Final Report of Major Research Project

SYNTHESIS AND CHARACTERIZATION OF NANO BANANA FIBRE REINFORCED POLYMER NANO COMPOSITES

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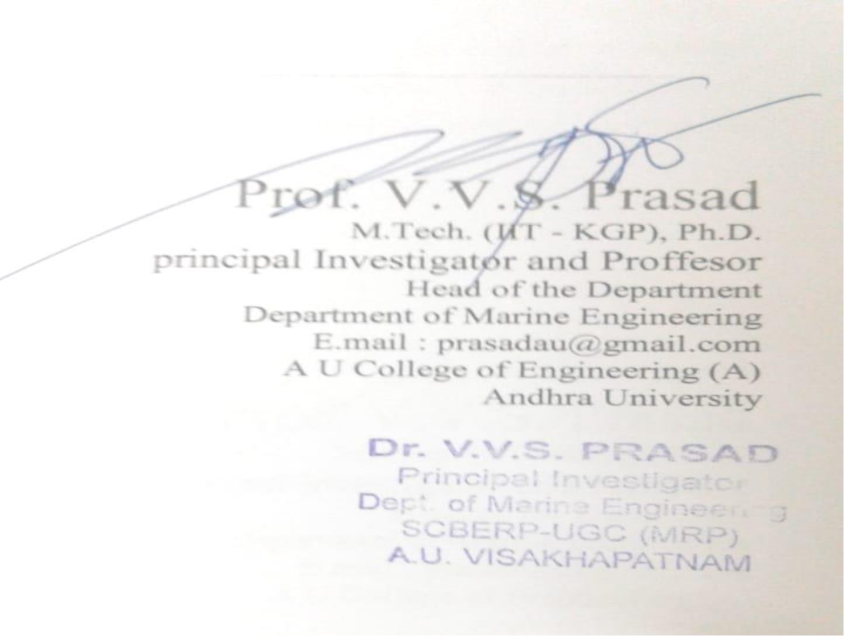

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ABSTRACT

Polymer nanocomposites are one of the important application areas of nanotechnology, as well as naturally derived organic nano phase materials of special interest. Recent years has seen the uses of eco-friendly composites due to its light weight and moderate strength. The potential of nanocomposites in various sections of research and application is promising and attracting increasing investments. The present investigation deals with the synthesis and characterization of banana nanofibers reinforced polymer composites. In this work, nanofibers are extracted from the stem of banana tree and undergoes chemical treatment and mechanical milling process. High energy ball milling is used for preparation of nanofibers to the required dimensions.

The most important factor in finding good fiber reinforcement in the composites is the strength of adhesion between matrix polymer and fiber. Due to the presence of hydroxyl groups and other polar groups in various constituents of banana, the moisture absorption is high, which leads to poor wettability and weak interfacial bonding between fibers and the more hydrophobic matrices. Therefore, it is necessary to impart a hydrophobic nature of the fibers by suitable chemical treatments in order to develop composites with better properties.

The characterization of nanofibers is done using a Fourier Transform Infrared Spectroscopy Analysis, X-ray diffraction and Scanning Electronic Microscope and found its average particle size to be in the range of 71micrometer to 47nm. The banana nanofibers have a high potential to be used in many different areas, particularly as reinforcement in the development of nanocomposites. In the present work, banana nanofibers are reinforced in epoxy polymer in different weight percentages to fabricate nanocomposites using hand lay-up technique. The influence of banana nanofibers reinforcing effect with an epoxy polymer on mechanical properties like tensile strength, impact strength, flexural strength, hardness and damping property of nanocomposites are investigated and ASTM standards are followed for preparing the samples. It is observed that the mechanical properties have improved when compared with that of neat composites. The maximum improvement is observed when the weight percentage of the banana nanofiber in the polymer is 4 wt.% and 6 wt.%. This is due to good dispersion of banana nanofibers in composites

which reduce the stress concentration and enhances the uniformity of stress distribution.

Environmentally beneficial composites can be made by replacing synthetic fibers with various types of cellulosic fibers. In the present work, the effect of alkali treatment on the moisture absorption is investigated. In the present work, it is observed that the alkali treated banana fiber absorbs less moisture than compared to other natural fibers.

Thermal properties of banana nanofibers composites are investigated using thermo gravemetric analysis (TGA), differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA). The influence of reinforcement of nanofibers in terms of glass transition temperature, thermal stability and thermal decomposition is discussed from TGA and DSC. The crystallization rate and crystallization peaks shift lower in the thermogram with the increasing nanofibers content. The nanofibers composite plays a vital role in controlling its rate of thermal degradation. Dynamic mechanical analysis results revealed the storage modulus of the nanocomposites increased as compared to neat composites where as the mechanical loss factor (Tan δ) decreased. This is due to the fact that the molecular motions become more restricted due to cross linkings and formation of stronger interracial bonds, resulting in the decrease in energy that can be dissipated throughout the sample.

Finally, regression analysis is carried out in order to mitigate the effect of error in the experimental data. The data set is interpolated to other additional data points which are then used to develop regression models for all the output parameters. The models are used to predict mechanical and thermal properties of nanocomposites. The models are found to be capable of predicting the above parameters with fair accuracy.

CHAPTER-1

INTRODUCTION

1.0 COMPOSITE MATERIALS - OVERVIEW

The high rate of depletion and the need for better materials have stimulated the search for newer materials compatible technically and with the environment. The waste disposal problems and criteria for cleaner and safer environment, have directed a great part of the scientific research into eco-composite materials that can be easily degraded or bio-assimilated. The natural fibers are abundantly accessible agro-waste is responsible for the new interest in research in sustainable technology. Agricultural related bioresources have received much attention due to their potential key components of biocomposites.

The possibilities of using all the components of the fiber crop provide wide ranging opportunities both in up and downstream processing for developing new applications. Agricultural crop residues such as cereal straw, corn stalk, cotton, bagasse and grass, which are produced in billions of tonnes around the world, represent an abundant, inexpensive and readily available source of lignocellulosic biomass. The exploration of these inexpensive agricultural residues as a bio resource for making industrial products will open new avenues for the utilisation of agricultural residues by reducing the need for disposal and environmental deterioration through pollution, fire and pests and at the same time add value to the creation of a rural agricultural based economy. Composites are used not only for their structural properties but also for their electrical, thermal, tribological and environmental application. Modern composite materials are actually optimized to achieve a particular balance of properties for a given range of application, maritime craft, because wood is becoming increasingly scarce and expensive, timber is losing favour with many boat builders and owners because wooden boats are easily degraded by seawater and marine organisms and therefore requires ongoing maintenance and repairs that can be expensive. Most maritime craft are built using glass reinforced polyester composites; although sandwiched composites and advanced FRP materials containing carbon and aramid fibers with vinyl ester or epoxy resin matrices They are commonly used for high performance structural applications. Their use in boats and their growing replacement of metals in ground transport systems is an uprising in material usage which is still accelerating. It finds application in composite, Automotive, sport goods, medical equipment & packaging Industry.

Most of the natural fibers are lighter due to their favourable density in comparison with other synthetic fibers and metallic materials. This attribute in combination with their excellent mechanical properties are beneficial, where stronger and lighter materials are required, especially in transportation application where energy efficiency is influenced by the weight of the fast moving mass. The physical and chemical morphology of natural fibers, their cell wall growth, patterns and thickness, dimensions and shape of the cells, cross-sectional shapes, distinctiveness of lumens, etc., besides their chemical compositions, influence the properties of the fibers. These fibers will also provide important opportunities to improve people’s standard of living by helping generate additional employment, particularly in the rural sector. Accordingly, many countries that have these natural sources have started to conduct R&D efforts with lignocellulosic fibers, seeking to take advantage of their potential social advantages [1].

A composite is a material made up from two or more other materials which give properties, in combination, that are not available from any of the ingredients alone. Nature continues to be generous to mankind by providing all kinds of resources in abundance for his living and existence. In this era of technology products depend on new varieties of materials that have special characteristics. Metal composites, plastic and fiber reinforced polymer composites are playing a vital role in all fields of Engineering and Technology. The performance of machine components depends mainly on the material that it is made of in the fields of automobile, railways, aerospace, structural applications, etc., and the strength to weight ratio of the material play an important role. Due to the enhanced physical properties the use of fiber reinforced polymer composites is increasingly replacing many of the conventional materials [2]. Over the last decade, polymer composites reinforced with natural fibers have received great attention, both from the academic world and from various industries. Nowadays, the use of natural fibers, especially in the automotive industry has become a rather common practice. Successfully implemented examples include both natural fiber-thermoset and thermoplastic composites for interior applications such as door panels, trim parts, seating and parcel shelves. The most important types of natural fibers used in composite materials are flax, hemp, banana, kenaf, and sisal due to their mechanical properties and ease of availability. Carbon materials (including composite materials containing carbon) are well-known for their structural applications, which relate to aerospace structures, aircraft brakes, concrete structures and lubrication. They are also known for their thermal applications relating to heat conduction and thermal insulation, environmental applications involving activated carbon and biomedical applications which pertain to implants. Less well-known are the electrical applications, though the electronic properties have long been studied for the purpose of fundamental understanding of the physics of carbons. These applications include electrical conduction, electrodes, electromagnetic reflection, heating, thermal conduction, thermoelectricity, sensing, electrical switching and electronic devices.

Nanotechnology is one of the most popular areas in current research and development in all technical disciplines. It shows great promise for providing many breakthroughs in the near future. This will change the direction of technological advances in a wide range of applications. It can be defined as the science and engineering involved in the design, synthesis, characterization, and application of materials and devices whose smallest functional organization in at least one dimension is on the nanometer scale or one billionth of a meter. On these scales, consideration of individual molecules and interacting groups of molecules in relation to the bulk macroscopic properties of the material or device becomes important since it is control over the fundamental molecular structure that allows control over the macroscopic chemical and physical properties [3-5]. Nanotechnology is also defined as the manipulation of materials, measuring 100 nm or less in at least one dimension where the physical, chemical, and biological properties are fundamentally different from those of the bulk material.

A nanometer is a billionth of a meter, or 80,000 times thinner than human hair. So, the nanometer domain covers sizes that are bigger than several atoms, but smaller than the wavelength of visible light. An increasing interest in the scientific community to work with materials on the nanometer scale has been observed since the introduction of the concept of nanotechnology by Richard Feynman in 1959 at a meeting of the american chemical society. However, nanocomposite materials have been widely studied only since the past twenty years So far, researchers have been working on the development of anisotropic natural fiber reinforced polymer (NFRP) composites. However, to improve the mechanical properties of NFRPs in anisotropic nature, it is required to explore new materials for reinforcement. With the emergence of nanotechnology, most of the materials of improvised products rely on changes in the physical properties when the feature sizes are shank.

Nanofibers take advantage of their dramatically increased high surface-to-volume and aspect ratios and this makes them ideal for use in polymeric materials. Such structures combine the best properties of each component to possess enhanced mechanical and superconducting properties for advanced applications. The high aspect ratio and nanofibers are the main characteristics of the cellulose fibers. For this reason the interface area offered by the cellulose surface is high. This might lead to the formation of an interphase in which mechanical properties of the matrix are modified like the polymer matrix reinforced with cellulose nanofibers. The polymer nanocomposites can be used as lightweight replacements for metals. Through nanotechnology the improvised materials enable a weight reduction accompanied by an increase in thermal stability and enhanced functionality. Any work in the direction of using nanofibers from natural fibers as reinforcement materials in polymers may bring about changes in the manufacturing scenario, since these fibers are biodegradable and eco-friendly. There is tremendous concern for using plant nanofibers like cellulose micro fibrils or whiskers to be applied in the new era of biodegradable composites for structural and non- structural applications.

1.1 NANOTECHNOLOGY IN COMPOSITE MATERIALS

Nanotechnology is the major driving factor for growth at every level of economy. At the 1 nanometer (nm) scales and below, quantum mechanics rules, and at dimension above 100 nm, classical quantum mechanics, physics and chemistry dictate properties of matter. Between 1 and 100 nm a hybrid exists and interesting things can happen such as mechanical, optical, electrical, magnetic, and a variety of other properties can behave quite differently. The development of polymer nanocomposites is rapidly emerging as a multi disciplinary research activity whose results could broaden the applications of polymers to the great benefit of many industries [6]. Polymer nanocomposites are produced by incorporating materials that have one or more dimensions on the nanometer scale (<100nm) into a polymer matrix. The nanomaterials are in literature are referred to as nanofillers, nanoparticles, nanoscale building blocks or nano reinforcements. Nanocomposites have improved stiffness, strength, toughness, thermal stability, barrier properties and flame retardancy compared to pure polymer matrix. Nano reinforcements are also unique in that they will not affect the clarity of the polymer matrix.

Polymer nanocomposites contain substantially less filler and thus enable grater retention of the inherent processibility and toughness of the neat resin. Only a percentage of these nanomaterials are normally incorporated into polymer and the improvement is vast due to their large degree of surface area [7-8]. The properties of nanocomposite materials depend not only on the properties of their individual parents but also on their morphology and interfacial characteristics. In the particular case of polymers reinforced with rigid nanofillers, various parameters seem to be of importance in characterizing the fillers. These may be geometrical factors such as the shape, the size aspect ratio, and intrinsic mechanical characteristics such as the modulus or the flexibility, surface properties of specific surface area and surface treatment [9].The type of polymer matrix used and the possible effects of nanofillers on its microstructure and its intrinsic properties also determine the composite properties.

1.1.1 Nano Particle

Nanoparticles are obtained from available natural resources and generally need to be treated because the physical mixture of a polymer and layered silicate may not form a nanocomposite. In this case a separation into discrete phases takes place [10]. The poor physical interaction between the organic and inorganic components leads to weak mechanical and thermal properties. In contrast, strong interaction between the polymer and the layered nanocomposites leads to the organic and inorganic phases being dispersed at the nanometer level [11]. As a result, nanocomposites exhibit unique higher properties than conventional composites.

1.1.2 Methods of Synthesis of Nano Particle

Nanoparticles may be created using several methods. Some of them may occur in nature as well. The methods of creation include attrition and pyrolysis. While some methods are bottoms up, some are called top down. Top down methods involve braking the larger materials into nanoparticles. Solids with nanosize particles cannot be prepared or treated by traditional methods simply because the reactants are not mixed on the atomic scale.

1.1.2.1 Hydrothermal Synthesis

A method to produce different chemical compounds and materials using closed system physical and chemical processes. Hydrothermal reactions are usually formed in closed vessels. The reactants are either dissolved or suspended in a known amount of water and are transferred to acid digestion reactors flowing in aqueous solutions at temperatures above 100°C and pressures above 1 atm.

1.1.2.2 Sol-gel Synthesis

Nanomaterials technology, assuming preparation of a sol with its subsequent transition into a gel, i.e. a colloidal system consisting of a liquid dispersion medium contained in a spatial grid formed by connected particles of the dispersed phase. Sol-gel synthesis is a very viable alternative method to produce nanocrystalline elemental, alloy and composite powders in an efficient and cost-effective manner. Nano crystalline powders can be consolidated at much lower pressures and temperatures.

1.1.2.3 Polymerized complex method

Wet chemical method using polymeric precursor based on the Pechini process has been employed to prepare a wide variety of ceramic oxides. The process offers several advantages for processing ceramic powders such as direct and precise control of stoichiometry, uniform mixing of multi components on a molecular scale, and homogeneity.

1.1.2.4 Chemical vapor deposition

Chemical Vapor deposition (CVD) is defined as the position of a solid on a heated surface from a chemical reaction in the vapor phase. It is a versatile process suitable for the manufacturing of coatings, powders, fibers and monolithic components.

1.1.2.5 Microwave synthesis

This is the process of heating and sintering of ceramics by microwaves. The use of microwave processing spans a number of fields from food processing to medical applications and chemical applications. Major areas of research in microwave processing for ceramics include microwave interaction, dielectric characterization, microwave equipment design, new material development, sintering, joining and modeling. A microwave chemical decomposition unit is used for the fabrication of carbon nano tubes and coils.

1.1.2.6 High energy ball milling process

Ball milling has been used in various industries to perform size reduction for a long time. Recently, materials with novel microstructures and properties have been synthesized successfully via high energy ball milling processes. Although different terms have been used to describe the high energy ball milling process, the three terms that are generally used to distinguish powder particle behavior during mechanical milling. There are some inherent advantages in processing nanomaterials via high-energy ball techniques such as excellent versatility, scalability and cost effectiveness. Therefore, high-energy ball milling techniques are well suited for manufacturing large quantities of nanomaterials.

1.2 Advantages of nanocomposites

The advantages of nanocomposites are significantly related to the possibility of designing and creating new materials and structures with unprecedented flexibility and physical properties. Secondly, nanoscale fillers are almost free of defects and their application in the composite area opens a window of opportunity to overcome the limitations of traditional micrometer scale. Finally, due to the high specific surface area, nanocomposites present a large volume of interfacial matrix material (interphase) with properties different from those of the bulk polymer. A uniform dispersion of nanoparticles leads to a very large matrix-filler interfacial area, change in the molecular mobility, related behavior and ensuing thermal and mechanical properties [12 and 13]. The major advantages of nanocomposites over traditional polymeric composites are:

- Dramatic improvement of mechanical properties
- Increased stiffness without loss of flexibility
- Increased dimensional stability
- Greater chemical and thermal stability
- High scratch resistance
- Very good transparency due to decreased scattering
- Relatively better biodegradation
- Easy processing and recycling due to avoidance of cumbersome processing techniques and low filler loading.

1.3 CHARACTERIZATION OF NANOCOMPOSITE MATERIALS

Characterization of nanocomposites can be performed using different techniques allows a qualitative understanding of the internal structures, spatial distribution of the various phases and direct visualization of defect structure. Scanning electron microscope can be used for structure and morphology determination of nanocomposites. X-ray diffraction has been widely used for the determination of crystallities and crystallinity index. Thermal properties are very important for nanocomposites. Differential scanning calorimetry to understand the nature of crystallization taking place in the matrix. Thermogravmetric analyzer provides information regarding polymerization reactions and the thermal stability of the nanocomposites. Dynamic mechanical analysis tests can also be used to evaluate nanocomposites performance under various conditions of temperature and relative humidity.

