Mechanical and Thermal Characterization of Bauhinia Vahlii Reinforced PP Composite

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ABSTRACT

In today’s scenario, natural fibers are used over synthetic fibers in fibers reinforced composites. Forthis present analysis, polypropylene composites with the content of Bauhinia vahlii stem fibers 10 wt% to 30 wt% will be prepared via Compression Moulding and characterised. The bauhinia vahlii stem fibers were modified with different chemical drafting and characterized. The mechanical, chemical, thermal and morphological analysis of the fiber as well as the composites will be carried out.

KEYWORDS

Bauhinia Vahlii Reinforced PP CompositePolypropylene Resin
Mechanical and Thermal CharacterizationBV Fibers
Coupling Agent

1. Introduction

The composite materials reinforced with natural fibers offered a broad scope of properties, which are reasonable for the vast number of building and development enterprises (roof framing, parcel sheets), packaging, shopper items, and designing the application. To produce cost-effective composites and decrease the burden on environment, investigators have found new manufacturing trends for composite using natural fibers as reinforcement filler. Their great accessibility, low cost, low density, and good mechanical properties make them an attractive organic alternative over synthetic fiber [1]. The natural fiber-reinforced composites are eco-friendly, and their bio-degradability characteristics play a vital role in solving environmental problems. However, due to some shortcomings, the natural fibers restrict their use in a composite. This chapter aims to discuss various methods to improve the properties of the natural fiber and how considerable improvements in the performance of the composite can be accomplished are discussed.

The interest in NFC (natural fiber-reinforced composite material) is increasing day by day. Natural fiber composites (NFC) became an alternative for glass or carbon reinforced composite. Natural fiber- reinforced composite contains natural fiber as reinforcement filler. The natural fibers have several tailor-made properties like bio-degradability, high aspect ratio, lightweight, high specific modulus, non-toxic, easy for processing, and plenty available in the world [2]. These benchmarking properties challenge the replacement of synthetic fiber and lessen plastic utilization. The comparison between natural fiber and synthetic fiber is presented in Table 1. The composite prepared from the natural cellulosic fiber is environment friendly and has a wide range of industrial applications [3]. The chemical composition of natural fiber is presented in Figure 1. The natural fibers are also called lingo- cellulosic fiber, mainly packed with cellulose bounded by hemicelluloses and lignin compounds. The major constituent of the plant fiber is the crystalline packed cellulose, which provides the fiber's strength, structural stability, and stiffness. It is a linear polymer. Natural fibers mechanical properties depend on its cellulose type because each type of cellulose has its cell structure and condition, which determine the mechanical properties [4]. Hemicellulose which exists in amorphous form is responsible for the formation of fiber network. The degree of polymerization of native cellulose is ten to one hundred times higher than that of hemicellulose. Both the cellulose and hemicelluloses are hydrophilic.

In the plant cell wall, another compound having an amorphous structure is the lignin. Lignin is a complex hydrocarbon. It is an aromatic compound that acts as an adhesive and hydrophobic. Lignin fills the gap between other elements in the plant cell wall to have structural integrity. The other constituents of the plant fiber are pectin, fat and waxy substances. Natural fiber's major drawback is the hydrophilic nature, which makes it incompatible with the hydrophobic matrix. The structural composition of fiber absorbs a significant amount of moisture and results in poor adhesion between the fiber and matrix. Hence, to overcome this pitfall, various methods are adopted to improve the interfacial adhesion between fiber and matrix, resulting in improved performance of the resulting composite [5].

REVIEW OF LITERATURE FACTORS AFFECTING COMPOSITE PROPERTIES

The properties of natural fiber reinforced composites predominantly depend on various factors such as fiber structure, fiber loading percentage, size and orientation of fibers, degree of interfacial adhesion, fiber surface treatments, thermal stability of fibers, moisture absorption of fibers, presence of voids, hybridization and incorporation of fillers and coupling agents [6]. The details are discussed below.

1.1 FIBER STRUCTURE

Natural fiber's structural composition consists of cellulose, hemicellulose, lignin, pectin, and waxy substances. The cellulosic molecular structure is distinguished through the crystalline or amorphous region. In the crystalline area, the molecules are arranged in a closely packed manner. This forms cellulose blocks by a strong hydrogen bond. Also, increases cellulose content resultsin improved tensile strength. In the amorphous region, the water molecules are being absorbed by hemicellulose, lignin, pectin, and waxy substances. This made the fiber hydrophilic/polar and became incompatible with the hydrophobic/non-polar matrix. Therefore, to eliminate the hydroxyl group, enlarging the crystalline region, and removing waxy substances, natural fiber surface needs to be modified to enhance interfacial bonding effectiveness.

1.2 FIBER ASPECT RATIO, LOADING AND ORIENTATION

The mechanical properties of the composites strongly depend on fiber and matrix properties and their interfacial shear strength. Fibers are the backbone of the composite material, which carry most of the load. The fiber length plays a vital role in controlling the strain-to-failure. The matrix transfers the load by shear along the fiber length at the interface. The composite's strength and stiffness depend on fiber loading, fiber orientation, and interfacial bonding between fiber and matrix. Fibers may be oriented longitudinally, transversely, and randomly. Higher tensile strength is obtained in case of longitudinally oriented fiber composite but due to buckling of fiber, there is decline in compressive strength. However, in transversely oriented fiber composite matrix strength is higher than the tensile strength of the fiber. Lastly, in the case of randomly oriented short fiber different mechanical properties were obtained due to the complications in the distribution of the load. Hence, enhancement of mechanical properties can be done by controlling the aspect ratio and fiber orientation.

