon fiber-PP composites reinforced with different number of fiber layers

Sample	Sample No	% Elongation at Break	Average % Elongation at Break
4 Layered 10 wt% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	6.58	6.98
	2	7.01	
	3	7.35	
4 Layered 15 wt% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	6.05	6.11
	2	6.10	
	3	6.18	
4 Layered 20 wt% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	5.89	5.94
	2	5.92	
	3	6.01	
4 Layered 24 wt% (treated areca: Nylon=1:1) fiber reinforced PP composites	1	5.68	5.8
	2	6.27	
	3	5.45	

Table 6 Tensile test results of areca waste nylon fiber-PP composites reinforced with different fiber ratio.

Sample	Sample No	Tensile Strength (MPa)	Average Tensile Strength (MPa)	Young's Modulus (GPa)	Average Young's Modulus (GPa)
4 Layered 20 wt.% (treated areca: Nylon=3:1) fiber reinforced PP composites	1	27.75	28.45	1.61	1.59
	2	28.13		1.57	
	3	29.47		1.53	
4 Layered 20 wt.% (treated areca: Nylon=3:2) fiber reinforced PP composites	1	26.76	27.03	1.72	1.72
	2	27.08		1.78	
	3	27.25		1.76	
4 Layered 20 wt.% (treated areca: Nylon=2:3) fiber reinforced PP composites	1	26.65	26.65	1.80	1.84
	2	26.38		1.89	
	3	26.91		1.83	
4 Layered 20 wt.% (treated areca: Nylon=1:3) fiber reinforced PP composites	1	24.76	25.11	1.89	1.88
	2	25.01		1.86	
	3	25.55		1.89	

Table 7 Elongation at break result of areca waste nylon fiber-PP composites reinforced with different fiber ratio.

Sample	Sample No	% Elongation at Break	Average % Elongation at Break
4 Layered 20 wt.% (treated areca: Nylon=3:1) fiber reinforced PP composites	1	4.44	4.37
	2	4.31	
	3	4.36	
4 Layered 20 wt.%	1	5.32	5.34

(treated Nylon=3:2) reinforced composites	areca: fiber PP	2	5.31	
		3	5.39	
4 Layered 20 wt.% (treated Nylon=2:3) reinforced composites	areca: fiber PP	1	6.61	6.52
		2	6.58	
		3	6.37	
4 Layered 20 wt.% (treated Nylon=1:3) reinforced composites	areca: fiber PP	1	7.29	7.34
		2	7.41	
		3	7.32	

Table 8 Flexural test result of areca waste nylon fiber-PP composites reinforced with different number of fiber layers

Sample	Sample No	Flexural Strength (MPa)	Average Flexural Strength (MPa)	Flexural Modulus (GPa)	Average Flexural Modulus (GPa)
2 Layered 10 wt% (areca: Nylon=1:1) fiber reinforced PP composites (2L)	1	30.8	31.8	1.98	2.1
	2	32.5		2.3	
	3	32.1		2.02	
3 Layered 10 wt% (areca: Nylon=1:1) fiber reinforced PP composites (3L)	1	34.1	33	2.35	2.4
	2	33.7		2.44	
	3	31.2		2.41	
4 Layered 10 wt% (areca: Nylon=1:1) fiber reinforced PP composites	1	34.5	33.3	2.67	2.7
	2	33.6		2.73	
	3	31.8		2.7	

(4L)					
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Table 9 Flexural test result of areca waste nylon fiber-PP composites reinforced with different fiber ratio.

Sample	Sample No	Flexural Strength (MPa)	Average Flexural Strength (MPa)	Flexural Modulus (GPa)	Average Flexural Modulus (GPa)
4 Layered 20 wt% (treated areca: Nylon=3:1) fiber reinforced PP composites	1	27.71	28.19	2.59	2.68
	2	28.67		2.71	
	3	28.19		2.74	
4 Layered 20 wt% (treated areca: Nylon=3:2) fiber reinforced PP composites	1	34.02	34.29	3.02	2.99
	2	34.45		2.97	
	3	34.40		2.98	
4 Layered 20 wt% (treated areca: Nylon=2:3) fiber reinforced PP composites	1	37.10	37.36	3.45	3.395
	2	36.67		3.32	
	3	38.31		3.42	
4 Layered 20 wt% (treated areca: Nylon=1:3) fiber reinforced PP composites	1	38.21	38.87	3.61	3.65
	2	37.87		3.67	
	3	40.53		3.67	

Table 10 hardness result of areca waste nylon fiber-PP composites reinforced with different number of fiber layers

Sample	Sample No	SHORE hardness A	Average SHORE hardness A
2 Layered 10 wt% (areca: Nylon=1:1) fiber reinforced PP composites (2L)	1	92.7	92.6
	2	92.3	
	3	92.8	
3 Layered 10 wt% (areca: Nylon=1:1) fiber reinforced PP composites (3L)	1	94.3	94.3
	2	94.5	
	3	94.1	
4 Layered 10 wt% (areca: Nylon=1:1) fiber reinforced PP composites (4L)	1	95.4	95.2
	2	95.4	
	3	95	

Table 11 : Hardness test results of areca waste nylon fiber-PP composites reinforced with different fiber wt.%.

Sample	Sample No	SHORE hardness A	Average SHORE hardness A
4 Layered 20 wt% (treated areca: Nylon=3:1) fiber reinforced PP composites	1	96.3	96.4
	2	96.2	
	3	96.7	
4 Layered 15wt% (treated areca: Nylon=3:2) fiber reinforced PP composites	1	96.9	96.8
	2	96.7	
	3	96.8	

4 Layered 20 wt% (treated areca: Nylon=2:3) fiber reinforced PP composites	1	97.5	97.6
	2	97.7	
	3	97.6	
4 Layered 25wt% (treated areca: Nylon=1:3) fiber reinforced PP composites	1	97.9	97.8
	2	97.6	
	3	97.9	

Table 12 Hardness results of areca waste nylon fiber-PP composites reinforced with different fiber ratio.

Sample	Sample No	SHORE hardness A	Average SHORE hardness A
4 Layered 20 wt.% (treated areca: Nylon=3:1) fiber reinforced PP composites	1	95.1	95.2
	2	95.2	
	3	95.3	
4 Layered 20 wt.% (treated areca: Nylon=3:2) fiber reinforced PP composites	1	96.8	96.5
	2	96.7	
	3	96	
4 Layered 20 wt.% (treated areca: Nylon=2:3) fiber reinforced PP composites	1	98.1	98.2
	2	98.4	
	3	98.1	

4 Layered (treated Nylon=1:3) reinforced composites	20 wt.% areca: fiber PP	1	98.9	98.8
		2	98.8	
		3	98.7	