1.4 BANANA FIBER REINFORCED POLYMER NANOCOMPOSITE

A nanocomposite material has significantly broadened in the last few years. This term now encompasses a large variety of systems combining one-two and three dimensional materials with amorphous materials mixed at the nanometer scale. Natural fibers are pervasive throughout the world in plants such as flax, sisal, banana, hemp, banana, wood, grasses etc. Among the all natural fibers banana fibers are easily available in fabric and fiber forms with good mechanical and thermal properties. Banana fibers are eco-friendly, low cost and low density fibers with high specific properties. Therefore banana based composite materials can be used in industrial, automobile, structural and aerospace applications.

Banana fiber is hydrophilic in nature which causes poor wettability with hydrophobic organic matrix resins like polyester when preparing composites. Hydrophilicity nature of banana fiber is reduced by chemical modification like alkalization, bleaching etc. These treatments not only decrease the water absorption capacity of the fiber but also increase the wettability of the fiber with resin and improved inter bond between fiber and matrix. The main elements of banana fiber are cellulose, lignin, hemicelluloses and pectin. The use of banana cellulose fibers derived from annually renewable resources as a reinforcing phase in polymer matrix composites provides positive environmental benefits with respect to ultimate disposability and raw material. The main advantage of banana cellulose fibers are

- Renewable nature
- Wide variety of fillers available throughout the world.
- Non-food agricultural based economy
- Low energy consumption
- Low cost
- Low density
- High specific strength and modulus
- High sound alternation of cellulosic base composites
- The recycling by combustion of cellulose filled composites is easier in comparison with inorganic filler systems.

The possibility of using banana cellulose fibers as a reinforcing phase has received considerable interest. In addition the intrinsic nanoscale properties of banana fiber cellulose material for developing advanced nanomaterials and composites.

1.4.1 Need of banana fiber nanocomposite

Cellulose fiber is the most abundant in nature biodegradable and relatively cheap, and is a promising nano-scale reinforcement material for polymers. The combination of biodegradable cellulose and biodegradable renewable polymers is a particularly attractive from an environmental point of view. The concept of nano structured material design is gaining widespread importance among the scientific community. The strong reinforcement effects at low volume fraction resulted in a tremendous interest from the industry and research circles. With this as an inspiration, the potential of nanoscale cellulose structures as reinforcement in novel composite material was extremely interesting. The concept of cellulose nanocomposites for load bearing applications is fairly new. The property enhancements are expected due to higher Young' modulus of pure cellulose reinforcement and finely distributed reinforcing microfibrils. Cellulose nanocomposites are usually fabricated by utilizing these microfibrils of 10-50 nm on width as reinforcement in polymer matrix.

The high strength and stiffness as well as the small dimensions of nanoscale cellulose may well as the dimensions of nanocellulose may well impart useful properties of composite materials reinforced with these fibers, which could subsequently be used in a wide range of applications. Cellulose nanofiber is obtained as stable aqueous suspensions and most investigation focused on hydrosoluble polymers. The main advantage is that the dispersion state of the nanofibers is kept when using an aqueous medium for the processing. After dissolution of the hydrosoluble polymer, the aqueous suspension of cellulose nanofiber. The ensuing mixture is generally cast and evaporated to obtain a solid nanocomposite. It can also freeze dried and hot pressed.

Natural banana fibers are sustainable and biodegradable with many advantages of low density, low cost, and high specific properties. They have been applied as reinforcement to eco-composites and bio-composites. The development of banana nanofiber composites based on nanocellulosic material is a rather new but rapidly evolving research area. The applications of nanofibers improve polymer mechanical properties such as tensile strength and modulus in a more efficient manner than is achieved in conventional micro and macro composites. The low thermal expansion of nanocellulose combined with high strength, high modulus and transparency make them a potential reinforcing material in roll-to-toll technologies.

1.4.2 Mechanical properties of banana fiber nanocomposite

Mechanical properties of nanocomposites provide a basis for determining the strength of a particular material for load bearing applications, designing a product and predicting its lifespan and performance characteristics. Therefore, selection of nanocomposite materials for a variety of applications is often based on mechanical performance such as tensile strength, elongation, impact strength, hardness and damping property. The level of interfacial adhesion is a fundamental importance in determining the transfer of stress in a composite. A strong and durable interface is required between the nanofiber and epoxy matrix for the resulting composite to have good mechanical properties.

The nano size reinforcement in composites increases modulus of elasticity and tensile strength. The high strength nanofiber cellulose together with its potential economic advantages will offer the opportunity to make lighter weight strong materials with greater durability. It is possible to extract a few trends for the behavior of polymer matrix nanocomposites based on the nature of the polymer matrix, particularly crystalline or amorphous nature of the polymer, and the interaction between the nanofiber and matrix. This interaction had larger impact in nanocomposites due to the large interfacial area between the filler particle and the matrix.

The mechanical properties of banana nanofiber may be used as reinforcing fibers in high toughness nanocomposite, where stiffness and low weight are important consideration. Nanocomposite offers a huge specific area and impressive mechanical properties compared to neat epoxy matrix. The macroscopic behavior of cellulose nanofibers based on nanocomposites depends as for as heterogeneous materials on the specific behavior of each phase, the composites (volume fraction of each phase) the morphology (spatial arrangement of the phases) and the interfacial properties. The mechanical properties of the banana nanofiber reinforced composites were tested by tensile strength, impact strength, and hardness and damping property.

1.4.3 Thermal properties of banana fiber nanocomposite

The thermal properties are critical for many applications, including the use of cellulose nanofiber for the production of nanocomposites. The banana nanofiber reinforced composites were determined from differential scanning calorimetry, thermal gravimetric analysis and dynamic mechanical analysis. DSC was used to characterize the melting point temperature, glass transition temperatures, and other material and material reaction characteristics such as specific heat, crystallinity, and reaction kinetics of all samples. In this study the melting and crystallization behavior of pure epoxy composite and banana nanofiber reinforced epoxy composites were studied. TGA technique is used to characterize polymer thermal stability, have been further employed for assessment of comparative thermal stability of banana nanofiber composites and prediction of material lifetime. It is used to investigate the thermal decomposition behavior of nanocomposites under nitrogen controlled atmosphere. The initial and final degradation temperature and corresponding percentage weight loss was recorded as a function of time. TGA curves used to analyze the thermal degradation temperatures of banana nanofiber composite.

DMA is a technique in which the elastic and viscous responses of a sample under oscillating load are monitored against temperature, time or frequency where the frequency of oscillation is proportional to the modulus (stiffness) of the material. DMA as a function of temperature including storage modulus (E'), which is a measurement of energy stored during deformation and related to solid-like or elastic portion of the elastomer, loss modulus (E''), which is a measurement of energy lost, usually as heat, during deformation and liquid like or viscous portion of the elastomer, and tangent delta (tan δ), which is related to material’s ability to dissipate energy in the form of heat. DMA was used here for developing a basic understanding of the interacting of different properties of epoxy resin with banana nanofiber composites in different weight percentages.

1.4.4 Banana fiber nanocomposite - statistical validation

Validation is one of the most important aspects of designing a Composite application. It is the monitoring agency’s responsibility to prevent, identify, correct, and define the consequences of monitoring difficulties that might affect the precision and accuracy and the validity of the measurements. In statistics, regression analysis is a statistical technique for estimating the relationships among variables. It includes many techniques for modeling and analyzing several variables, when the focus is on the relationship between a dependent variable and one or more independent variables. More specifically, regression analysis helps one understand how the typical value of the dependent variable changes when any one of the independent variables is varied, while the other independent variables are held fixed. Analytical or numerical models help to predict the properties of a material without conducting any experiments. However, these models have to be extensively validated with experimental data before adopting them in practice on a large scale. In order to compositions, models, one based on mathematical regression have been used to predict the banana nanofiber composites of different weight percentages.

1.5 PROBLEM DEFINITION AND SUMMARY

Polymer nanocomposites are an emerging class of new materials which have become an interdisciplinary field and one exciting research area is the isolation of nano cellulose from bio resources using the top-down technique. Polymer nanocomposites comprise a new class of materials where nano scale particles are finely dispersed within the resin. Nano particles take advantage of their dramatically increased surface area to volume ratio. Polymer composites can be reinforced by nano particles, resulting in novel materials which can be used as lightweight replacements for metals. Such nano technologically improved materials enable a weight reduction accompanied by an increase in stability and enhanced function. The direction of using nano fibers from natural fibers as reinforcing materials in plastics may bring changes in the manufacturing scenario since banana fibers are biodegradable and eco-friendly. These are being widely developed to meet the requirements of automotive, aerospace, structural, nonstructural and electrical applications. Nano fibers from natural fibers may evolve as an alternative to conventional nanoparticles in the future. One of the future processing possibilities of composites could be biomimetic processing approaches in view of their great potential in the development of new high performance materials with low environmental impact, it being environmentally friendly and energy efficient. In the case of development of diverse nanocomposites consisting of different combinations of nano materials (fibers or particles or platelets or tubes), and polymer matrices at various weight fractions or volume fractions, the self-assembly process, a simple approach belonging to biomimetic process of making nanocomposites, can be adapted. Even wood and other plant based fibers can also be considered as natural, but a complex and highly sophisticated composites, since cellulose microfibrils in these materials are embedded in lignin matrix self-assembly process.

These can provide some insight along with inspiration regarding the design and fabrication of light weight composite materials with unique properties. It is reported that new insights for the development of high performing materials such as fibrous composite materials ones and technical textiles is provided by systematic approach whereby biological structures, processes, and functionality could be comprehended thus providing a brilliant opportunity for transferring ideas whereby production processes comparable to those used by nature could be used for the development of such materials.

This chapter provides an exhaustive review of research works on various aspects of polymer composites and nanocomposites reported by pervious investigators. From the literature review, attempts have been made to produce nanofibers from banana fiber. The use of nanofibers as nanocomposites is not yet explored, though the fibers are biodegradable and eco-friendly with high specific strength and stiffness to produce isotropic composite. The work reported making use of nanofibers and its synthesis then there will be need to produce isotropic nanofiber composites and to study the water absorption, mechanical and thermal properties.

1.6 LAYOUT OF THE PROJECT

The present work involves the use of banana natural fibers to be used in different forms for the preparation of composite material. Banana natural fibers are mechanically synthesized and various characterization techniques are used for converting the raw material into a required form.

The work involves the following steps:

a) Synthesis of banana fibers by chemical treatment and mechanical milling into nanofibers.
b) Characterization of treated and untreated banana fibers is carried out by using fourier transform infrared spectroscopy analysis, X-ray diffraction, and scanning electron microscopy analysis. The chemical composition of the raw material is

analysed using using fourier transform infrared spectroscopy and the particle size of the nano powder obtained by synthesis process is found out using X-ray diffraction technique. The structure of the raw material is analysed using scanning electron microscopy.

c) Fabrication of banana nanoparticles reinforced polymer composites of neat composite composites (70 wt.%/30 wt.%) and with reinforcement of different weight percentage of banana nanofibers in the polymer resin (neat composite, 2, 4, 6 and 8 wt.%) composites. Testing of prepared banana nanocomposites specimens for mechanical, water absorption and thermal properties.
d) Preparation of nanocomposite specimens for testing mechanical properties like tensile strength, flexural strength, impact Strength, hardness and damping property which is tested as per ASTM standards.
e) Preparation and testing of banana nanocomposite samples for thermogravometric analysis, diffraction scanning calorimetric and Dynamic Mechanical Analysis.
f) Mathematical models formulated using regression and the results from the predicted models are compared with the experimental data and validation of the mechanical and thermal properties.

CHAPTER - 2

EXPERIMENTAL WORK

2.0 INTRODUCTION

One of the novel fields is the use of natural fibers in polymer reinforcement. Use of banana nanofibers is another new era due to the fact that the use of banana nanofibers in composite materials can exploit its high stiffness property. The production of nano scale banana fibers and their application in composite materials have gained increasing attention due to their high strength and stiffness combined with low weight, bio-degradability and renewability. This can be done by breaking down the hierarchical structure of the plant into individualized nanofibers of high crystallinity with a reduction of amorphous parts.

The Natural nanocomposites affect the structural characteristics of a composite structure in terms of mechanical properties and thermal Properties, when it is used as a matrix in the laminates of reinforcement of banana nanofibers. The fabrication of nanocomposites with banana nanofibers in a high aspect ratio for load bearing applications is relatively new. A high aspect ratio to the fibers is desirable as this enables a critical length for stress transfer from the matrix to the reinforcing phase. The aspect ratio of banana nanofibers is a major parameter that controls the nanofibers dispersion and e-glass fiber matrix adhesion that helps to give the optimally enhance the performance of polymer nanocomposite.

2.1 PREPARATION OF BANANA FIBER POLYMER NANOCOMPOSITES

The basic raw materials required to prepare nanofibers reinforced composites are banana nanofibers, epoxy resin, hardener and e-glass woven mat.

a. The banana fibers are synthesized by chemical treatment and mechanical milling process.
b. The matrix consisted of diglycidyl ether of bisphenol-A (DGEBA) a di-functional epoxy resin (LY-556) system and a tri-ethylene tetra amine (TETA) araldite hardener (HY-951) mixed in properties of 100:12.
c. Woven roving glass fiber mat of 310 gsm.

2.1.1 Banana fibers

Banana fiber is a natural based fiber. It has its own physical and chemical characteristics and many other properties that make it a fine quality fiber. Banana fiber had a very limited application and was primarily used for making items like ropes, mats, and some other composite materials. With the increasing environmental awareness and growing importance of eco-friendly fabrics, banana fiber has also been recognized for all its good qualities and now its application is increasing in other fields too such as polymer composites with natural fibers. Banana fibers are procured from M/S Lakshmi Group Pvt. Ltd., Mangaligiri, Andhra Pradesh. The banana fibers were shown in Fig. 2.1.

Fig.2.1 Raw banana fibers

2.1.2 Alkali treatment of banana fibers

Chemical treatment is carried out on the banana fiber in order to improve the interfacial bond between the fibers. It increases the amount of crystalline cellulose and removes impurities producing a rough surface topography. The banana fibers are cut into small length and weighed before an alkaline treatment is applied as shown in Fig. 2.2. The weighed banana fibers are soaked into 1% of sodium hydroxide solution at 80°C for 5 hours and continuously stirred. After the reaction was completed, the fibers are taken out and thoroughly washed several times with distilled water until the fibers become neutral. These fibers were dried in an oven at 80°C under vacuum till a constant weight was achieved. The percentage of lignin and impurities removed is measured as the difference in the weight loss of banana fibers before and after alkali treatment is repeated for 2%, 3%, 4% and 5% of NaOH Solution.

Fig.2.2 Submerged banana fibers in NaOH solution

2.1.3 Synthesis of banana fibers

For the past two decades, considerable efforts have been made to develop nanofibers with controlled shape, size and composition arrangement. Synthesis is one of the most effective strategies for attaining a high degree of control. The production of banana fibers into nano-scale elements requires intensive mechanical treatment. However, depending upon the raw material and the degree of processing, chemical treatments may be applied prior to mechanical fibrillation. The nanofibers from banana fibers were obtained by high energy ball milling process.

2.1.3.1 High energy ball milling

Banana fibers at the nano level are prepared using a high energy planetary ball mill. The planetary ball mill PM 100 pulverizes and mixes soft, medium-hard to extremely hard, brittle and fibrous materials. The PM 100 is a robust, compact floor model with one grinding station for grinding jars which can accommodate jars of nominal volume of 12 to 500 ml, dry as well as wet grinding can be carried out. The direction of movement of the sun wheel is opposite to that of the grinding jar in the ratio 1:2. The grinding balls in the jar are subjected to superimposed rotational movements, the coriolis forces. The difference in speeds between the balls and grinding jar produces an interaction between frictional and impact forces, which releases high dynamic energies. The interplay between these forces produces high and very effective degree of size reduction of the planetary ball mill shown. These are arranged on a rotating support disk, and a special drive mechanism causes to rotate it around its own axis.

The centrifugal force produced by the jars rotating around its own axis and that produced by the rotating support disk both acts on the vial contents, consisting of material to be ground and the grinding balls. As the jar is the supporting disk rotates in opposite directions, the centrifugal forces alternately actin like and opposite directions. This causes the grinding balls to run down the inside wall of the vial .The frictional effect, followed by the material being ground and grinding balls lifting off and travelling freely through the inner chamber of the vial and colliding against the opposing inside wall - the impact effect. Grinding jar and balls are available in eight different materials-agate, silicon nitride, sintered corundum, zirconia, chrome steel, Cr-Ni steel, tungsten carbide and plastic polyamide.

Table 2.1 Details of milling process

Milling Hours	Type of Balls	Rotational speed of Balls (RPM)	Dia. of the Balls (mm)	Ball to Fiber ratio
20 (20 Hrs)	Tungsten Carbide	200	10	10:1
40 (20 Hrs)				
60 (20 Hrs)				
80 (20 Hrs)			5	
100 (20 Hrs)				

The wet ball milling as a potential means to decrease the particle size of banana fiber with a minimum loss of crystallinity. As a result of high energy ball milling, the sizes of the banana fibers were decreased. Final particle size reduction with finer media with 10mm and 5mm diameter balls were used at a speed of 200RPM respectively, for 20 hours each as shown in Table 2.1. The particle size distribution of the original banana and the ground samples obtained were used to monitor the variations occurring in the size of the fibers. It is observed that the crystalline size of the banana fibers is reduced with increasing milling time; the size reduction goes into saturation after 100hours of milling it's the optimum milling time.

2.2 MORPHOGOLICAL STUDIES OF BANANA FIBER

Morphological studies of the banana fiber in terms of its polymer structure, molecular shape and the other properties are carried out using various equipments. The arrangement fixed by chemical bonding between adjacent monomeric units and between the atoms of individual monomeric units. Several tools can be used such as Fourier transform infrared spectroscopy , X-ray diffraction and Scanning electron microscope.

2.3 E-glass fibers

Glass fibers are coated with chemicals to enhance their adhesion properties. These chemicals are known as “coupling agents”. Glass fibers are the most common of all reinforcing fibers for polymeric matrix composites. The most widely used glass for fiber reinforced composites is called E-glass. The principle advantages of glass fibers are that they are cheaper than other relatively high modulus fibers and because of their flexibility do not require very specialized machines or techniques to handle them.