1.3 PRESENCE OF VOIDS

Void formation in the composite plays an important role. During fabrication, air may be trapped inside the composite, which results in poor mechanical properties. The curing and cooling rate aremainly responsible for void formation. Sudden failure of the composite occurs after the curing process due to the formation of micro-voids. Also, high fiber content leads to high void content, which in turn results in variation in mechanical properties.

1.4 MOISTURE ABSORPTION OF FIBERS

The fibers are very much sensitive to moisture absorption due to their hydrophilic nature. When the fiber expose to atmosphere, the hydrogen bonds present in the fiber cell wall break. The hydroxyl group present in the amorphous part of the fiber structure holds the water molecules, which can easily penetrate the moisture. When fiber and matrix interact, swelling of the fiber occurs. This results in weak interfacial adhesion, and matrix cracking occurs. Finally, there is dimensional instability and poor mechanical property.

1.5 THERMAL STABILITY OF THE FIBER

The thermal stability of the natural fiber depends on the constituents of the fiber. The constituents like cellulose, hemicellulose, lignin etc, degraded at different temperatures. It was observed that cellulose starts degrades at 200℃, and hemicellulose, lignin, pectin degrades at temperature range from 160-350℃. Due to the higher moisture content of the fiber, the thermalstability decreases.

1.6 PROPERTY ENHANCEMENT METHOD OF NATURAL FIBER

The physical, chemical, mechanical, and thermal properties of the natural fiber-based composite depend on the type of fiber-matrix, properties, and interfacial bonding between fiber-matrix. But, due to the natural fiber's hydrophilic nature, there is an incompatibility between fiber and matrix. So, there are various methods by which the compatibility betweenthe two components can be achieved as discussed below.

1.7 CHEMICAL TREATMENT OF FIBER

The fiber and matrix interface is a diffusion or reaction region. In this interphase, both fiberand matrix are chemically as well as mechanically merged. Stronger the interfacial bond between fiber/matrix, better will be the mechanical properties of the composite [3]. The primary issue of natural fiber composites is the hydrophilic nature of the fiber and hydrophobicnature of the matrix. The compatibility between these two-phase results in weak interfacial adhesion. To enhance the interfacial adhesion between fiber/matrix, fiber surface should be reformed with various chemical treatments. Chemical treatments activated the hydroxyl groups and exposed more reactive groups on the fiber surface, which encourages good bonding with the matrix resulting in enhanced properties of the composites. Many research works have been conducted to improve the inter locking of fiber/matrix through various chemical treatments such as alkali, acetylation, benzoylation, peroxide, acrylation, and isocyanates, permanganate, stearic acid, fungal, and others.

1.8 PHYSICAL TREATMENT OF FIBER

The structural and surface properties of fiber can be changed by physical treatment. Various physical therapies are plasma, corona, heat treatment, and steam pre-treatment. In plasma treatment, by gas feed, an ionized region is formed [8]. This carries high-energy photon, ions, electrons, radicals, which alter the surface of the material. By corona treatment, the corona effect is produced. At atmospheric pressure and low temperature, the corona effect is more prominent, which is a function of high energy electromagnetic field. This results in ionization where excited ions, radicals are present. Later, these are active in surface modification. In case of heat treatment, fiber is heated at a temperature close to its degradation temperature. Due to this, heating cellulose is heated and undergoes physical and chemical changes. This change affects the weight, strength, crystallinity, and degree of polymerization. The stream pre- treatment is also called steam stabilization. Through hydrothermal treatment, cellulosic fiber's deformation occurs at high temperature and stabilizes the fibers in a typical configuration.

1.9 USE OF COMPATIBILIZER OR COUPLING AGENT

Besides the surface treatment of fibers, the composite properties can be improved by utilization of compatibilizer or coupling agents to transfer stress across the interface effectively. The compatibilizer can be polymers, for example, malleated ethylene, malleated propylene and acrylic grafted linear polymer [9]. Among all compatibilizer, malleated coupling agents give effective interfacial bond among fiber and network. Maleic anhydride eliminates the hydroxyl groups from the fiber cell wall and results in decreased hydrophilic inclination. The addition of coupling agents additionally prompting improved properties of the composite. The coupling agent creates a chemical bridge at the fiber-matrix interface. Various coupling agents are silane, zirconate, or titanate. Using a coupling agent wettability of the fiber can be increased, and interfacial locking can be improved. Utilizing coupling specialist wettability of the fiber can be expanded, and interfacial locking can be improved.

1.10 PLASMA SURFACE MODIFICATION

Plasma is comprising of free electrons and ions. Plasma can be generated by warming gasses to very high temperatures or by exposing the gas to strong electromagnetic fields. By following both the ways electrons can generate from the atoms and molecules. Generally, these electrons have enough dynamic energy, so that if there is an occurrence of collision, produces further electrons from molecules and atoms. These impacts make ionization measure, making new ions and electrons, which are adjusted by the recombination of these specimens until the plasma attains at equilibrium. The molecular bindings of molecules present in the plasma may be broken, because of the collisions with electrons and ions, resulting production of new highly reactive and unstable molecules and monatomic specimens.