Their elastic modulus is, however, low when compared to many other fibers. The specific gravity of glass is relatively high. The poor specific value of the mechanical properties of glass fibers means that they are not ideal for structures requiring light weight as well as high properties, high chemical resistance, and excellent insulating properties. The disadvantages are relatively low tensile modulus and high density sensitivity to abrasion during handling relatively low fatigue resistance and high hardness [14].

Fig.2.3 E-glass fiber woven roving 310 gsm

The two types of glass fibers commonly used in fiber reinforced plastics industry are E-glass and S-glass. Another type, known as C-glass, is used for chemical applications requiring greater corrosion resistance to acids. E-glass has the lowest among them. These glass fibers are available in woven form, such as woven roving and woven cloth. Woven roving is a coarse drapable fabric in which continuous roving woven is found in two mutually perpendicular directions shown in Fig. 2.3.

2.4 BANANA FIBER REINFORCED POLYMER NANOCOMPOSITES

The banana nanofibers with varying percentage weight (neat composite, 2, 4, 6 and 8 wt.%) reinforced in epoxy resins to prepare nanofiber composites by hand lay-up technique. A rectangular mould of size (300x300x3) mm^3 is used for making the nano fibers reinforced polymer composite. For fabricating composites the mould is coated with one layer film of liquid polyvinyl alcohol as an easy releasing agent. The composites were prepared by using a glass fiber woven mat and epoxy resin with weight fraction. The banana nanofibers with different weight percents were mixed by using a magnetic stirrer at different RPM for 30 minutes at room temperature. The mixture was then placed in the vacuum oven, at room temperature to remove the air bubbles, which resulted in good dispersion of nanofibers in the epoxy resin. Then, for each 100 gm of epoxy resin, 12% of curing agent TETA was added to the mixture by weight and thoroughly mixed until it became uniform. The combination of polyester resin, e-glass fiber and banana nanofibers preparation process for making nanocomposites.

A plain roller soaked with a mixture of polymer resin rolled over the mould surface to make the first layer of banana nanofibers epoxy resin, followed by the fibers in the form of a mat or woven roving. Entrapped air and volatiles are removed by rolling the composite laminate with a smooth steel roller, which also ensured that the resin is uniformly distributed over the glass fiber surface. The process is repeated to get the composite laminate thickness of 3 mm plate. Finally, the composite has been allowed to fully cure at room temperature for 24 hours. The finished laminate was used to prepare samples for testing the mechanical, thermal properties and water absorption studies as per ASTM standards.

2.5 MECHANICAL STUDIES OF BANANA FIBER POLYMER NANOCOMPOSITES

Mechanical studies at nanometer scales are becoming important role to understand, areas of the greatest contributions, multidisciplinary field, atomic-level effects. The great interest in nano-structured from the observations and expectations of unique mechanical properties of these materials. Particular interest is its potential technological applications. Although enhanced mechanical properties are often associated with the reduction in grain sizes. Such peculiar behavior has been observed in nano-phase materials (e.g., nano-structured) with porous grain boundaries by means of experiments.

2.5.1 Banana fiber polymer nanocomposites - Tensile test

Tensile properties are the most widely specified properties and are used as an indication of the strength of polymers. The tensile test measures the ability of a material to withstand the applied forces that tend to pull it apart, and the extent of deformation before breaking. This test method covers the determination of tensile properties of Banana fiber reinforced polymer nanocomposites in the form of a standard dumbbell shaped specimen. This test method is designed to produce tensile property data for the control specification of plastic materials. This data is also useful for qualitative characterization, and research and development. The material specifications should be carefully studied before using this test method as tensile properties may vary with the specimen preparation.

2.5.1.1 Tensile test equipment

In order to analyse the behavior of the fabricated composite, it was subjected to tensile test. The tensile tests were carried out using the Tinus Olesan UTM machine as shown in Fig.2.4. Different types of self-aligning grips are used for holding test specimens in the machine. There are basically five testing speeds mentioned in the ASTM D-638 standards, testing speed for conducting the experiment is 5 mm/min. The equipment is fitted with load cell and extensometer to record the test load and extension accurately.

Fig.2.4 Computerized UTM (Tinus Olesan)

The fabricated Banana fiber reinforced polymer nanocomposites are prepared for the required specimen size as per ASTM D-638 Type-I to conduct the tensile test shown in Fig.2.5. The width and the thickness of each test specimen are measured accurately in the narrow parallel portions with accuracy. The initial position of the grips is adjusted and the specimen is mounted between the grips and aligned. The test speed depends upon the nature of the material to be tested. Generally, low speeds are used for rigid materials and high speeds for flexible materials. The specimen was placed in the grip of the test machine at a specified grip separation and pulled at a crosshead speed of 5 mm/min until failure occurred. The tensile test was carried out on three identical samples of each specimen and the average of these was taken to be the tensile strength.

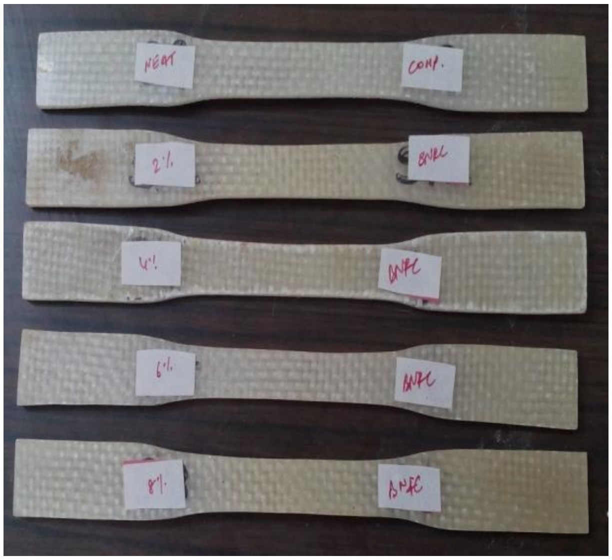

Fig. 2.5 Specimens for tensile test

2.5.2 Banana fiber polymer nanocomposites - Impact test

The Izod impact strength property of polymeric materials depends mainly on the toughness of the material. Toughness is defined as the ability of the polymer to absorb applied energy. The area under the stress, strain curve is directly proportional to the toughness of the material. The high impact strength of the material, the higher is the toughness of the material and vice versa. Impact resistance is the ability of a material to resist breaking under a shock loading or the ability to resist fracture under stress applied at a high speed. Molecular flexibility has a great significance in determining the relative brittleness and the toughness of the material. The objective of the impact test is to measure the relative susceptibility of a standard specimen to the pendulum type impact loading. The results are expressed in terms of kinetic energy consumed by the pendulum in order to break the specimen.

2.5.2.1 Impact test equipment

The impact properties are one of the widely specified mechanical properties of polymeric material. The different weight percentage of banana nanofiber reinforced composite specimens were tested by Izod impact test machine shown in Fig.2.6. The Izod impact test was modified by georges charpy to hold the specimen in a horizontal rather than a vertical position. This is the primary difference between the Izod and the charpy impact test. The Izod impact test differs also in that the notch is positioned facing the striker. The specimen size and shape vary with the Izod impact test according to what materials are being tested. Specimens of metals are usually square, and polymers are usually rectangular being struck perpendicular to the long axis of the rectangle. The impact strength is determined by the loss of energy of the pendulum as determined by precisely measuring the loss of height in the pendulum's swing.

Fig.2.6 Izod impact test machine

The standard test method, ASTM D-256, for studying the impact properties of banana nanofibers reinforced composites has been used to test composite specimens. The specimens are prepared dimensions shown in Fig.2.6. A V-notch is provided with a sharp file having an include angle of 45° at the centre of the specimen and at 90° to the sample axis. The depth of the specimen under the notch is 10.16 ± 0.05 mm. The specimen used in the Izod test must be notched. The reason behind notching the specimen is to generate a stress concentration area that promotes a brittle rather than a ductile failure. The impact values are severely affected because of the notch sensitivity of a certain plastic material. The impact testing equipment complies with ASTM D-256 standards depending on the weight fraction of the specimen, if a particular sample absorbs more than 85% energy of a particular hammer, then a higher range hammer has to be used for testing. The hammer is fixed to the pendulum in such a way that it will make initial contact with the specimen on a line 22 mm above the top surface of the clamping vice.

The sample is fixed to the vice as a vertical cantilever beam in such a way that the notch faces the striking edge of the hammer and aligns with the surface of the vice The pendulum hammer is released from its locked position which is at an angle of 90° to the axis of the specimen with a striking velocity of 2.46 m/s. The sample is stripped and the energy is indicated in joules by the pointer on the respective scale. The impact strength of three samples of each identical specimen is tested and the average values were taken.

The impact strength is given by

$$I = EI/t \quad J / mm^2 \tag{3.2}$$

Where

EI = Impact energy in joules value obtained from the scale (J)

t = thickness of the specimen (mm^2).

Fig.2.7 Specimens for impact test

2.5.3 Banana fiber polymer nanocomposites - Flexural test

The testing machine used for the flexural tensile strength test shall be one that has a load capacity greater than the maximum tensile capacity of the test specimen and is capable of applying loads at the prescribed loading rate shown in Fig.2.8 its configuration shall enable the tensile force to be applied by pressing with a bending device at the prescribed curvature on the test specimen bent to a 90° angle. The width of the straight portion of the test specimen shall be measured to within approximately 0.1 mm at two locations on the anchoring side and two locations on the fastening side.

Fig.2.8 Flexural test machine (3 point bent test)

2.5.3.1 Flexural test equipment

The test specimen shall be attached by fastening the pin anchorage in the fastening portion of a special device and applying a bending device with the same curvature as the bending portion of the test specimen, and then fastening the anchorage of the portion of the device of the tensile testing machine shown in Fig.2.9. The test specimen ASTM D-790 shall be fastened so that the long axis of the test specimen coincide with the loading axis, and that the bending portion of the test specimen and the bending portion of the device overlap.

The bending portion of the test specimen moves a minute distance toward the anchorage portion before failure, due to expansion of the test specimen itself, loosening of the device, etc. Therefore, the test specimen should be positioned on the fastening portion side so that the bending portion of the test specimen comes in contact with the device during failure. The loading rate shall be adjusted so that the strain rate in the test portion is approximately 1.0-2.0% per minute. The scope of test loading shall be performed until the test portion fractures and measurements shall be made and recorded continuously or at regular intervals until the maximum capacity occur.

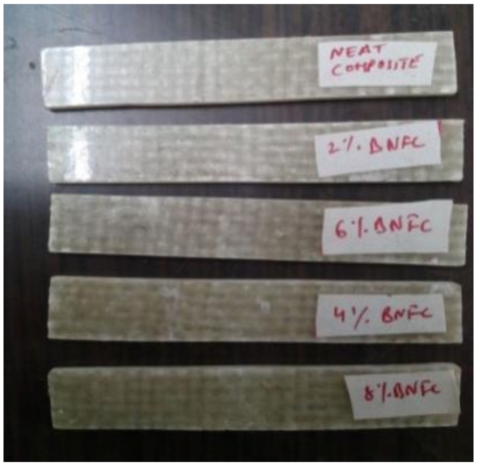

Fig.2.9 Specimens for flexural test

2.5.4 Banana fiber polymer nanocomposites - Hardness test

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. However, the term hardness may also refer to resistance to bending, scratching, abrasion or cutting. The surface mechanical properties of the material relate to the forces that are applied on the surface of the product and is transmitted to the entire product. Mechanical stress is applied at the surface of plastics and due to that, deformation and reduction of properties take place. The surface properties include hardness, abrasion resistance, co-efficient of friction, wear and scratch resistance etc. Hardness is an important surface property which represents the viscoelastic response of the material.

The resistance of a material to an indentation deformation is hardness. The higher the hardness, the better is the material's resistance to the indenting deformation. Hardness is used for quality control and is used as an indicator of surface durability. It can also be used to predict the length of time that a product will give a trouble free service. The hardness test of thermoset is used to find out whether thermosetting resins have been completely cured or not. Uncured resins show lower hardness than cured ones. The barcol hardness tester is used for measuring the hardness of reinforced and unreinforced rigid plastics. This test is based on the penetration of a specified indenter on to the surface of the object and the hardness is read directly from the dial indicator. Barcol tester is a digital hardness tester especially suited to testing flat or lightly curved specimens such as: glass fiber reinforced plastics, droplets, hard thermoplastic, finished and semi-finished goods. The hardness of materials has probably long been assessed by resistance to scratching or cutting.

2.5.4.1 Hardness test equipment

The barcol hardness tester is a portable type of equipment and consists of a sharp indenter. The Indenter is a frustum of 26° cone with a flat tip surrounded by a concentric sleeve. The indenter is mob and made of hardened steel. The Hardness indicator device is having division from 0 to 100. The barcol hardness tester is shown in Fig.2.10. The composite specimen sample should be flat and at least 1.5 mm thick. The samples are placed on a rigid flat surface and the equipment indenter is placed over the specimen. Hand pressure is applied to the sample against the surface until the dial indication reaches the absolute barcol number which is directly read from the dial indicator.

The hardness reading is higher than the largest value stamped on the test disc, then the plunger upper guide nut is tightened further into the frame. Test again, if the reading is lower than the smaller value stamped on the test disc, loosen the plunger upper guide nut. Test again, if reading within the specified limits cannot be obtained by the plunger upper guide nut adjustment, loosen the lock nut and make minor adjustments to the lower plunger guide. Retighten the lock nut and test again. Repeat until the readings are between the values stamped on both test discs.

Fig.2.10 Specimens tester by barcol hardness tester

2.5.5 Banana fiber polymer nanocomposites - Damping test

Damping is an important modal parameter in the design of structures for which vibration control and cyclic loading are critical. Damping is also a significant factor for the fatigue life and impact resistance of structures. It varies with different environmental effects such as frequency, amplitude of stress, temperature and static load. It can be classified into two categories, namely material damping and system damping. Material damping is defined as its inherent property to dissipate energy in a volume of macro continuous media. System damping relates to the energy dissipation of the total structure. It includes the energy dissipation effects of joints, fasteners and interfaces in addition to energy dissipation due to the material. The mechanical vibration damping can be defined as the ratio of energy dissipated to maximum strain energy per cycle of vibration. Temperature is usually one of the most important factors for damping in polymers and polymeric materials.

There are several ways to set up the vibration damping experiment in practice. A frequently used method is to gather experimental data concerning dynamic material behavior by studying the free and forced transverse vibrations of cantilever beams made of the material. The information on beam behavior collected in the experiments is interpreted by comparing it with the behavior of a uniform, homogeneous bernoulli-euler cantilever beam, vibrating in its various principal modes. The widely accepted experiment was done by detecting the response of the cantilever beam excited by a vibration exciter. Resonant frequencies were determined by observing the peaking of response by the varying excitation frequency at constant excitation amplitude. Free vibration decay measurements were usually made by exciting the beam at a resonant frequency, cutting power to the exciter, and analyzing the decay trace [15-16].

2.5.5.1 Damping test equipment

The fabricated nanocomposite laminates were prepared as a specimen size of a 300x25x3 mm^3 rectangle beam to determine the damping property of banana nanofiber composites as shown in Fig.3.20. The beam is clamped to simulate a fixed end boundary condition. The accelerometer is positioned in the beam, and sinusoidal excitation is provided by the exciter. The load is applied to the specimen to induce vibration using a spherical steel ball hammer. The response of the beam is measured by the accelerometer and is processed through the signal condition equipment. The banana nanofibers composite specimen is clamped in the vice as a cantilever beam shown in the Fig.2.11. The resulting response is stored in a computer for later analysis. This process is repeated five identical samples for each specimen and the readings are recorded for analysis.

Fig.2.11 Damping test experimental set up

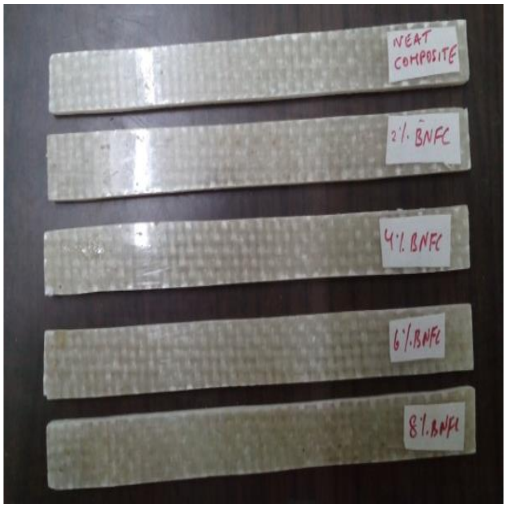

Fig.2.12 Specimens for damping test

2.6 BANANA FIBER POLYMER NANOCOMPOSITES FOR THERMAL PROPERTIES

Thermal properties play a vital role in evaluating the composite performances as well as predicting the possibility characteristics in polymers for specific applications. These properties are of great importance to process engineers and designers in designing the machines and tool for the shaping of polymer composites.

2.6.1 Studies on thermogravimetric analysis

Thermogravimetric analysis measures the amount of weight change in the weight of a specimen are recorded as the specimen is heated in air or in a controlled atmosphere of a material, either as a function of increasing temperature, or isothermally as a function of time, in an atmosphere of nitrogen, helium, air, other gas, or in vacuum. Thermal gravimetric analysis can be interfaced with a mass spectrometer TGA to identify and measure the vapors generated, though there is greater sensitivity in two separate measurements. Inorganic materials, metals, polymers, plastics, ceramics, glasses, and composite materials can be analyzed. Temperature range from 25°C to 900°C. The maximum temperature is 1000°C. Sample weight can range from 1 mg to 150 mg. Sample weights of more than 25 mg are preferred, Samples can be analyzed in the form of fibers or small pieces so the interior sample temperature remains close to the measured gas temperature.

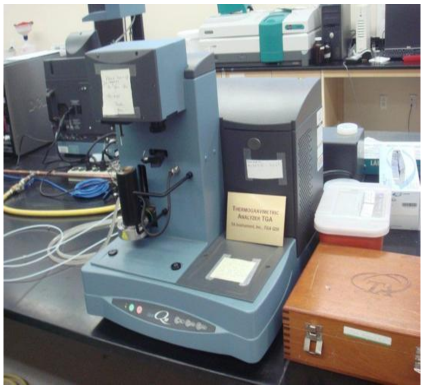

Fig.2.14 Thermogravimetric analyzer

This type of testing is performed on samples to determine changes in weight in relation to a change in temperature. Such analysis relies on a high degree of precision in three measurements. Thermograms provide information regarding polymerization reactions, the efficiencies of stabilizers and activators, the thermal stability of final materials and direct analysis. The weight loss curves may require transformation before the results may be interpreted. Again, interpretation is limited without further modifications and de-convolution of the overlapping peaks may be required. These curves are used to study the thermal behavior of the nanocomposites. This test is used to determine the degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives and solvent residues, thermogravimetric analysis equipment as shown in Fig. 2.14.

Thermogravimetric analyzer provides superior performance, which is achieved using a vertical balance design with a horizontal gas purge. It consists of an automated pan loading and furnace movement plus software controlled dual mass flow controllers with automated gas switching. The accessory kit contains platinum sample pans calibration and test materials plus tools. Thermogravimetric analysis is used to determine the heat to force reactions and physical changes in materials. TGA provides a quantitative measurement of the mass change in materials associated with transition and thermal degradation.

Thermogravimetric analysis records change in mass from dehydration, decomposition and the oxidation of a sample with time and temperature, curves are given for specific materials and chemical compounds due to the unique sequence from physical and chemical reactions occurring over specific temperature ranges and heating rates. Thermogravimetric analysis, the percentage weight loss of a test sample is recorded while the sample is being heated at a uniform rate in an appropriate environment. The loss in weight over specific temperature ranges provides an indication of the composition of the sample, including volatiles and inert filler, as well as indications of thermal stability. Set the inert (usually N_2 gas) and oxidative (O_2) gas flow rates to provide the appropriate environments for the test. Place the test specimen in the specimen holder and raise the temperature of the furnace. Set the initial weight reading of 100%, and then initiate the heating program. The gas environment is preselected for either a thermal decomposition (inert- nitrogen gas), an oxidative decomposition (air or oxygen) or a thermal-oxidative combination.

In the present investigation the thermal degradation behavior of the banana fiber nanocomposites was characterized. The sample was approximately 10 mg of banana nanofibers composites were heated from room temperature to 800°C at a rate of 10°C/min under nitrogen atmosphere. The magnitude of the decomposition affect depends on the material properties and the contact area between the specimen and the pan. The purge gas determines the type of decomposition. The inert purge gas such as helium, nitrogen, are suitable for determining purely thermal decomposition. Oxygen and air serve as oxidizing purge gas for the measurement of thermo-oxidative decomposition. The heating rate has a major effect on the results. Doubling the heating rate, shifts the initial degradation temperature to comparatively higher. The oil filled elastomers having high molecular weight oils and low molecular weight polymer content may not be separated based upon temperature stability.

2.6.2 Studies on differential scanning calorimetry

Differential scanning calorimetry measures the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes. Differential scanning calorimetry is a thermo analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at very nearly the same temperature throughout the experiment. Measurements of specific heat and enthalpies of transition can be carried out on quite small samples in a differential scanning calorimeter. In differential scanning calorimetry, an average temperature circuit measures and controls the temperature of the sample and the reference holders to conform to a predetermined time-temperature program. This temperature is plotted on one axis of an x-y recorder. A schematic drawing of a differential scanning calorimetery experimental flow chart as given in Fig.2.15. There during the experiment, the sample undergoes a thermal transition, the power supply to the two heaters is adjusted to maintain their temperatures and a signal proportional to the power difference is plotted on the second axis of the recorder. The area under the resulting curve is a direct measure of the heat of transition.

Two sample chambers a ‘reference’ chamber and the ‘sample’ chamber. An empty pan is placed in the reference chamber and a pan filled with a composite specimen is placed in the sample chamber. The sample and reference are maintained at the same temperature during the entire experiment. A thermal event occurs in the sample to maintain zero temperature between the sample and the reference. Therefore, the reference should not undergo any physical and chemical changes at the temperature range of the experiment. The sample and reference are placed in identical environments, namely, metal pans on individual bases, each of which contains a platinum resistance thermometer and a heater. An empty pan is often used as a reference. The temperatures of the two thermometers are compared and the electric power supplied to each heater adjusted so that the temperatures of the sample and the reference remain equal, i.e. any temperature difference which would result from a thermal event in the sample is compensated for. If an exothermic change occurs in the sample, more heat has to be supplied the reference. During an endothermic process, an additional amount of energy has to be supplied to the sample heater. The difference in the heat supplied to the sample and the reference is recorded as a function of temperature.

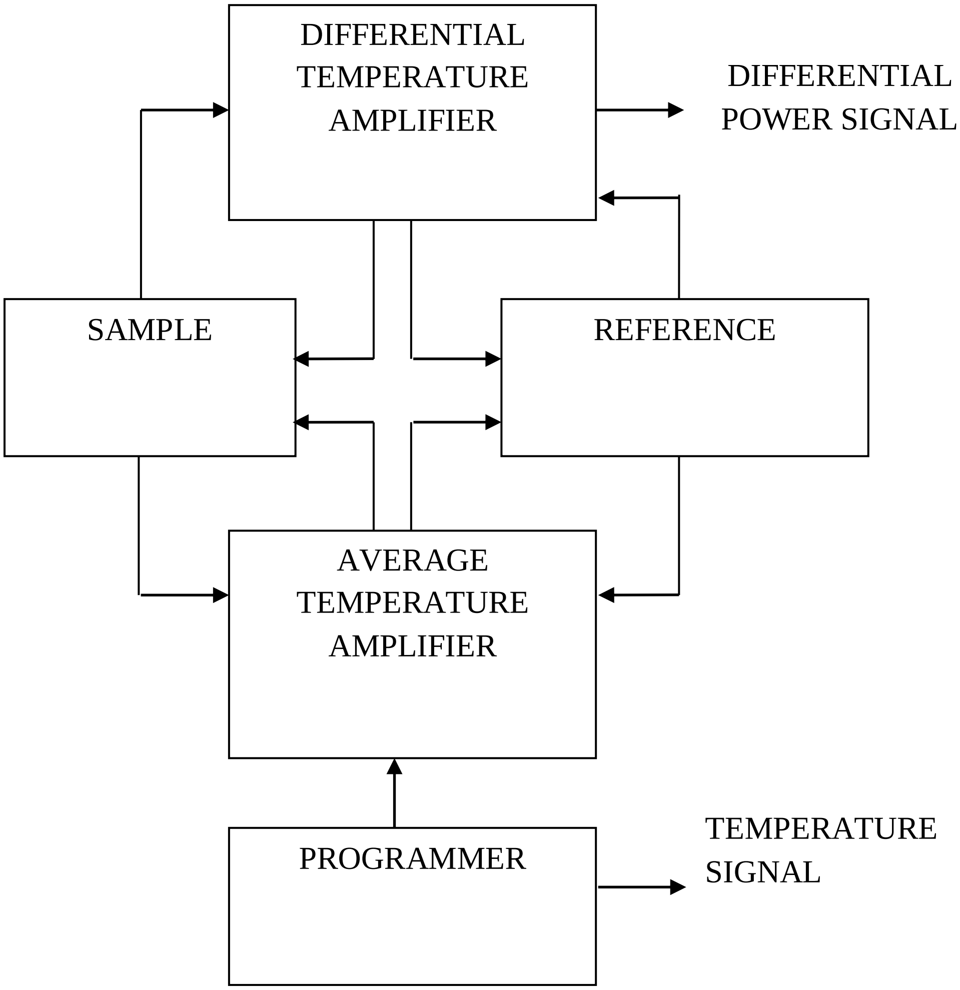

Fig.2.15 Differential scanning calorimetery experimental flow chart

The signal is proportional to the specific heat, which determines the amount of heat that is necessary to change the sample temperature by a given amount. Any transition that is accompanied by a change in specific heat produces a variation in the power signal. Exothermic and endothermic processes give peaks with areas proportional to the total enthalpy change of the events. The two chambers are then heated or cooled uniformly at the same rate and their temperatures are maintained at the same level. The difference in the energy supplied to the two chambers per unit time or (dq/dt) is proportional to the heat capacity of the sample. The changes in the sample that is associated with absorption or evolution of heat cause variations in the differential heat flow which are then recorded as peaks. The area of an individual peak is directly proportional to the enthalpy change of the sample and the direction of the peak indicates whether the thermal event is exothermic or endothermic. It measures the heat flow which is converted into heat capacity.

An alternative approach of differential scanning calorimetery is to measure the temperature difference. In particular, the reference and sample chamber can be supplied with the same amount of heat. The amount of heat supplied can be adjusted to give various heating rates. Then, instead of maintaining equal temperatures and measuring differential heat flow, it is possible to maintain a constant heat flow and measure the difference in temperature between the reference and sample chambers. The temperature difference will give a good measure of the heat capacity. When the heat capacity of the sample chamber increases the temperature in the sample chamber will lag behind the reference chamber and there will be a negative temperature differential conversely, when the heat capacity of the sample chamber decreases the temperature in the sample chamber will be ahead of the reference chamber and there will be a positive temperature.

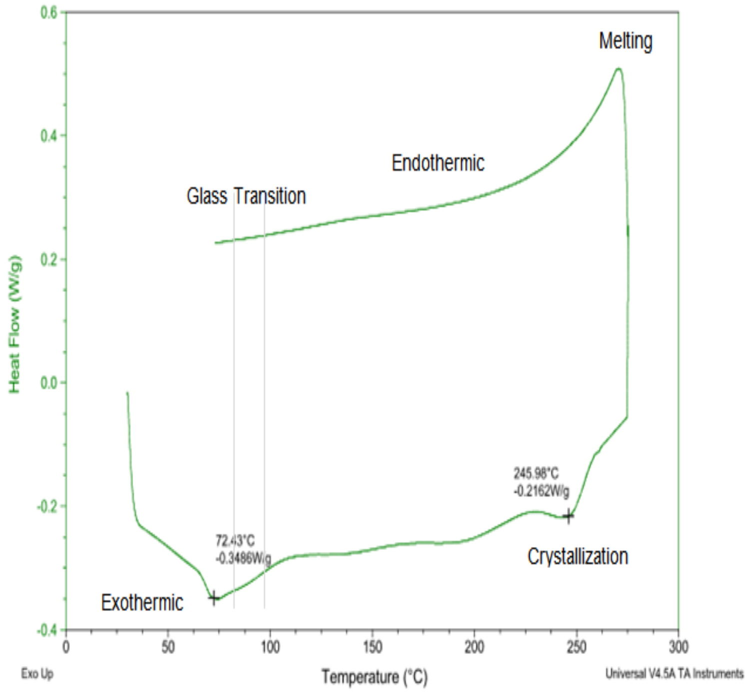

Fig.2.16 Sample differential scanning calorimetery curve

Differential scanning calorimetry can be used to measure a number of characteristic properties of a sample. Using this technique, it is possible to observe fusion and crystallization temperature (T_c) as well as glass transition temperatures (T_g). differential scanning calorimetery curves as shown in Fig. 2.16 can also be used to study the heat flow, crystallization and melting point with respect to temperature, glass transitions may occur as the temperature of an amorphous solid is increased. These transitions appear as a step in the baseline of the recorded DSC signal. This is due to the sample undergoing a change in heat capacity, no formal phase change occurs. As the temperature increases, an amorphous solid becomes less viscous. At some point the molecules may obtain enough freedom of motion to spontaneously arrange themselves into a crystalline form. This is known as the crystallization temperature (T_c).

This transition from amorphous solid to crystalline solid is an exothermic process and results in a peak in the differential scanning calorimetery signal. As the temperature increases the sample eventually reaches its melting temperature (T_m). The melting process results in an endothermic peak in the differential scanning calorimetery curve. DSC equipment‘s shown in Fig.2.17. The ability to determine transition temperatures and the enthalpies make DSC an invaluable tool in producing phase diagrams for various chemical systems.

Fig.2.17 Differential Scanning Calorimetry

The instrument was calibrated with two metal reference materials. Indium and zinc were used for temperature calibration and indium was used for heat of fusion for heat-flow calibration. During crystallization, the temperature can easily change several degrees celsius, which influences the data obtained and the results of any kinetic studies. It is important to ensure that the DSC cell will achieve an isothermal temperature. The type of purge gas and flow rate affects calibration and therefore should be controlled. The purge gas pressure does affect measurements of the specific heat capacity C_p which by definition varies with pressure. Purge gas pressure has little effect on T_{pm} and ΔH_m but a greater effect on C_p. Nitrogen is used as the sample purge gas because it is inert and calibration is least affected by changes in flow rate.

The addition of fillers or reinforcements significantly affects the transition peaks. The incorporation of fillers affects the crystallization rate and hence the crystallization peak shifts lower in the thermogram with increasing filler content [13 and 14]. The thermal history of the samples also affects the transition peaks, and the quenched materials exhibit an amorphous structure showing a strong glass transition associated with the melting and crystallization peak in the thermogram. The slow cooled material on the other hand shows the presence of a strong melting peak and very weak glass transition in the thermogram. In the present investigation, the melting and crystallization behavior of the pure epoxy polymer and nanofibers composites with different weight percentages Neat Composite, 2, 4, 6 and 8wt.% of Banana nanofibers were investigated under nitrogen atmosphere using a sample of 10 mg. A heat-cool-heat regime was applied. It was heated from room temperature to 300°C at a rate of 10°C/min before cooling down to room temperature at a rate of 50°C. The initial and final degradation temperature and the corresponding percentage weight loss of the samples were recorded for the analysis.

2.6.3 Studies on dynamic mechanical analysis

Dynamic mechanical analysis is a thermal analysis technique that measures the properties of materials as they are deformed under periodic stress. In dynamic mechanical analysis a variable sinusoidal stress is applied, and the resultant sinusoidal strain is measured. If the material being evaluated is purely elastic, the phase difference between the stress and strain sine waves is 0°. If the material is purely viscous, the phase difference is 90°. However, most real world materials, including polymers are viscoelastic and exhibit a phase difference between those extremes. This phase difference together with the amplitudes of the stress and strain waves are used to determine a variety of fundamental material parameters, including storage modulus, loss modulus, tan delta, complex and dynamic viscosity, storage and loss compliance, transition temperatures, creep, and stress relaxation, as well as related performance attributes such as rate and degree of cure, sound absorption and impact resistance, and morphology. Dynamic mechanical analysis equipment as shown in Fig.2.18 measurements are made using a single frequency and constant deformation amplitude while varying the temperature. Measurements, where the amplitude of deformation is varied or where multiple frequencies are used, provide further information.

The dynamic properties are expressed in terms of storage modulus, loss modulus and damping factors which are dependent on temperature, time and frequency. The storage modulus is an indication of the capability of a material to retain energy and relates to the stiffness of a material while the loss modulus represents the energy dissipation characteristics, owing the viscous motions within the material. The loss tangent is an indication of the viscoelastic behavior of the composite materials. It is independent of the shape and dimensions of the samples and is a dimensionless quantity. The tan δ is obtained as the ratio between the loss and storage modulus.

The sample is clamped between the ends of two parallel arms. The distance between the arms is adjustable by means of a precision mechanical slide to accommodate a wide range of sample lengths from <1 mm upto 65mm. An electromechanical motor attached to one arm drives the arm/sample system to a selected strain or amplitude. As the arm of the sample system is displaced, the sample undergoes deformation. A linear variable differential transformer mounted on the driven arm measures the sample response, strain and frequency as a function of the applied stress and provide feedback control to the motor.

The sample is positioned in a temperature-controlled chamber. This heating system is precise and gives accurate control of the sample temperature. The instrument is calibrated prior to test using the same heating rate or schedule to be used for standard specimens. The specimens are subjected to the three point bending test. The test specimens should be conditioned a 23±2°C and 50±5% relative humidity for not less than 40hrs prior to the test.

Fig.2.18 Dynamic mechanical analyzer

The maximum strain amplitude should be within the linear viscoelastic range of the material. In the present work the dynamic behavior of the Banana nanofibers composite samples was investigated. The banana nanofibers composites of different weight percentages neat composite, 2,4,6,8 weight percentage of banana nanofibers specimen samples were prepared as rectangular bars of size 40x10x3 mm^3 as per ASTM standards as shown in Fig.2.19.

Fig.2.19 Specimens fabricated for DMA (3-point bending)

The ends of the sample are freely supported and the load is applied to the midpoint. To ensure direct contact with the specimen, an additional inertial member needs to be applied. It is a simple arrangement, but additional shear stress in the midpoint plane of the specimen must be taken into account. This test arrangement is suitable for very stiff materials, such as metals, ceramics and composites. The nanofibers composites of Neat composite and different weight percentage nanocomposites composites are carried out at a fixed frequency of 1Hz and the temperature range is between 0°C to 150°C at a scanning rate of 5°C/min under controlled conditions. The data collected from the experimental results were used to study the storage modulus, loss modulus and tan delta properties of the nanocomposites.

2.7 SUMMARY

The chapter explains the various steps involved in the synthesis of the banana natural fibers into nano fibers with the use of mechanical milling using a high energy ball mill. The fourier transform infrared spectroscopy is used to study the chemical structural of treated and untreated banana fibers. The characterization of nanofibers is carried out on XRD and SEM. The particle size of the nanofibers is analyzed by the X-ray diffraction. The scanning electron microscope is used to characterize the morphology of banana nano fibers. The material required for fabrication of nanofiber composites is also discussed. The nano fibers with different weight percentage mixed with epoxy is prepared using a hand lay-up technique. The workpiece samples are prepared strictly as per the ASTM standards. The fabricated composite material is analysed in terms of its mechanical properties, water absorption studies and thermal properties.

CHAPTER - 3

MECHANICALPROPERTIES OF THE BANANAFIBER POLYMER NANOCOMPOSITE

3.0 INTRODUCTION

In this chapter, the physical and mechanical properties of nanocomposite were observed experimentally reported. The main factors, which influence the properties of the polymer nanocomposites. The bananafibers were extracted is done by chemical treatment and mechanical milling processes. The nanofibercomposites are prepared by reinforcing banananananofibers in epoxy resin. The chemical treatment of bananaFibers was analyzed by fourier transform infrared spectroscopy analysis. The morphological studies werestudied by the X-Ray diffraction and the scanning electron microscope. The nanocomposites were fabricated by reinforcing the banana nanofibers with different weight percentagesin the epoxy polymer matrix.Experiments were carried out as per ASTM standards to determine the mechanical properties and water absorption capacity of the nanocomposite. The addition of banana nanofibersinfluenced on mechanical propertiesand water absorption was discussed.