1.11 COLD PLASMA

This type of plasma are produced by electric or electromagnetic fields maintaining low temperature, same as room temperature, so that the damage to the materials i.e. being treated can be avoided. For continuous ionization there is a requirement of maintaining high temperature, however it is possible to keep the plasma temperature low by maintaining a low degree of ionization. This implies that only a small fraction (less than 1%) of all the molecules and atoms in the gas are ionized at any given moment. As a result, the average kinetic energyof the molecules, atoms and ions is low, however the electrons are accelerated to high velocities due to their much lower mass. The cold plasma is the outcome of thermal equilibrium, of cold heavy particles, i.e. atoms, molecules and ions and hot electrons.

Generally, the plasma treatments are made at vaccum, maintaining the pressure ranging from 0.1pascal to 100pascal depending on the type of application and technique. These low-pressure plasmas are very useful for the surface modification of polymer and textile.

However, the plasma operation at atmospheric pressure have become popular these days because of its simplicity, still there is some drawbacks when the process being carried out at atmospheric pressure, such as consumption of the more quantity of process gas, lower control over the reaction taking place, higher temperature because of high degree of ionization. For the formation of plasma discharges with electric or electromagnetic fields, different types of designs and power sources exist. this work is of two categories: i.) Glow discharge plasma and radiofrequency (RF) ii) inductively coupled plasma (ICP).

1.12 PLASMA MODIFICATION OF NATURAL FIBERS

The process of plasma treatment with natural fiber surfaces is complicated, where many chemical reactions along with physical effects are happened concurrently. Some of the processes regarding the plasma modification of natural fibers [10,11] are discussed as follows

a) Cleaning - by splatter or etching of the surface impurities; Carrier gas is non polymerizing such as air, helium.

b) Exclusion - removal of fiber material by physical or chemical means, like etching, sputtering, etc. resulting in rougher surfaces, which enhance good mechanical bonding with the matrix; Carrier gas is non polymerizing such as air, helium.

c) Plasma polymerization – development of a surface coating due to the reactions of the gases those are used and having a tendency for polymerization (fluorocarbon, hydrocarbon etc.)

d) Surface chemical modification – Amalgamation of the functional groups on the fiber surface itself, generally replacing atoms or fragments of the fiber detached by the plasma. This is because of direct reaction or by development of free radicals followed by reaction with species in the plasma (neutrals or ions)

Because of surface treatments of fibers, a better adhesion bonding among fiber and matrix is possible, result in improved mechanical properties of the composites [12] Many researchers have reported the improvement in the tensile strength, elastic modulus, flexural and compression strengths of the composites

In a research work [13] the ramie fibers were treated in a helium atmospheric pressure plasma atmosphere, some of the fibers were pre- treated in ethanol for a time period of 10 minutes. Then the composites were fabricated taking polypropylene as the matrix material. It was found that the composite that comprises with only plasma treated fibers shows only a 4% enhancement in interfacial shear strength , because of the rough surface developed on fiber surface resulting from the plasma treatment, while composites that comprises with fibers i.e. pre-treated with ethanol and then treated with plasma, had a 50% enhancement in IFSS .This is due to the modified hydrophobicity of the fibers, having an presence of a better concentration of C– C bond and the fiber contact angle increases 40% higher than the previous case.The wax and oils are get excluded from the fiber surface, because of the plasma treatment. The erosion

i.e. produced on the surface of fiber, result in open pores facilitates the water absorption by the cellulosic fibers. The water absorption is more in case of plasma treated fiber than that of untreated one. Plasma treatment produces changes at the fiber surface only, increasing the activation energy as compared to untreated fibers, thus improving matrix/filler adhesion [14]

Natural fiber-based composite is now emerging because of environmental concerns. Many industries replace synthetic fiber composite with natural fiber composite due to their superior qualities like biodegradable, recyclable, acceptable specific strength, ease of availability, low density, low cost, and easy processing. Surface modification is required to enhance its properties. Thus, natural fiber-based composites are being used in the automotive, construction,packaging, sports, and toy industry.

The surface modification by using plasma technology is found eco-friendly. By modifying the surface of fiber the mechanical properties of the fibers get enhanced as found by many researchers. The interfacial bonding among the matrix and fibers becomes stronger by combined operations of specific chemical treatment and plasma operation. The advantage of Plasma treatment is, there is changes at the fiber surface and improvement in the activation energy as compared to untreated fibers, thus improving matrix/filler adhesion.

2. Material and Methods

2.1 Fiber and Fiber Extraction

The stems of BV were collected from the forests of Sambalpur, Odisha, India as shown in Figure 1.The fibers from BV stem were extracted through the following procedures. First the outer bark wasremoved manually by scalpel after beaten with hammer for a time period of 10 minutes. Then after removal of the bark, the fibers were extracted from the rest part of the stem manually as shown in Figure 2. It was observed that the weight of outer bark was found 15% to 20% of the total weight of the stem whereas the weight of extracted fibers was found 80% to 85% of total weight of the stem. Then the fibers were thoroughly washed with distilled water to remove any impurities and dried in sun to remove moisture from the fiber. After that, the dried fibers were cut into small pieces by scissors and finally gone through various surface modification for further use.

FIGURE 1 Collected *Bauhinia Vahlii* stem [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 2 Extracted fiber from *Bauhinia Vahlii* stem [Color figure can be viewed at wileyonlinelibrary.com]

2.2 Preparation of BV Fibers:

The BV stem fibers are done by tearing the strips of outer bark and chopping the inner bark into minor pieces about 1mm wide and 3-4 mm long.