3.1 PHYSICAL BEHAVIOR OF BANANAFIBERS

The change in the nature of relaxation can be due to the nature of the fiber. As well, due to the physical and chemical changes involved during the relaxation process. The composites with better mechanical properties and environmental performance, it becomes necessary to increase the hydrophobicity of the cellulose fibers and improve the interface between the matrix and fibers. Good interracial adhesion, a low melting point and poor resistance towards moisture make the use of plant cellulose fiber reinforced composites less attractive. Pre-treatment of the cellulose fiber can clean the fiber surface, chemically modify the surface, stop the moisture absorption process and increase the surface roughness.

3.1.1 Mercerization of bananafiber surface treatment

The fibers were treated with NaOH 1wt. % to 5wt.% aqueous solution for an hour at a room temperature,the fiber to solution weight ratio was maintained at 1: 25. It was then washed with distilled water until all the sodium hydroxide is eliminated, that is until this water no longer indicated any alkalinity reaction, submerged banana fibers in NaoH Solution as shown in Fig.3.1. The fibers were dried at 80°C for 24hours, It is observed that when the fiber is submerged in 3wt.% of NaOH, there is more loss of weight than when compared to that of 1,2,,4 and 5wt.% by continuous stirring for 20 min. The fiber in the solution is continuously stirred which is carried out for 20 min. Then the fibers were immersed in the solution and left for one hour under agitation. Afterwards, the fibers were dried at 60°C for 24hours.

$$\% \text{ of Lignin removed} = \frac{w_i - w_f}{w_i} X\ 100 \qquad (4.1)$$

Where, w_i = weight of the fibers before chemical treatment.

w_f = weight of the fibers after the chemical treatment.

Fig.3.1 submergingbanana fibers in NaOH solution.

The raw banana fiber by chemical treatment observed that loss in weight of banana fibers,this loss is due to lignin present in the banana fibers. As a percentage of

NaOH increases the weight loss of banana fiber at 3% of NaOH, after decreasing for higher concentration of NaOH,the alkali treatment has removed the surface debris from the fiber, most of the lignin and pectin remove from the fiberrough surface. This rough surface facilitates both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups to the matrix, thereby increasing the fiber matrix adhesion [204-205],It is observed that the fibers are somewhat leached and appears to be finer. This is due to the chemicaltreatment which reduces the hydroxyl group in the cell wall of natural fiber molecules, thus decreasing the water absorption of the fiber.Thegraph shown in theFig.3.2.Thus 3% NaOH is the optimum concentration of NaOH in removing lignin content present in the fibers.The Fig.4.3.Showsuntreated and treated fibers.

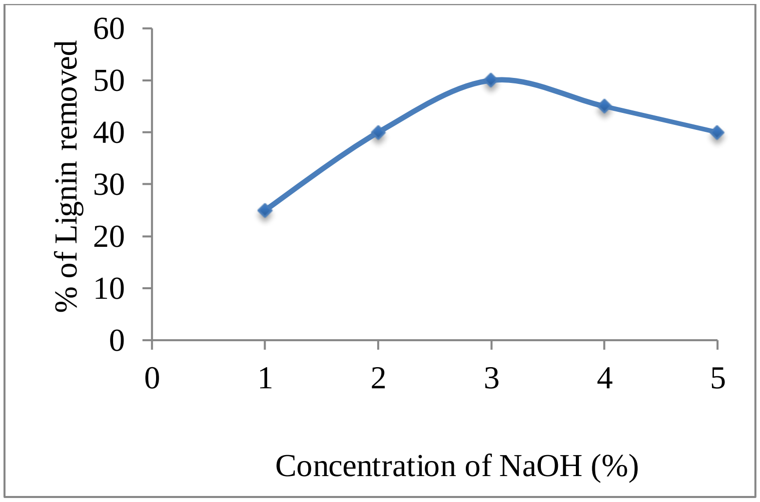

Fig.3.2 Percentage of lignin removed with concentration of NaOH

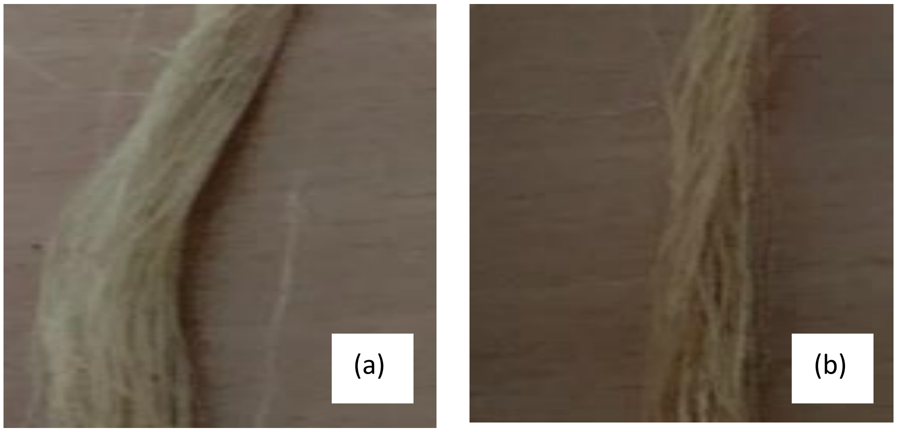

Fig.3.3varioussteps of (a) Raw bananafiber (b)Chemical treatedbananafiber

3.2 BEHAVIOUR OF ALAKALI TREATED BANANA FIBER BY USING FOURIER TRANSFORM INFRARED SPECTROSCOPY

Fourier transform infrared spectroscopy study the interaction between infrared light and matter, identification of compounds in chemistry. Fourier transform defines a relationship between a signal in time domain and its representation in the frequency domain. FTIR is a non-destructive method for studying properties of lignocellulolosic materials. The FTIR spectra of treated and untreated bananafibers absorbance peaks of interest in this study have been identified.The rawbanana fiber treated with 3% NaoH at 80°C. Alkaline treatment reduced hydrogen bonding due to removal of the hydroxyl groups by reaction with sodium hydroxide.

The peak arising at 3572 cm^{-1} corresponds to the OH stretching vibration of the water and alcohol group. The peaks at 2950 cm^{-1} and 2653 cm^{-1} indicate the existence of the CH_2 vibration and aromatic CH stretching vibration respectively. The peaks around 1856 cm^{-1} and 1726 cm^{-1} can be attributed to the stretching of the carbonyl group of lignin and aromatic ring skeleton vibration respectively. The peaks appearing at 1563 cm^{-1} correspond to the bending of the CH_2 group. The peaks at 1226 cm^{-1} and 1078 cm^{-1} illustrate the presence of phenolic CO of lignin and C-O-C stretching vibration, respectively.

FTIR analysis of the chemical treated banana fiber,infrared measurements were performed to find out the removal of lignin, hemicellulose and waxy substances. The increase in the cellulose, as confirmed by the FTIR analysis, is shown inFig 4.4. The strong O-H stretching absorption value 3456 cm-1.The characteristic peaks of cellulose and lignin are 1622 cm^{-1} to 1676 cm^{-1} increased, which confirmed that the content of cellulose increased and the content of lignin decreased.The strong peak indicates that the lignin was removed by chemical and physical treatments. This result is consistent with the treated fiber structure that the lignin content decreased and the cellulose content increased. From this analysis, it is observed that alkalization provides surface improvement of treated bananafibers. The chemical structure of alkaline treated and untreated bananafiber has been analyzed by the fourier transform infrared spectroscopy the results shown in the givenTable 3.1.

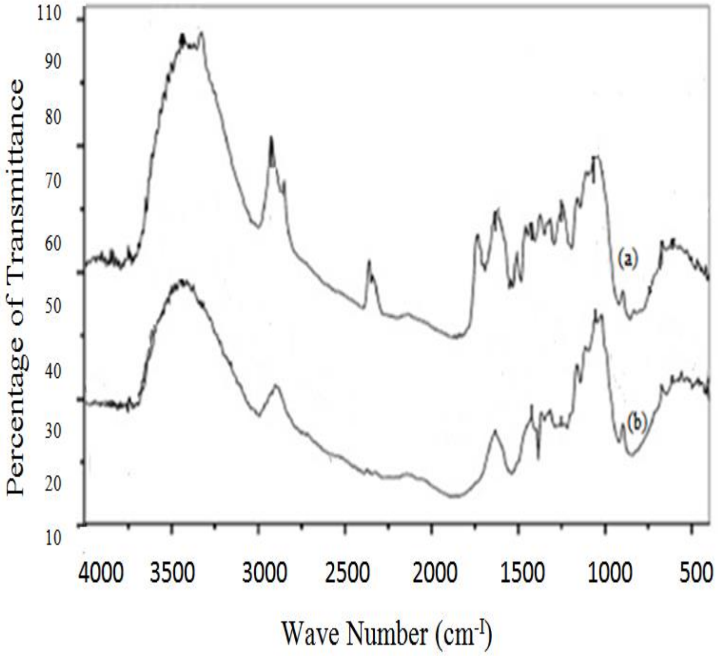

Fig.3.4 FTIR Spectra of (a) Raw bananafibers and (b) 3% NaoH treated bananafibers

3.3 MILLING OF BANANAFIBERS

Ball milling has been widely utilized in various industries to perform size reduction for a long time. The materials with novel micro structures and properties have been synthesized successfully by high-energy ball milling processes. The morphology of bananafibers changed from the micro to the nano scale during the process. It is clearly observed that the chemical treatments reduce the Fiber size and surface roughness. The milling process imparted sufficient energy to break the bundles into smaller particles.The finer particles of Fibers are defibrillated and the size is reduced from the micro to nano level. The milling speed of 200 RPMand the ball diameter 10mm and 5mm were experiments carried out at different periods milling hours. The reduction in the particle size was generally higher at 20 hours of milling, it was used due to high impact force generated during milling. However, once the particle size was reduced, later milling hours are increased 20hourslike, worked better for further size reduction. The constant speeds and constant ball size during the ball milling process.As the fragments decrease in size, fracture resistance as well as a tendency to aggregate increases, and particle fineness approaches a limit.

The finer particles are produced with a constant in the ball diameter and an increase in milling hours. It may be concluded that the amount of energy transmitted to the system is the main factor in determining particle size in the relatively early period, At later milling times, when the particle were already reduced to a sustainable smaller size. The small impact force may be less conductive for the generation of aggregation, but sufficient for fracturing small particles. After that, further milling does not help much in terms of particle reduction. The crystallite size of the milled banana fiber at different time of milling hours is determined using X-Ray diffractometer. The changes in the crystalline size in the bananafiber after milling have been found out with the help of wide angle X-ray diffraction studies. The average crystallite size is determined from the full width at half maximum (FWHM) of the X-Ray diffraction using.

3.4 X-RAY DIFFRACTION STUDIES ON BANANA FIBERS

X-ray fiber diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is fine, homogenized and average bulk composition is determined. The bananananofiberswere obtained by high energy ball milling was characterized by the X-ray diffractometer to estimate the broadening of an observed diffraction peak, crystalline size and phase identification by generating 0 to 60° of 2 theta scanning angle by using copper a target $\lambda = 1.54$ [A°] at 40 MV, 40 MA.The phase identification of the banana nanofiberis shown inTable 3.2.The XRD analysis shows that the banana nanofiber has a hexagonal structure as indicated as cellulose. The intensity lines were $2\theta = 24.6°$. The intensity line coincides with the diffraction peak maximum. The geometry of an X-ray diffractometer is such that the sample rotates in the path of the collimated X-ray beam at an angle θ while the X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of 2θ. The instrument used to maintain the angle and rotate the sample is termed a goniometer. For typical fiber patterns, data is collected at Bragg's angle 2θ, angles that are preset in the X-ray scan. By considering all the intensity lines, it is seen that only a few coincide with the diffraction peaks.

The average crystalline size was determined from the full width at half maximum (FWHM). The phase identification of bananafiber is cellulose and its crystal structure is identified in XRD. The XRD graphs shows that the intensities andsharp peak at 24.6° is the banana nanofibers due to the plane of 002 of cellulose-I, X-ray fiber diffractogram of different milling hours of XRD Scan as shown in Fig.3.6,3.7,3.8 and 3.9. The other sharp peaks appearing which signify that many new crystalline were generated due to ball milling. The crystalline size of bananafiber as received after different milling hours were calculated by using Scherrer formulae.

Scherrer formulae:

$$\text{Crystalline Size (d)} = \frac{K\lambda}{\beta\ (\text{Cos}\ \theta)} \tag{3.2}$$

Where

K = Shape factor (0.9 spherical particles)

λ = Wave length of copper K_α =1.540598

β= Full width half maximum (FWHM)

θ= Centre of the peak in degrees.

XRD results have been observed that the particle size decreased very rapidly as the diameter of the ball was reduced. This is due to the high impact force generated during ball milling, the collision action of media,fibers could not get flattened and both fracture and mechanical bonding of particles could occur. It is concluded that the average crystalline size of banana nanofiberhaving diameters in the range of 50-100 nm as received after 100 hrs of high energy ball milling, further milling will not affect the size.

3.5 SCANNING ELECTRONIC MICROSCOPE STUDIES

The scanning electron microscope (SEM) is a powerful and frequently used instrument, The SEM has an extremely large depth of focus and is therefore well suited for topographic imaging. The specimen is bombarded by a convergent electron beam, which is scanned across the surface. This electron beam generates a number of different types of signals, which are emitted from the area of the specimen where the electron beam is impinging,SEM was employed to monitor the surface morphologies. It representative SEM phase images of banana fibers. After alkali treated fibers exhibited significant changes in their surface characteristics. The surfaces of raw fibers have an irregular phase, which was also observed in the SEM images as shown in Fig.3.5. It is observed that the surface roughness value is 100 µm because of a layer of pectin, lignin and waxy material, which covers the surface of the fiber. After alkali treated fibers exhibited significant changes in their surface characteristics a roughness indicated by the surface roughness value 10 µm corresponds with the fibrillar structures on the surface as shown in SEM images Fig.3.6.

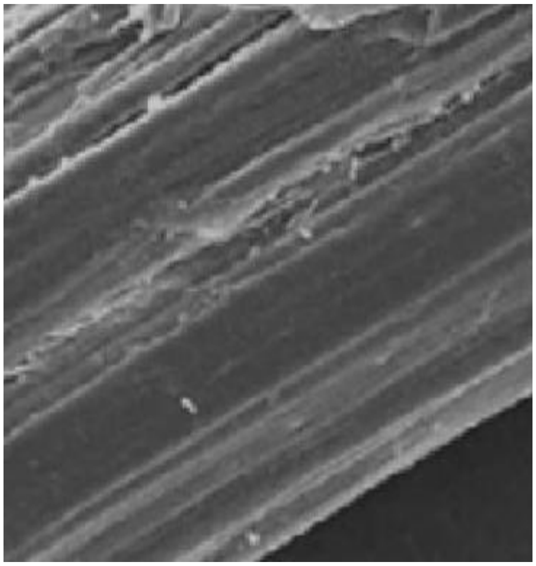

Fig.3.5 SEM of raw banana Fiber

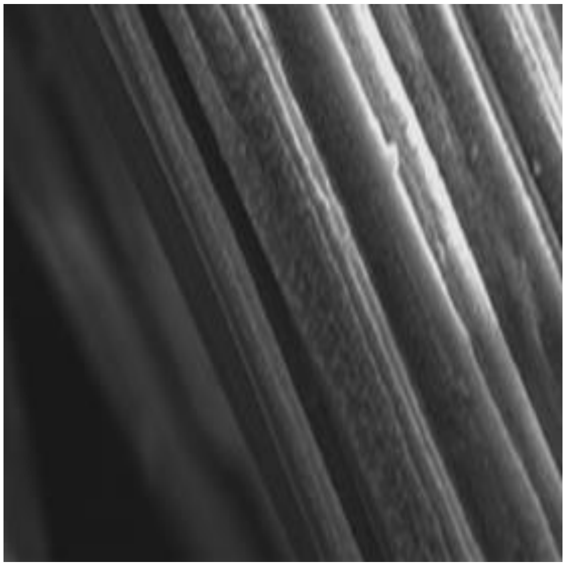

Fig.3.6 Alkali treated banana Fiber

The milled bananananofibers and micro fibrils were used for morphology analysis by the JEOL model scanning electronic microscope. SEM analysis observed that the effect of milling on bananafibers different milling hours of 20, 40, 60 and 80 hrs of ball milling. Fig.3.6 shows that the sizes of the fibers are reduced as compared with actual bananafibers. Bananananofibers were irregular and its average diameters of size are 350 nm after 20 hrs of milling. The SEM image of nanofiber, it is clear from the picture that the average diameters after 40 hrs of milling, which is lower than the size of 20 hrs milling. Fig. 3.7 shows the SEM observation was carried out to verify the microstructure characteristics. The SEM images show that at 20 hours milling the fibers are large and non-uniform in size. The nanofibers exhibited an elliptical shape and its length was a few hundred nanometers. The size of the particle is reduced by increasing the milling speeds. It demonstrates that the obtained cellulose banana fibrils are micro scale to nano scale. The crystalline structure of the banana nanofibers did not collapse completely even when long milling times were employed. A faster milling speed of 200 RPM at 60 hrs to 80 hrs milling led to smaller particle size while the crystalline was hardly affected is shown in Fig.3.8. When the small particles could be obtained only after longer milling times and the size span became smaller.