The crude filaments are first completely eroded with refined water to expel dirt and any outside issues adhering to the surface of the fiber. The filaments were put inside a hot air stove at 60 ºC for 24 hours to evacuate the dampness totally. The subsequent filaments were set apart as artificially untreated strands whose substance piece is specified in Table. The dried filaments are exposed to different apparent changes by various synthetic concoctions like potassium permanganate, benzoyl peroxide, sodium hydroxide maleic anhydride, and acrylic corrosive distinctly in command to increase the interfacial adhesive stuffs of fibers with different mediums. The constituents of BV fiber is as follows

Constituents	Composition (%)
Cellulose	54.00
Hemi-cellulose	18.20
Lignin	1.35
Moisture	1.35
Pectin	1.35
Ash	1.20
Wax	1.00

Table 4.1

2.3 Composite Fabrication

A hand layup technique was followed by compression moulding was adopted for composite fabrication. Surface modified chopped BV fibers were impregnated with epoxy resin in a mould of dimension 100m*100mm*3mm for fabricating the composite. The different class of fabricated composites are presented in table below:

Composite	Composition
Virgin Polypropylene	Polypropylene test specimen
Composite 1	Polypropylene (90%) + BV fibers (10%)
Composite 2	Polypropylene (80%) + BV fibers (20%)
Composite 3	Polypropylene (70%) + BV fibers (30%)

Table 4.2- Composition table

2.3.1 MECHANICAL TESTSTensile Test

The tensile test is generally performed on flat specimen. Tensile test of composite sample is carried out in ASTM 638B test standard. The geometry of the specimen was 150x 15 x 3mm. A uniaxial load was applied through both the end in this test. The results have also been presented for the purpose of determining the optimum weight percentages of the composites in order to particularize the work.

Impact Test

The impact strengths of composites were determined by pendulum type Izod Impact testing machine as per ASTM D 256 standard. The samples size was 60 x 15 x 3mm. During the test, specimens were clamped vertically as a cantilever beam and then struck by a single swing of the pendulum released from a fixed distance from the clamp. The hammer velocity and hammer weight were 3.46 m/s and 0.905 kg respectively. At each fiber loading total of five tests were conducted for both the composites and the average of the readings were recorded for result analysis.

Hardness Test

The hardness tests were performed on the different samples for its Rockwell hardness. The specimens were prepared according to ASTM D2240. A steel ball indenter was used for the test on Rockwell hardness tester at ITER material testing lab.

Flexural test

ASTM D7264 outlines testing of flexural properties of polymer matrix composites using a bar of rectangular cross section supported on a beam and deflected at a constant rate. All the composite samples were tested according to the standard with rectangular size samples of dimension 127*15*3mm

3. Results:

MECHANICAL CHARACTERISTICS OF COMPOSITE

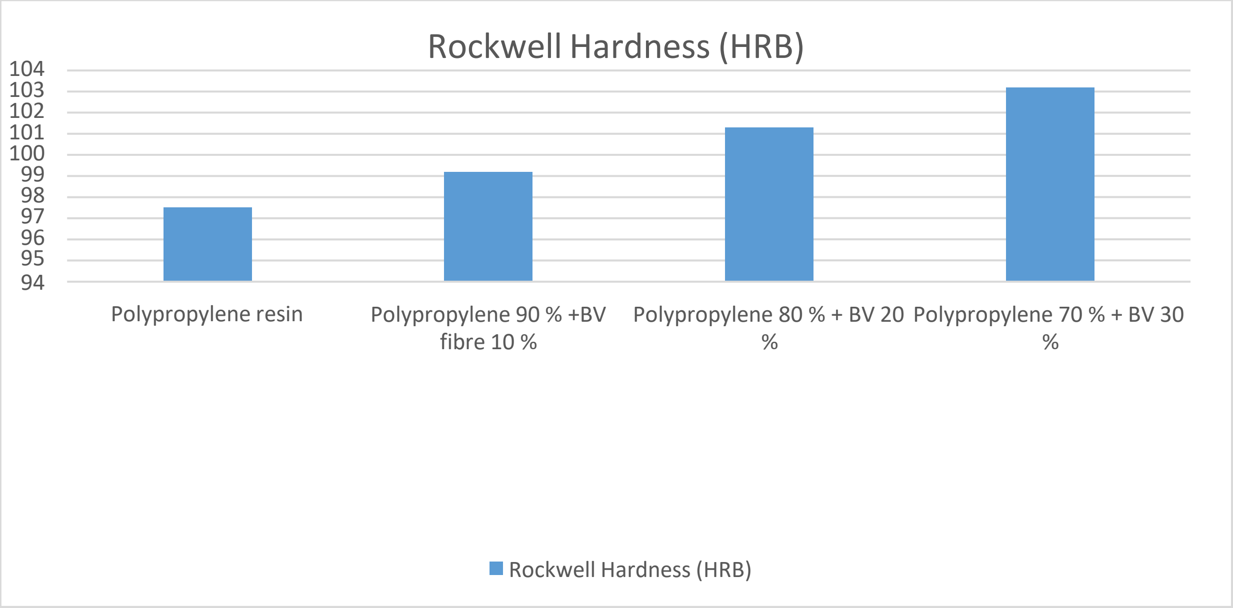
3.1 EFFECT OF FIBER LOADING ON HARDNESS OF COMPOSITE

The variation in hardness of BV and glass fiber composites with different fiber loading are being shownin table 3.1.

It has been observed that with the increase in fiber loading, the Rockwell hardness values significantly go on increasing up to 20 wt.% natural fiber content, however further increase in fiber loading the value decreases irrespective of fiber orientation. Composites with 20 wt.% of fiber loading with natural fibers show maximum hardness value.

Samples	Rockwell Hardness (HRB)
Polypropylene Resin	97.5
Polypropylene 90 % +BV Fiber 10 %	99.2
Polypropylene 80 % + BV Fiber 20 %	101.3
Polypropylene 70 % + BV Fiber 30 %	103.2

Table 3.1



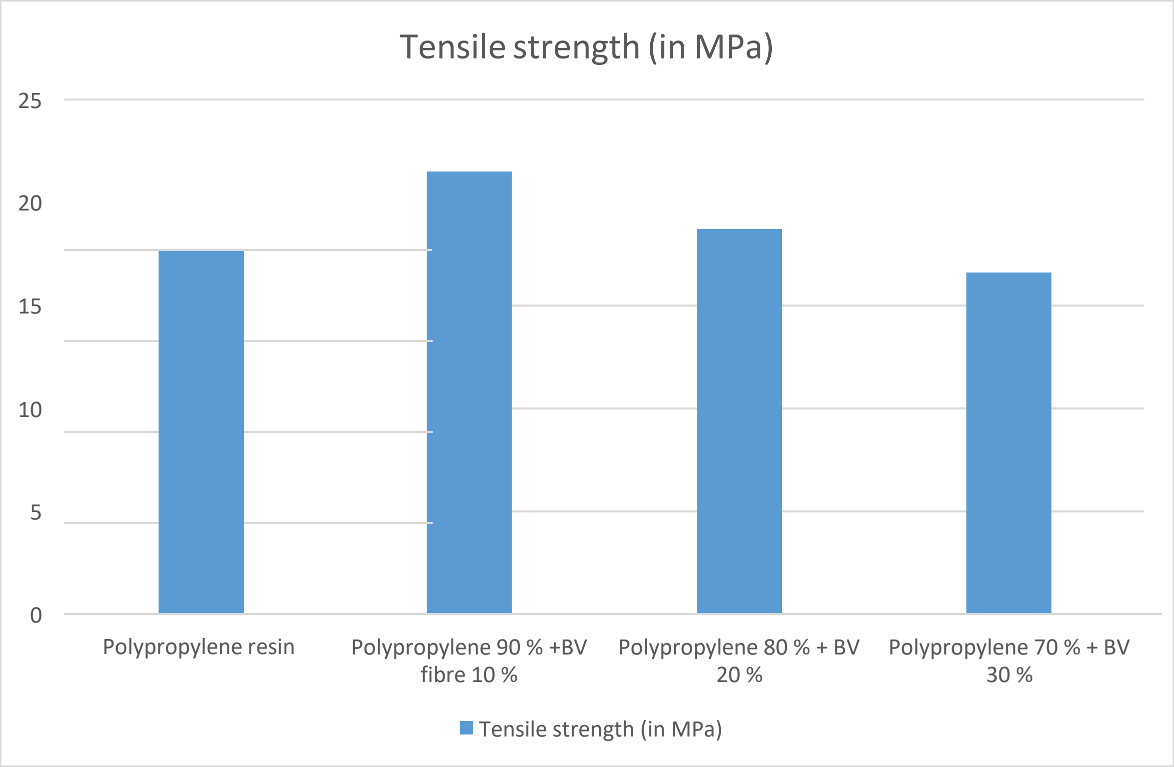
Graph 3.1- Table showing effect of fiber loading on hardness of natural FRPComposites

3.2 EFFECT OF FIBER LOADING ON TENSILE PROPERTIES OF COMPOSITES

The influence of fiber loading on the tensile strength and tensile modulus of the composites is shown in table 3.3 respectively. It is evident from the Figure 5.3; the tensile strength of the composites increases with increase in fiber loading. The maximum tensile strength is observed for composite with 20 wt.% fiber loading. The possible reason of increase in tensile strength and modulus may be the proper adhesion between fiber and matrix in case of composite with 20% fiber contents. The BV fibers reinforced polypropylene composite showed poor tensile strength because of low interaction between fiber and matrix.

Samples	Tensile strength (in MPa)
Polypropylene Resin	20
Polypropylene 90 % +BV Fiber 10 %	21.5
Polypropylene 80 % + BV Fiber 20 %	18.7
Polypropylene 70 % + BV Fiber 30 %	16.6