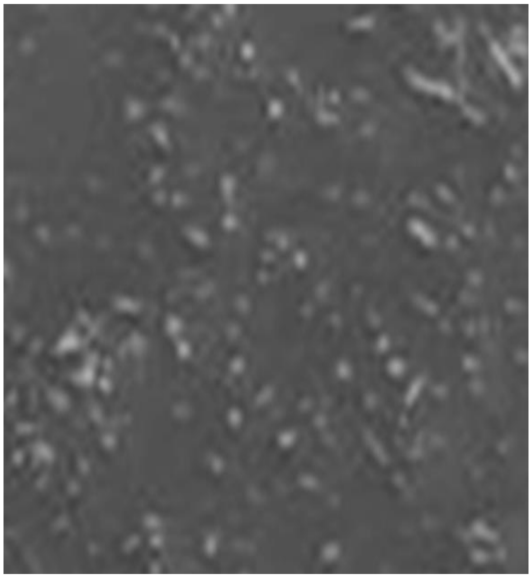

Fig.3.7SEM micrograph of banana nanofibers after 60hrs milling

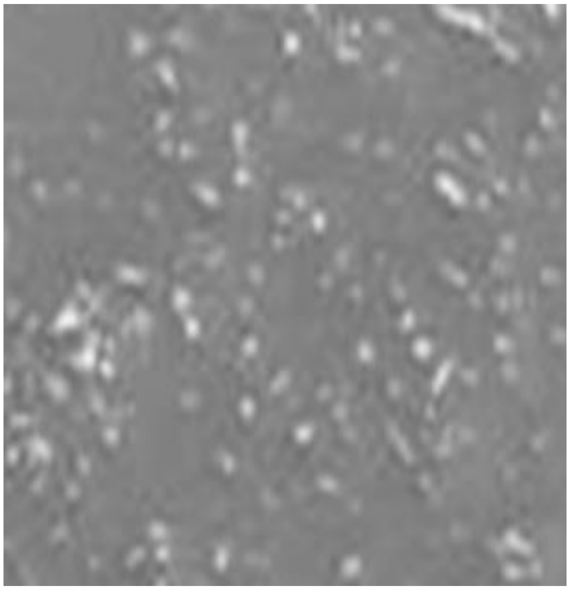

Fig.3.8 SEM micrograph of banana nanofibers after 80hrs milling

3.6 MECHANICAL PROPERTIES OF BANANAFIBERNANOCOMPOSITE

The mechanical properties such as tensile strength, flexural strength, impact strength, hardness and damping properties of nanocomposites are discussed. In polymer reinforcednanocomposites, the role of the resin is to bind the reinforcement together and transfer the external load from resin to reinforce uniformly. For this the primary requirement is that the resin should spread uniformly as a thin coating on the entire available Fiber surface. For each composite, three identical specimens were tested and the mean values were taken and plotted in graphs. The experimental values obtained for various mechanical tests conducted are represented in Table 3.3 (APPENDIX-I).

3.6.1 Effect ontensile strength

A detailed investigation was carried out to study the mechanical properties of banana nanofiberincreasing weight fraction. Tensile test was conducted for different percentage weights of neatcomposite,2, 4, 6 and 8 wt. %banana nanofiberreinforced composites. From Fig.3.9 it is observed that the tensile strength was improved with nanofiber reinforcement in an epoxy matrix. The tensile strength of neat epoxy composite is 76.54 MPa. The tensile strength reaches a maximum value of 170.65 MPa at 4 wt. %bananananofiber, showing an improvement of 96%. Later the tensile strength was found to decrease by a further reinforcement of 6 wt. %banana nanofibercontent. The influence of the nanofibers especially in the composite strength indicates that nanofibers have good mechanical properties and that there is an interaction between the epoxy resin and bananananofibers.

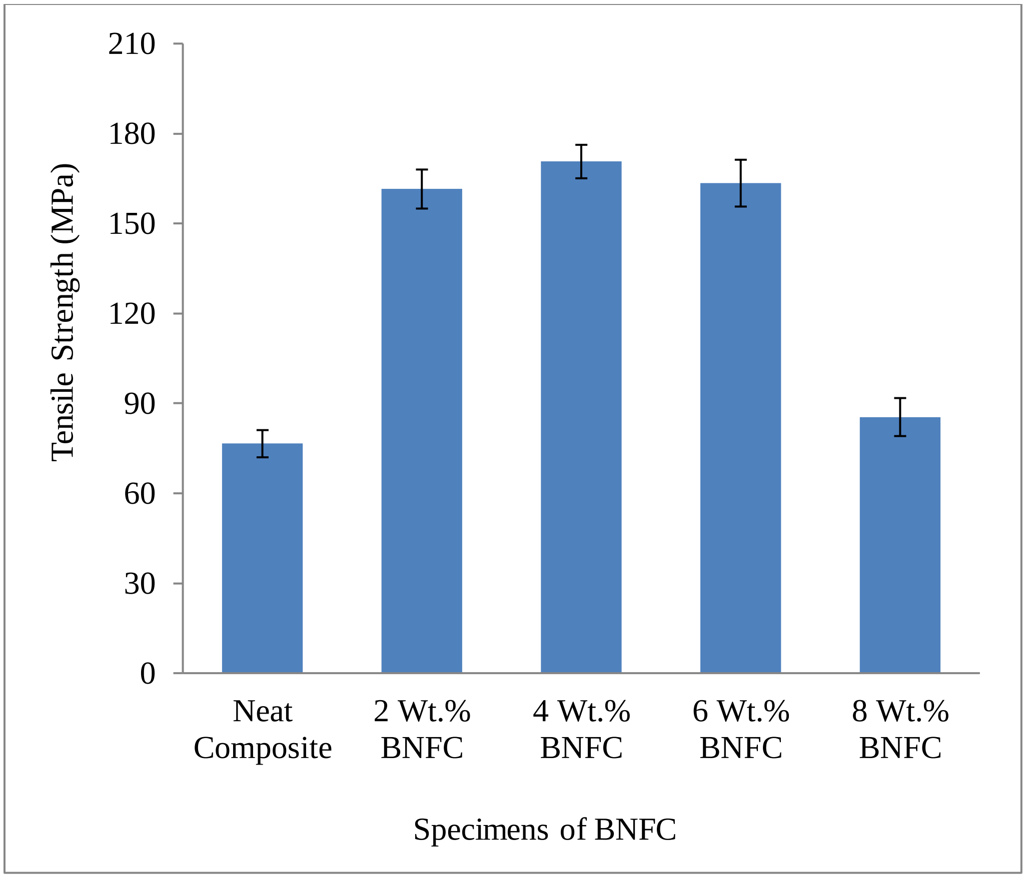

Fig.3.9Neat composite and various percentages of banana fiber nanocomposites on tensile strength

The improvement indicates that the filler is properly dispersed in the matrix structure. On the other hand, the deformation at break decreases as the banana nanofiberconcentration increases after6wt.% banana nanofibercomposites. The reason for the decrease may due to the banana nanofiberaggregated in the matrix, causing a poor interface between the nanofiber and epoxy resin [16 and 17].Several other possible reasons are responsible for the decrease in tensile strength. One of the flaws existing in the nanocomposites is the weak boundaries between particles and the bubbles trapped during the sample preparation. Another possible reason is the non-homogeneous network density of the samples. During the preparation of nanocomposites, the viscosity of the epoxy and the banana nanofibermixture was very high, probably due to the dramatically increased interfacial area and interaction between the highly exfoliated banana nanofiberand epoxy resin. Similar results have been observed by the other researchers.The stress transfer to the reinforcement phase is facilitated by the tremendous coupling between the surface area of the bananananofibers and the polymer matrix which improves the tensile properties of nanocomposites.

Fracture is a very important concept which plays a big role of laminated materials. It is regarded as a process in which an increase in loading suddenly causes accelerated growth of pre-existing cracks.The fracture surfaces of the neat epoxy and nanofiber reinforced polymer composites with different weight percentages have been studied. The addition of nanofibers in a polymermay result may affect.If the bananananofibers are randomly dispersed in the matrix, then the structure is called an exfoliated structure.The fractured surface of neat epoxy and nanocomposite specimen under a tensile test was investigated using the SEM process.

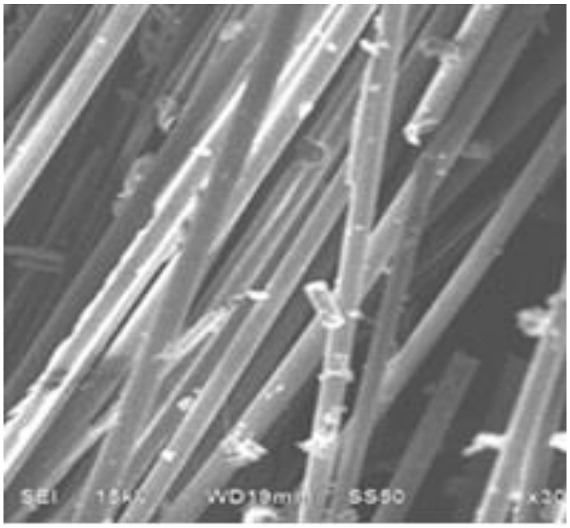

Fig.3.10SEM micrograph of fractured surface neatcomposite

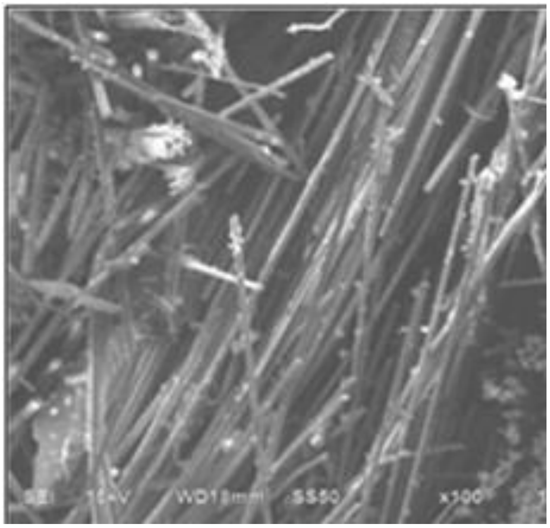

Fig.3.11SEM micrograph of fractured surface of 2wt.% banana nanofiber composite

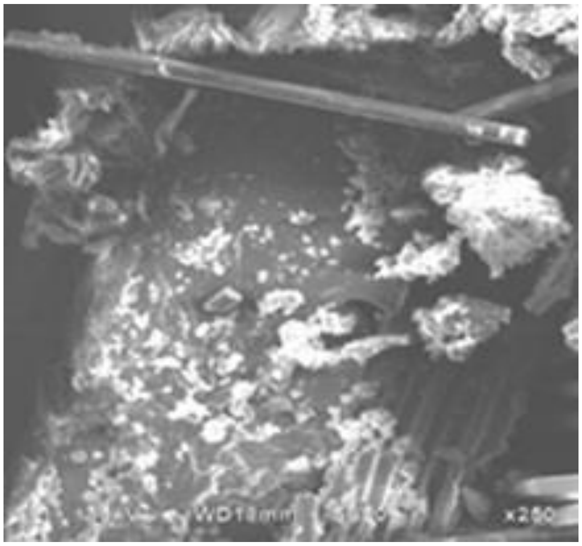

Fig.3.12SEM micrograph of fractured surface of 4wt.% banana nanofiber composite

Fig.3.13SEM micrograph of fractured surface of 6wt.% banana nanofiber composite

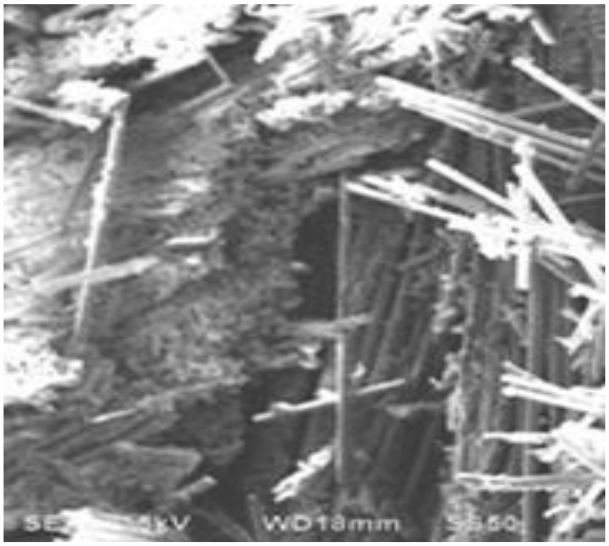

Fig.3.14SEM micrograph of fractured surface of 8wt.% banana nanofiber composite

The Fig.3.14 shows the SEM images of the fractured surface of neat epoxy composite with a smooth fracture surface. This failure mechanism is characteristic of a brittle material suggesting that the material had undergone a reduced deformation under failure. The fracture surface of nanofiber composites shows irregular, forced cracks. This result further suggests that the structural change in the nanofiber composite was excited at the amount of nanofiber increased in the neat epoxy composite. Fig.4.18 shows the fracture structure of 2wt.% banana nanofiber composite which indicates that the nanofiber is poorly dispersed and forms aggregates. Which are exposed on the surface because the cracks penetrated.

The fracture structure of 4wt.% banana nanofiber composite has high stress zones which indicate the increased reinforcement of the polymer matrix and good dispersion of nanofibers in composites as shown Fig.3.12. This high density, grain boundary shows a strengthened matrix. This structural phenomenon may be linked to the maximum improvement of mechanical properties. Fig.3.13, 3.14 shows that the fracture structure of 6 and 8wt.% banana nanofiber filler composites have a large agglomeration which indicates that the interface interactions of polymer molecules cause poor interfacial adhesion leading to a reduction in mechanical properties.

3.6.2 Effect onimpact strength

The impact behavior of e-glass fibers reinforced polyester of various banana nanofiber laminated composites was experimentally investigated using notched Izod impact test specimen. The impact property of a composite material is its capacity to absorb and dissipate energies under impact or shock loading. The neat epoxy resin has very low impact resistance reinforcement with the Banananananofibers increasing its impact strength significantly. The impact energy level of the composite depends upon several factors such as the nature of the composite, geometry, Fiber arrangement and Fiber matrix interface. The matrix de-bonding and Fiber pullout are important failure modes observed in the Fiber composites due to impact loading.The load is transferred through shear and when the shear force exceeds the Fiber matrix interaction force, the Fiber matrix de-bonding takes place. The Fiber fracture will be pre-dominating when the stress level exceeds the Fiber stress and then the fractured Fibers are pulled out of the matrix. The fracture energy recorded in an impact test reflects the integrative force of the sample over the range of sample deflection.

Fig.3.15shows the improvement of the impact strength of nano composites compared to the neat epoxy composite. The impact strength of neat composite is 2.72J.

The impact strength reaches maximum value of 3.63Jof 6wt.% banana nanofiber, thus showing an improvement of 33.45%. Later, the impact strength was found to decrease by a further reinforcement of 8wt. % and abovebanana nanofiber content. The decrease in impact strength at high fiber content reinforcement is due to the probability of banana nanofiberagglomeration which results in regions of stress concentration that requires lesser energy for crack propagation.

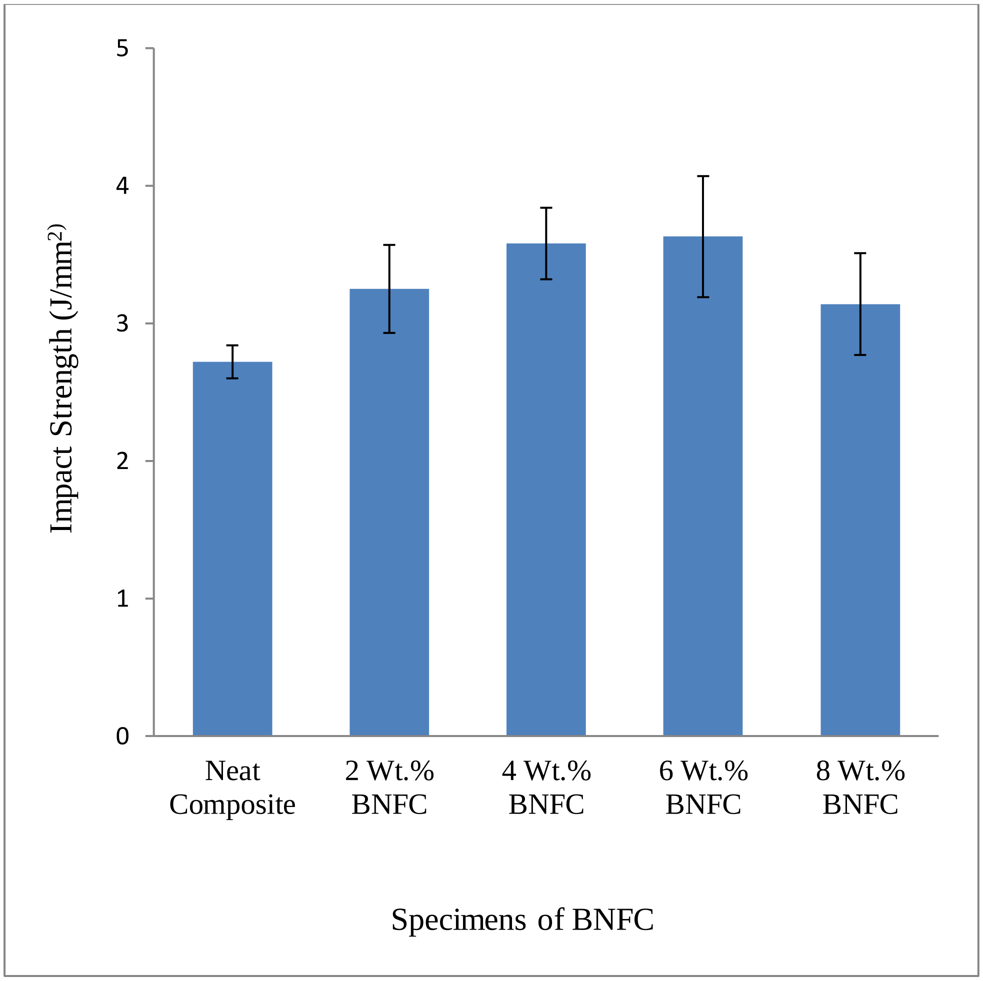

Fig.3.15NeatComposite and various percentages of bananafiber nanocomposites on impact strength

3.6.3 Effect on flexural strength

Flexural strength and stiffness are not basic material properties. They are the combined effects of a material’s basic tensile, compressive and shear properties. That is, when a flexural loading is applied to a specimen, all three of the material's basic stress states are induced. Material failure, then, is dictated by which of the three basic stresses is the first to reach its limiting value. The specimen is loaded while in a horizontal position, and in such a way that the compressive stress occurs in the upper portion and the tensile stress occurs in the lower portion of the cross section. If the specimen is symmetrical about the midplane of its cross section.Fig.3.16 shows the improvement of the strength of nano composites compared to neatcomposite. The neatcomposite is 112.56 MPaand maximum value of 172.89 MPaof 4wt.%banana nanofiber, thus showing an improvement of 53.59% Later, the strength was found to decrease by a further reinforcement of 6 wt.% and abovebanana nanofiber content.