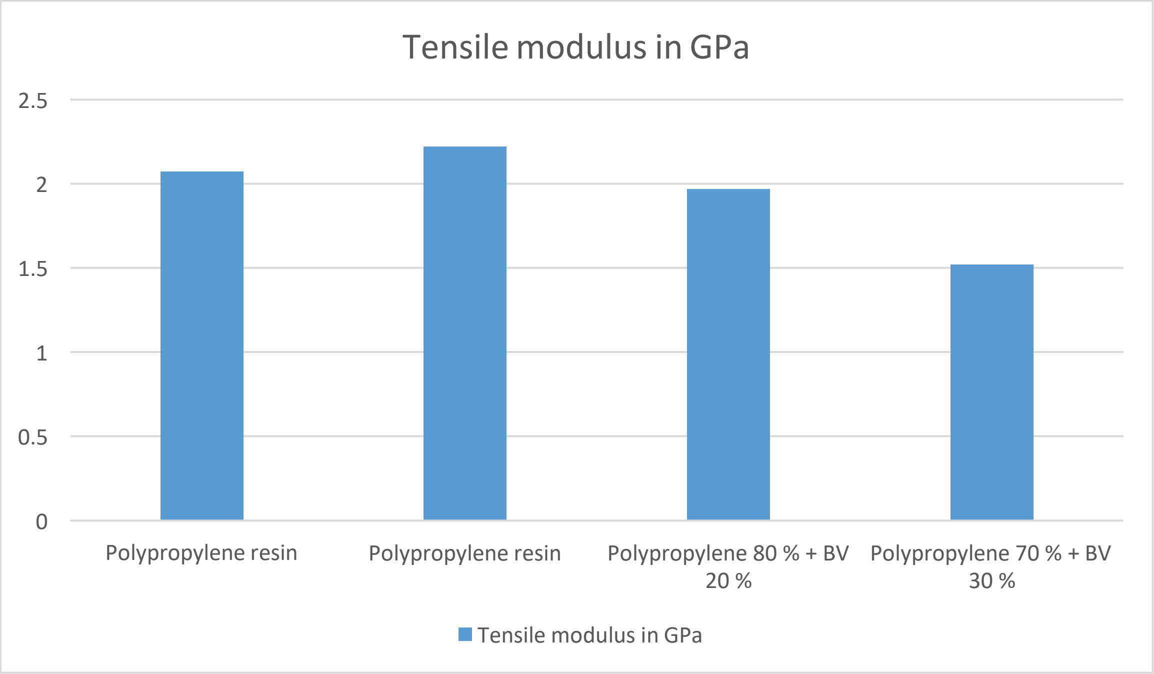
Table 3.2



Graph 5.2

Samples	Tensile Modulus in GPa
Polypropylene Resin	2.07
Polypropylene 90 % +BV Fiber 10 %	2.22
Polypropylene 80 % + BV Fiber 20 %	1.97
Polypropylene 70 % + BV Fiber 30 %	1.52

Table 3.3



Graph 3.3

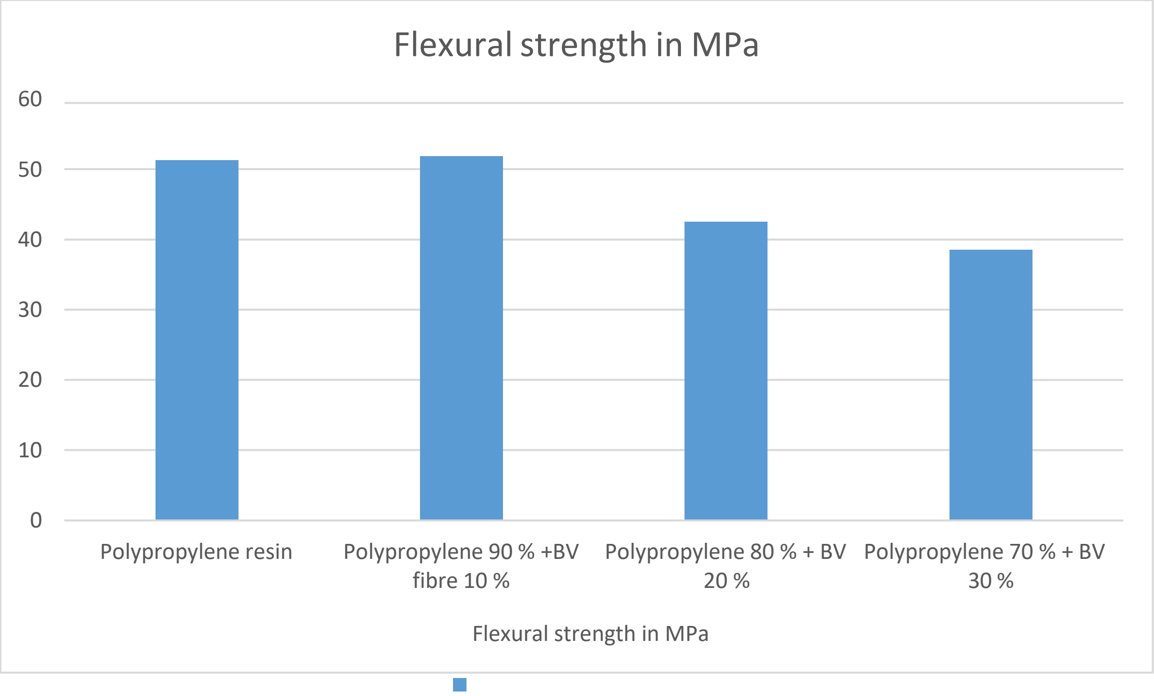
Graph 5.3- Table showing effect of fiber loading on tensile strength and tensilemodulus of composite

3.3 Effect of fiber loading on Flexural strength and Flexural modulus of composites

Flexural modulus of BV fibers in PP composites is shown in Figure 3.5. Flexural modulus of composites increases acrylic acid treated BV is added into composites. The treated composites show increment of flexural modulus at higher fiber loading. Due to more compatibility between less hydrophilic fiber with matrix that present in the continuous interfacial, better and efficient stress transfer in composites is achieved and thus increases the flexural modulus of composites

Samples	Flexural strength in MPa
Polypropylene Resin	51.25
Polypropylene 90 % +BV Fiber 10 %	51.86
Polypropylene 80 % + BV Fiber 20 %	42.56
Polypropylene 70 % + BV Fiber 30 %	38.54

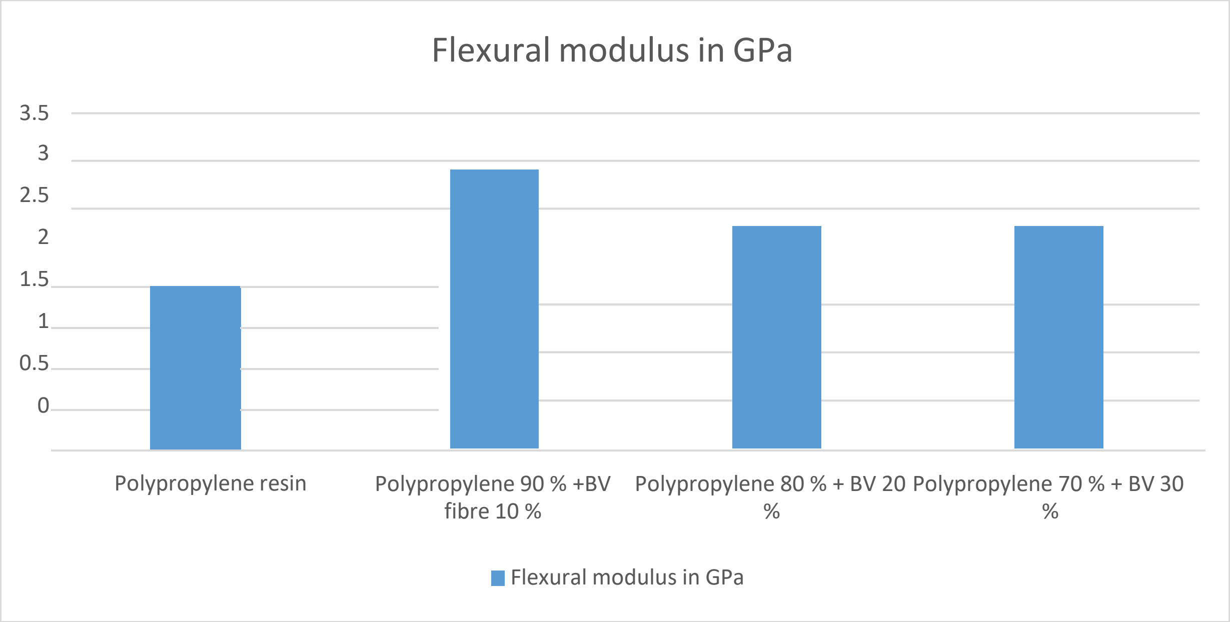
Table 3.4



Graph 3.4

Samples	Flexural modulus in GPa
Polypropylene Resin	2.09
Polypropylene 90 % +BV Fiber 10 %	2.91
Polypropylene 80 % + BV Fiber 20 %	2.32
Polypropylene 70 % + BV Fiber 30 %	2.32

Table 3.5



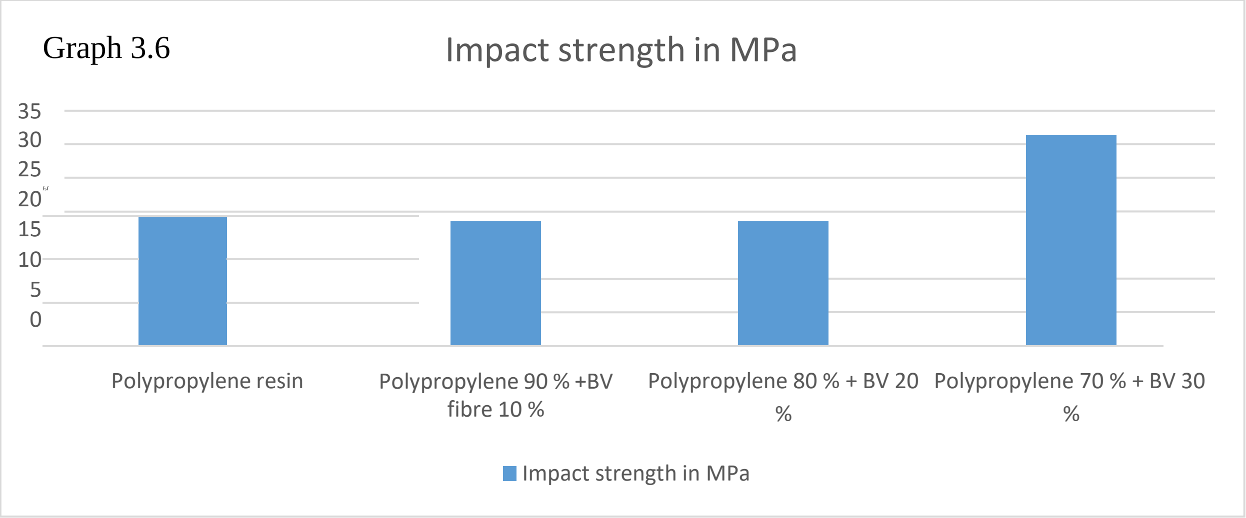
Graph 3.5

3.4 Effect of fiber loading on Impact strength of properties

Two types of composite are prepared. One class of composite content 80% Polypropylene and 20% glass fiber. Another class of composite is fabricated taking Polypropylene as matrix material and Bauhinia vahlii same fiber as reinforcement.