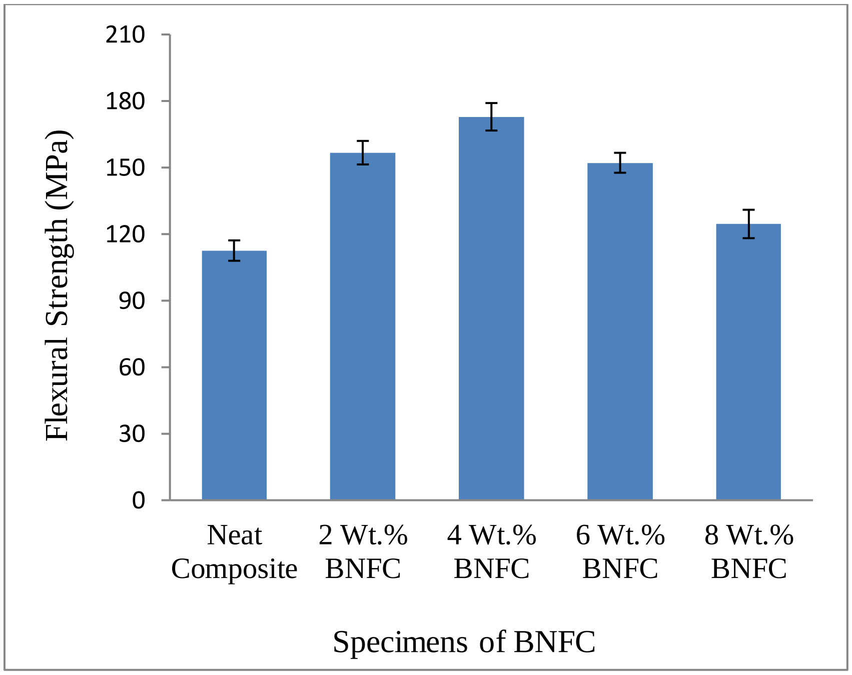

Fig.3.16Neatcomposite and various percentages of banana fiber nanocomposites on flexural strength

3.6.4 Effect on Hardness

The hardness of an FRP composite is a direct result of the resin matrix and how it is cured. The more rigid the resin the higher the level of hardness, whereas the more flexible laminate will have a lower hardness level. As a resin matrix cures and cross-links, the hardness value continue to rise. When the resin fully cures, the hardness properties will be of the highest value.The hardness of the banana nanofiber composite was tested by the Barcoll Hardness tester. The hardness of the nanocomposites is not much different by the reinforcement of nanofibers in an epoxy matrix. but a little improvement has been observed in the hardness of the nanocomposite. The Barcoll hardness of the nano composites increases up to 88,when the nanofiber is reinforced in polymer composites. The maximum improvement in hardness is obtained by 7.5% in 6 wt.% in banana nanofiber composites as shown in Fig.3.17.This is because of the strong interfacial bonding strength between the Fiber and matrix which greatly increases the hardness of nanofiber reinforced composites by curing. Due to an increase in the bananananofibers content the composite becomes stiffer and harder,and thus there is an increase in hardness. The composite decreases form8wt.%banana nanofiber composites.

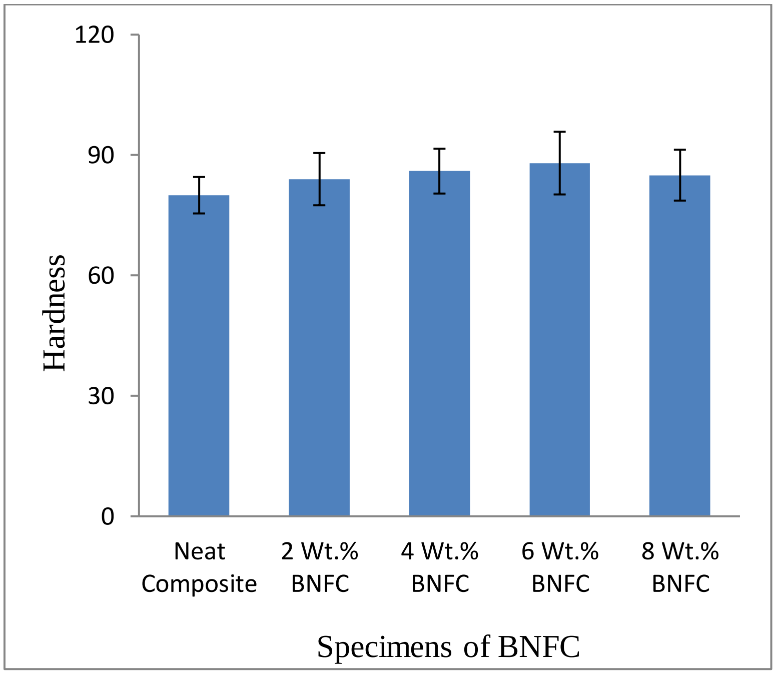

Fig.3.17NeatComposite and various percentages of banana fiber nanocomposites on hardness

3.6.5 Effect on damping

The internaldamping results from mechanical energy dissipation within the material due to various microscopic and macroscopic processes. Internal damping of materials orginates from the energy dissipation associated with microstructure defects such as grain boundaries and impurities, thermo-elastic effects caused by local temperature gradients, non-uniform stress as in vibrating beams, eddy current effects in ferromagnetic materials, dislocation motion in metals and chain motion in polymers. It is observed that material damping is the energy dissipated within the materials of construction and is due to internal hysteresis in materials arising from a non-linear stress-strain behaviour, inter ganular friction and thermo-elasticityimprovement in the natural frequency as noticed for both modes of nanofiber composites. Table 4.4 (APPENDIX -I) represents the experimental values obtained. Neat composite, 2,4,6 and 8 wt.% of banana nanofiberare compared.

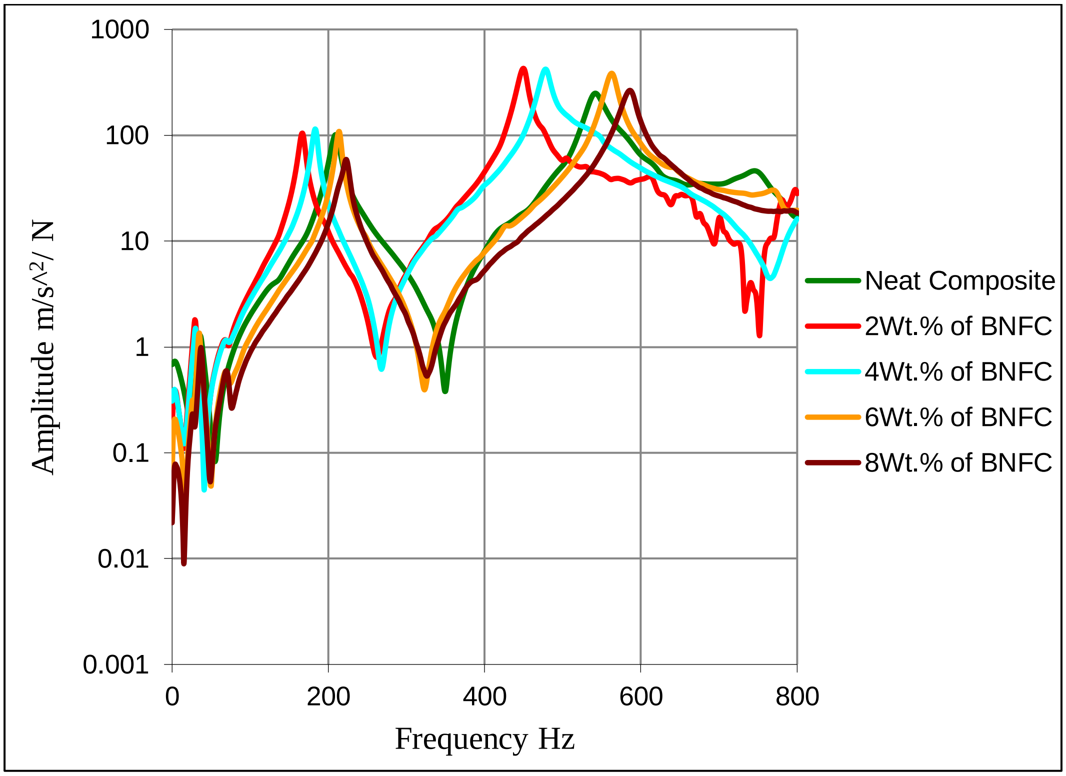

Fig.3.18Amplitude Vs natural frequency for neat composite and bananananofiber composite

Fig.3.18 shows the improvement in the natural frequency as noticed for both modes of nanofiber composites. The increased natural frequency is due to the good dispersion of banana nanoFiber reinforcement in the matrix which results in improved stiffness. It may be noted that the addition of banana nanofiber has a considerable effect on the damping behaviour of nanocomposites.From the experimental data mentioned in the Table 3.4, an improvement in natural frequency is noticed in the banana nanofibers reinforced composites when compared with pure epoxy composite. The neat composite has a damping ratio of 0.019 with a natural frequency 112 Hz. It was observed that the damping ratio was 0.036 and increased by 89.47% from4wt.% banana nanofiber composite when compared to neat composite.

Table 3.4 Natural frequencies and damping ratios of nanocomposites

Composite Specimens	**Modes**	**Natural frequency (Hz)**	**Damping ratio**
Neat Composite	2	112	0.019
2wt.% BNFC	2	172	0.032
4wt.% BNFC	2	186	0.036
6wt.% BNFC	2	218	0.028
8wt.% BNFC	2	208	0.021

This may be due to the composites possessing high stiffness on account of the high modulus of banana nanofibers and its uniform distribution. The banana nanofibers are uniformly distributed, which results in a good bonding between the reinforcement and the matrix. This provides a large interfacial area between the matrix and banana nanofibers. This increases the modulus value as well as the energy dissipating interface. The nanofiber composites with a high concentration above 4wt.% banana nano composite show a decrease in damping ratio. Clustering of banana nanofibers at a higher weight percentage causes a decrease in its damping ratio.

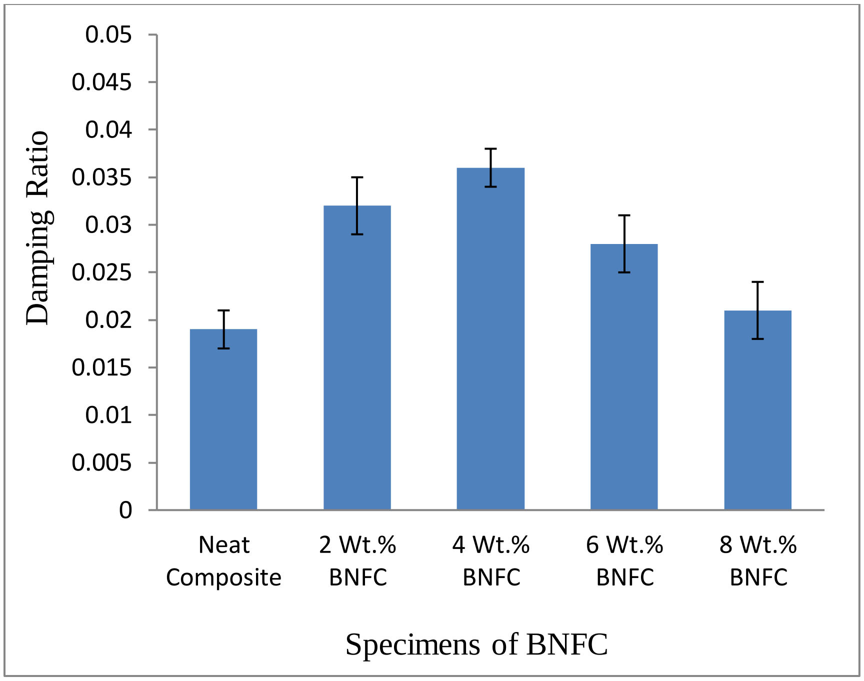

Fig. 3.19Effect of damping ratio of the neat composite and banana nanofibercomposites

Fig.3.19 shows the variation of damping ratio for different amounts of banana nanofiber in nanocomposites.when compared with neat composite The increased natural frequency is due to the good dispersion of banana nanofibers in the epoxy matrix which results in improved stiffness. For higher velocities the damping increases due to low contact time and dissipated low vibration energy in the laminate.The addition of banana nanofiber provides a good damping effect.

3.8 SUMMARY

It is found that alkali treatment plays a significant role in improving the mechanical properties and less moisture absorption rate of the nanocomposite specimens. The optimum alkali percentage was obtained as 3%, as further increasing the alkali concentration results in surface damage, observed from the scanning electron microscope images.This indicates that the alkali treatment is a simple and efficient way of improving the mechanical properties.

CHAPTER–4

THERMAL PROPERTIES OF BANANA FIBER POLYMER NANOCOMPOSITE

4.0 INTRODUCTION

Thermal analysis is used for measuring different types of transitions effect of fillers, additives, and the influence of production conditions. The thermal property of a composite plays a vital role in evaluating product performance as well as predicting the processibility characteristics in polymers for specific applications. Thermal methods are based upon the measurement of the dynamic relationship between temperature and some property of the system such as mass and heat absorbed. The influence of nanofibers reinforcement composites are studied by thermal gravimetric analysis, differential scanning calorimetry and dynamic mechanical analysis was conducted for banana fiber nanocomposites to study thermal behaviors of polymer composites.

4.1 THERMOGRAVIMETRIC ANALYSIS

Thermogravimetric curves help to provide information regarding polymerization reactions, the efficiencies of stabilizers and activators, the thermal stability of final materials and direct analysis, TGA provides a general technique to determine the amount of highly volatile matter, medium volatile matter, combustible material and the ash content of compounds. It measures the rate of change in the mass of a sample as a function of temperature and time in a controlled atmosphere. The TGA measurements are used primarily to determine the thermal and oxidative stabilities of materials as well as their compositional properties.

The technique can analyze materials that exhibit either mass loss or gain due to decomposition. The thermal degradation behavior of neat composite, 2, 4, 6 and 8wt. % banana fiber polymer nanocomposites have been carried out using the TGA Q50 instrument in a temperature range of 30°C – 800°C with heating rate 0.01°C/min. Thermal transition changes are plotted in the Fig.4.1. The experimental values are represented in Table 5.1 shown in Appendix-I. The varying weight loss and temperature of the nanocomposites. All the specimens show an initial weight loss of about 20-30% in the region of 75-150°C caused by the evaporation of water. The second degradation region is located between 250°C and 400°C and is due to the pyrolysis of banana nanofibers and degradation of composites. The weight loss being 50%–60% of all composites, the second degradation mainly involves dehydration reactions and the formation of volatile products [19]. The third stage of weight loss occurs above 400°C and comprises the decomposition of carbonaceous matter of composites, which is a similar type of results.

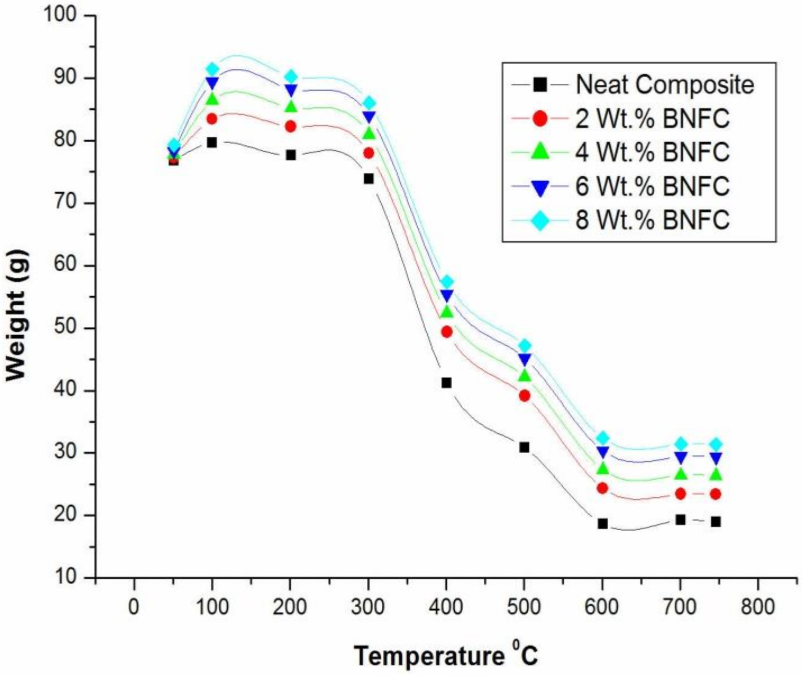

Fig.4.1 TGA curves of different percentages of banana fibers nanocomposites

A sudden drop in the mass of the sample indicates the thermal degradation of the materials. However the addition of banana nanofibers to the epoxy matrix increases the degradation temperature onset in the composites and also increases the decomposition temperature. The major reason of the thermal stability improvement is due to the fact that highly cross linked multilayered epoxy matrix which produces additional intermolecular bonding between fibers and matrix, allowing more thermal energy to be distributed over these bonds within the interface.

The thermal degradation of neat composite started at 332°C and 100% decomposition was noticed at 533°C. However, with the incorporation of banana nanofibers, there was a substantial enhancement in the thermal stability of the nanocomposites with an initial degradation temperature [20]. The rate of degradation was found to be lower in case of 8wt. % in banana fiber polymer nanocomposites. This indicates that a significant increase in the nanofibers content of composite plays an important role in controlling its rate of thermal degradation. [21-22]. The nanofibers reinforcement in between the matrix resin molecules offered some resistance towards their thermal degradation and showed a lowering trend with the increase in nanofibers content.

Thermogravimetric curves are given for specific materials and chemical compounds due to a unique sequence of physicochemical reactions occurring over specific temperature ranges and heating rates. Measurements are usually carried out in an inert atmosphere, and the weight change is recorded as a function of increasing temperature, as the Fig.4.2 plot indicate thermal degradation of the neat composite is 332°C where as the nanofibers reinforcement improves the thermal degradation temperature of the composites this gives a good indication of thermal resistance of fibers nanocomposites. Thermal degradation of polymers is molecular deterioration as a result of overheating. At increasing temperatures the components of the long chain backbone of the polymer can begin to separate and react with one another to change the properties of the polymer. The chemical reactions involved in thermal degradation lead to change the initially specified properties.