Samples	Impact strength in MPa
Polypropylene resin	14.38
Polypropylene 90 % +BV fiber 10 %	18.61
Polypropylene 80 % + BV 20 %	18.61
Polypropylene 70 % + BV 30 %	31.43

Table 3.6


Graph 3.6

EFFECT OF FIBER LOADING ON FLEXURAL STRENGTH OF PROPERTIES

3.5 Thermogravimetric Analysis (TGA)

The TGA curves of treated BV fibers, composite and virgin PP sample are shown in Figure 5. It was found that the thermal degradation of natural fibers occurs in two major steps. The curves demonstrate that the weight of plasma treated BV fiber reduces rapidly in temperature range of 50°C- 150°C in comparison to PP and BV fiber reinforced composite. This is due to the moisture evaporation from the fiber. In case of the composite (20%), the composite was found tound to be stable upto 380-400°C after which the weight loss was found to be 80%.. The temperature range around 180°C-350°C corresponds to depolymerization of hemicellulose, pectin and the degradationof lignin (Mabrouk et al., 2017).

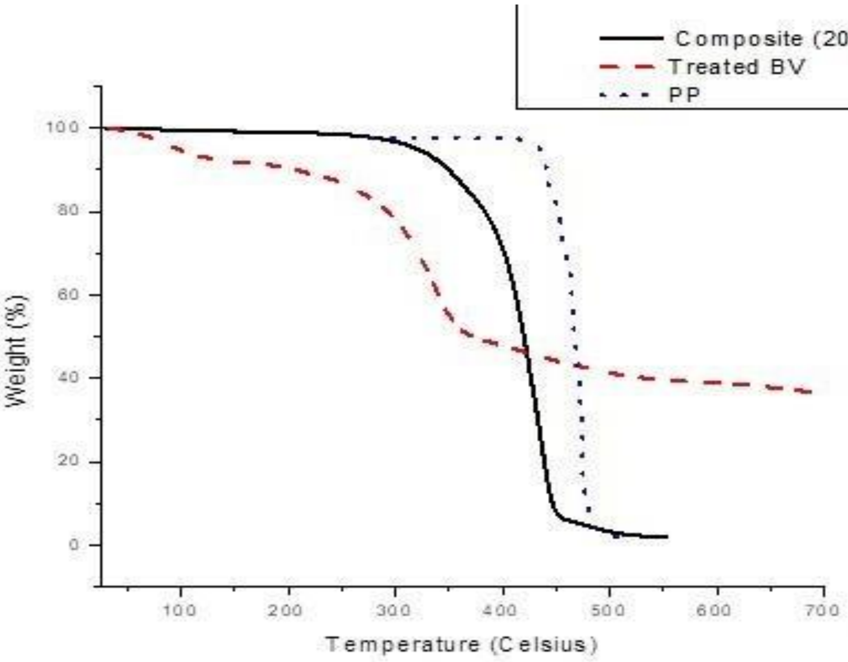


Fig 5- TGA of Composite and Virgin PP

4. Conclusions:

The test concentrate on the impact of fiber stacking on physical and mechanical conduct of Bauhinia vahlii stem fiber fortified polypropylene-based composites prompts the accompanying ends:

Various weight percentages (e.g. 10% and 20%) of Maleic anhydride treated short BV fibers and bidirectional glass fibers have been reinforced with Polypropylene resin matrix to fabricate composite materials by hand lay-up method. Several surface modifications such as alkaline, benzoylation, permanganate, peroxide and maleic anhydride have been given to BV Stem fibers to reduce the hydrophilicity of fibers and to increase the interfacial adhesion between the fiber and matrix. Out of these, maleic anhydride treated fiber is found to be the best one which gives best results which has been later used for composite fabrication. Different fiber loading samples were fabricated and tested and it was concluded that tensile strength, hardness and impact strength increases with increase in fiber concentration up to 20%.

Fig-4.1 Photo of sheets (10%)

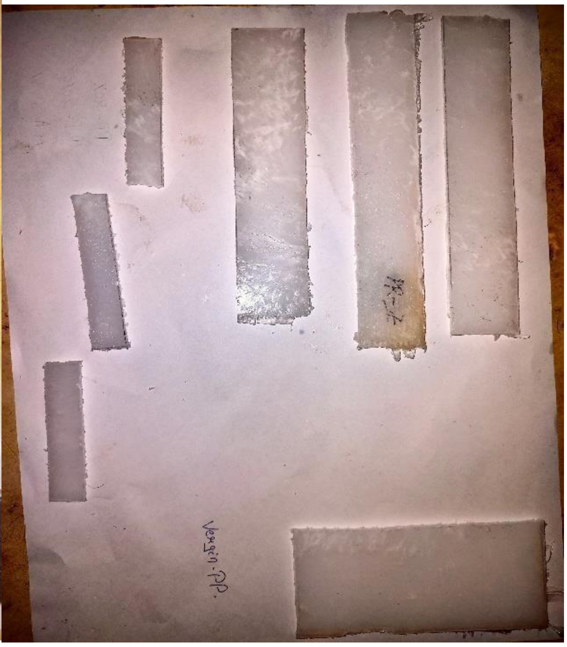


Fig-4.2 Photo of sheets virgin PP

In this present work it is found that the composite with 10% BV fiber composition shows good mechanical and flexural properties, whereas by increasing the fiber content in the composite the impact strength found to increase and at 30% fiber content, the composite exhibits the highest impact strength among all the composites.

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