Thermal decomposition is a chemical reaction in which heat breaks a compound down into two or more products. As heating rate is increased, the thermal decomposition temperature increased and the point where the weight loss suddenly increased [24]. The above Fig.4.3 shows the thermal decomposition of the Neat composites 544°C and all the fibers nanocomposites specimens are higher decomposition temperature, it means that the thermal stability of the nanocomposites are higher. The activation energy increased due to the reinforcement effect of banana nanofibers. The decomposition temperatures drastically decrease with increasing temperature. The experimental values are represented in Table 4.2 (Appendix- I).

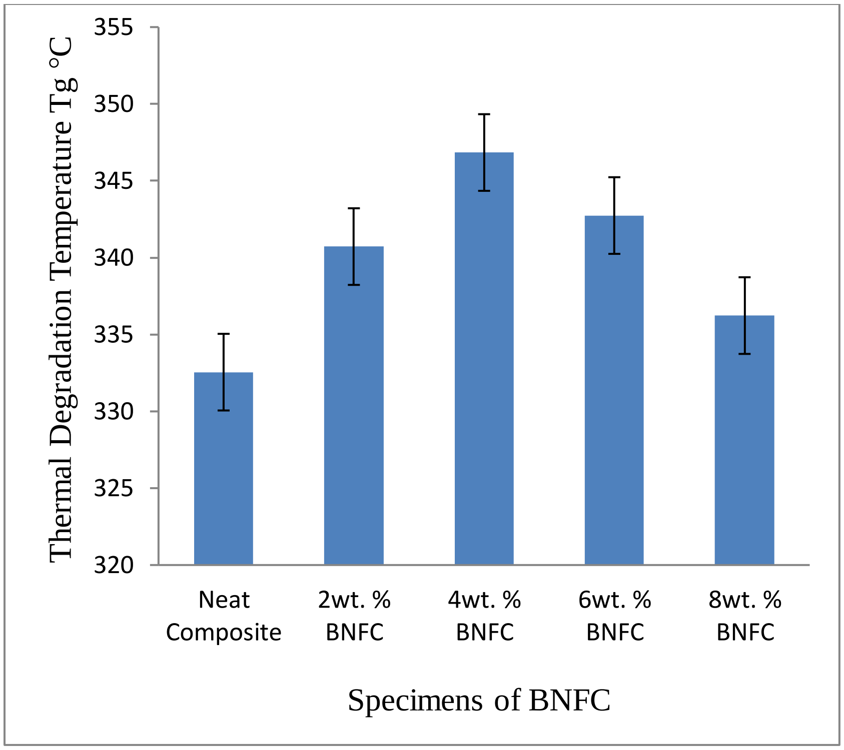

Fig.4.2 Thermal degradation temperature of the different weight percentage of banana fibers nanocomposites

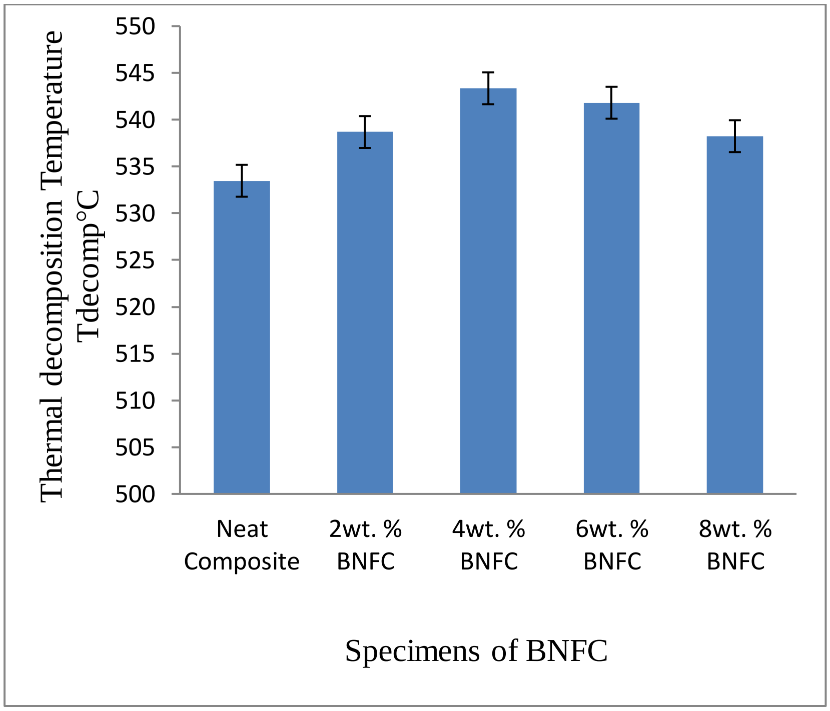

Fig.4.3 Thermal decompostion temperature of the different weight percentage of banana fibers nanocomposites

5.2 DIFFERENTIAL SCANNING CALORIMETRY

Differential scanning calorimetry is used to study the polymers specimens upon heating, measure heat absorbed or liberated during heating or cooling. DSC monitors the heat effects associated with phase transitions and chemical reactions as a function of temperature. Chemical reactions and many physical changes, such as melting, result in the absorption or release of heat energy as they occur. Energy is given out to the surroundings during a reaction is called exothermic reactions. Energy is taken in from the surroundings, the absorption of heat by a material is called an endothermic reaction. Measuring the occurrence of these exotherms and endotherms, possible to determine the state changes in the specimen. The mechanical behavior of polymers changes in the glass transition temperature and is an important characteristic of every polymer composite.

The thermal degradation begins to occur only after the materials have absorbed certain amounts of heat energy. The heat initiates the degradation processes and the breaking down of the fibers and matrix structures by causing molecular chain ruptures [15]. DSC curves are used to obtain thermal information such as the crystallization temperature and melt temperature. The DSC curves of banana nanocomposites with neat composite, 2, 4, 6 and 8wt. % were studied in the temperature range of 30° to 300°C to determine thermal transitions. Fig.4.4 shows the heat flow vs. temperature plots of neat composite and banana fibers nanocomposites which show the large thermal changes. The experimental values are represented in Table 4.3 (Appendix- I).

In the entire case, the nanocomposites show fewer changes in thermal behavior than Neat composite. This may be due to moisture in the sample. The incorporation of nanofibers affects the crystallization rate and hence the crystallization peak shifts lower in the thermogram with increasing nanofibers content the addition of nanofibers reinforcement alters the overall melting behavior of the nanocomposites. This broadening of the melting point suggests that the crystals formed in the presence of banana nanofibers are more heterogeneous than those in the neat composite.

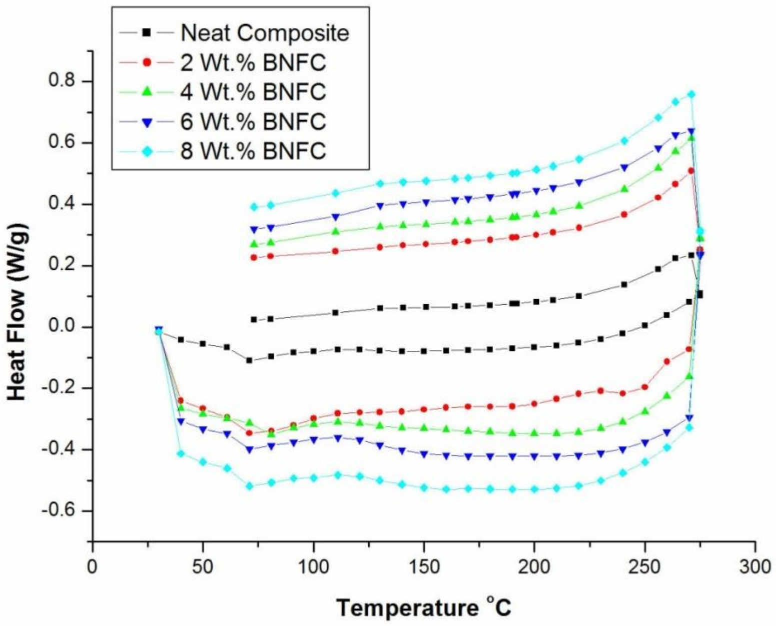

Fig.4.4 DSC Heat curves of the different weight percentage of banana fibers nanocomposites

It is observed that the crystallization temperature T_c of the neat composite is 83°C and the melting temperature is 131°C. The addition of banana nanofibers affects the crystallization behavior of the polymer composite. The crystallization temperature of banana fibers nanocomposites is in the range of 83° C to 88°C. This result indicates that the nucleating effect of banana fibers nanocomposites was strengthened due to stronger interaction between the banana nanofibers surface and chains [26]. The nucleating effect of banana nanofibers could also explain the increase in crystallinity. The nano scale dispersion of the filler and its orientation in the matrix are among these factors.

The fibers nanocomposites have an impact on crystallization temperature, the nanofibers accelerates the crystallization and depends upon the weight percentage of nanofibers reinforcement. Fig.4.5 shows that a maximum crystallization temperature was observed in 4wt. % banana fiber polymer nanocomposite, beyond that the T_c decreased for 6wt. % and 8wt. % of Specimens. The DSC can measure the thermo systems such as epoxy based on identifying energy changes associated with a change in the relationship between crystalline and amorphous in the polymer.

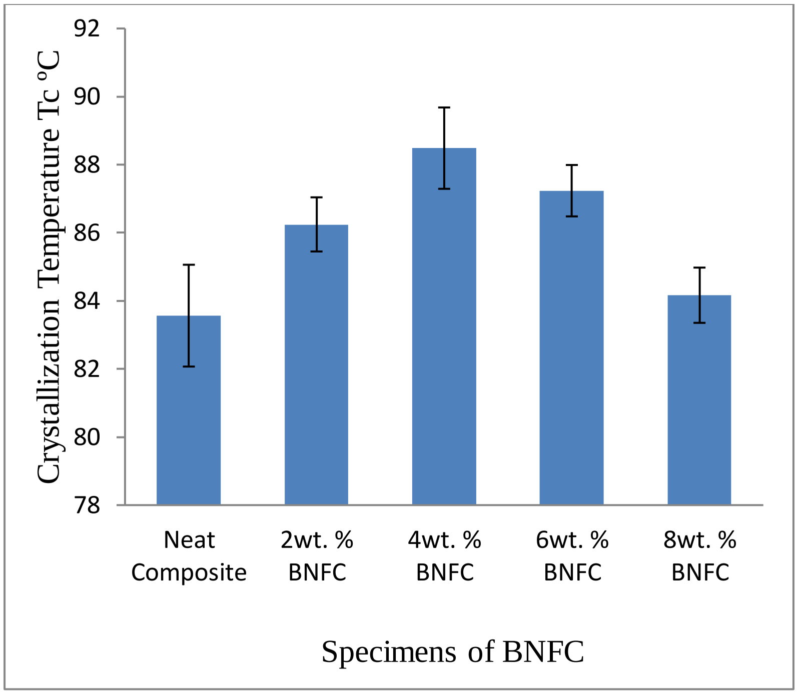

Fig.4.5 Crystallisation temperature of the different weight percentage of banana fibers nanocomposites

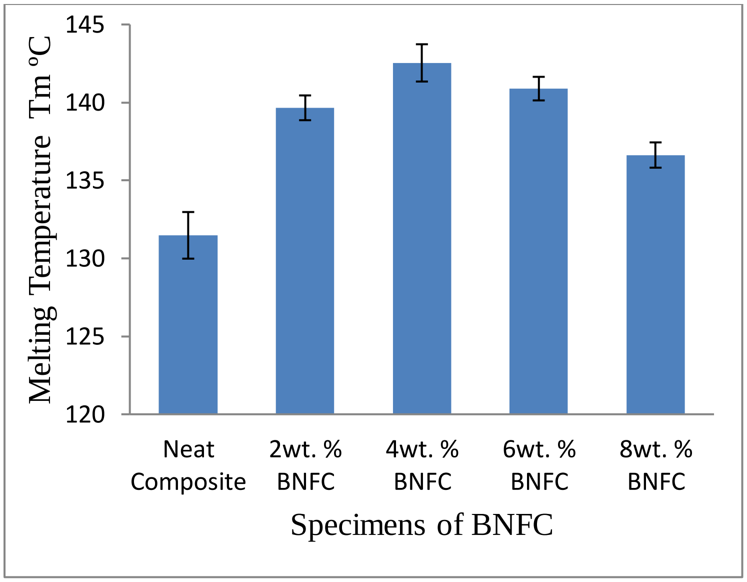

Fig.4.6 Melting point temperature of the different weight percentage of banana fibers nanocomposites

The melting point temperature of the fibers nanocomposites was in the range of 131°C to 144°C as shown in Fig.4.6. Melting temperature of the fibers nanocomposites was improved when compare with neat composites. Maximum melting point temperature was observed in 4wt. % banana fiber polymer nanocomposite, beyond that the T_m decreased for 6wt. % and 8wt. % banana fiber polymer nanocomposites. It means that melting point broading suggests that the crystal formed in the presence of banana nanofibers increases more heterogeneous than the case of neat composite nanofibers reinforcement increases the crystallisation temperature and melting point temperature by the reinforcement of banana fibers nanocomposites. The experimental values are represented in Table 4.4 (Appendix- I).

5.3 DYNAMIC MECHANICAL ANALYSIS

Dynamic mechanical analysis is an important tool in interpreting the interface region in the composite, which reflects the nature of interfacial bonding between the fiber and the matrix and has the most significant contribution to the performance of the composites [27]. Dynamic mechanical analysis is a thermal analysis technique used to measure changes in the viscoelastic response of a composite material as a function of temperature, time or deformation frequency by subjecting it to a small oscillating force. In DMA a sample is deformed mechanically and its response is measured. The deformation can be applied sinusoidal, in a constant fashion, or at a fixed rate. The response to the deformation can be monitored as a function of temperature. DMA determines elastic modulus, loss modulus and damping coefficient as a function of temperature frequency or time.

The effect of banana nanofibers reinforcement with polymer. The influence of percentage weight of neat composite, 2, 4, 6 and 8wt. % nanofibers reinforcement has been studied by dynamic mechanical analysis. The neat composite and nanofibers composite samples were subjected to dynamic mechanical analysis in order to understand the banana nanofiber interaction in the composite with the increase in nanofibers. The dynamic mechanical results expressed as storage modulus, loss modulus and loss tangent as a function of temperature and resin composition show the changes in a composite material, with increasing temperature.

5.3.1 Storage modulus, loss modulus and loss tangent

The storage modulus E´ represents the stiffness of a viscoelastic material and is proportional to the energy stored during a loading cycle. Storage modulus is a function of temperature of the composites. The banana fibers nanocomposites with different weight percentages were tested from the three point bending method and the storage modulus comparison graphs were shown in Fig.4.7 and the experimental values are represented in Table 4.5(a) in Appendix- I. It is observed that the nanocomposites show a slightly higher storage modulus with the addition of nanofibers. The composite samples dropped drastically between 65 – 80°C which was their glass transition region. The glass transition temperature of neat composite has been found to be 65°C, whereas the transition temperature of banana fibers nanocomposites was at 70 – 75°C. The storage modulus of banana nanofibers reinforced composite increases with an increased fiber content in the glassy as well as rubbery region.

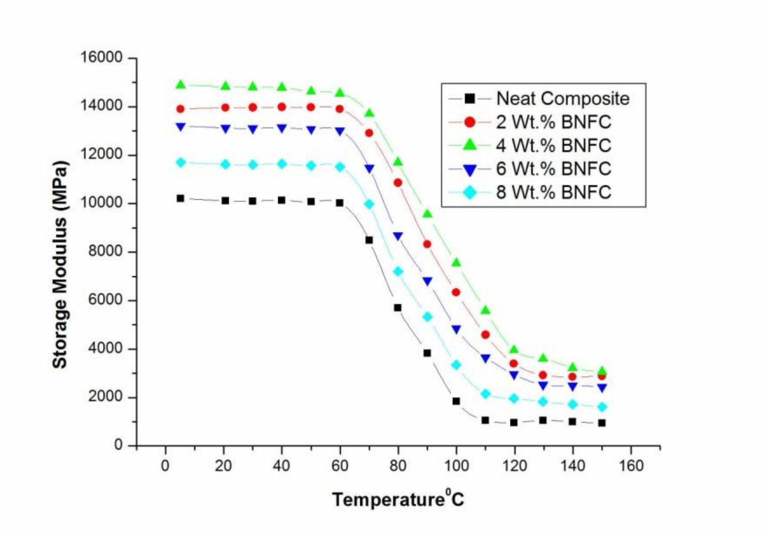

Fig.4.7 Storage Modulus vs Temperature of the different weight percentage of banana fibers nanocomposites

This observation is most likely related to the interaction between the hydroxyl group of epoxy and hydroxyl groups that are known to exist on damaged sites on the banana nanofibers surface or as a result of atomic scale defects formed. The increase in glass transition also suggests that the degree of interaction between the polymer chains and the surface of the banana nanofibers increase. It might be implied that the nano filler increases the glass transition temperature of the soft-phase by restricting the segmented chain motion and generating the inter phase region [27]. The storage modulus increases in the presence of the banana nanofibers which could be concluded as a combined effect of the nanofibers embedded in a viscoelastic matrix and the mechanical limitations. At high concentrations the fibers reduce the mobility and deformation of the matrix, and in this case, the stress can be transferred from the epoxy matrix to the banana nanofibers reinforcement.

Loss modulus E´´ is defined as being proportional to the energy dissipated during one loading cycle. Energy lost as heat, and is a measure of vibrational energy that has been converted during vibration and that cannot be recovered. The loss modulus value which reflects the amount of viscous dissipation in the composites revealed some interesting behavior. The loss modulus is a function of temperature for Banana fiber nanocomposites specimens. The comparison between the loss modulus vs temperature plots of the Neat composite with nanocomposite specimens Fig.4.8 shows. The experimental values are represented in Table 4.5 (b) in Appendix - I. The T_g of the Banana fibers nanocomposites are slightly shifted to a higher temperature with a broader range of the transition region than the pure epoxy composite sample.

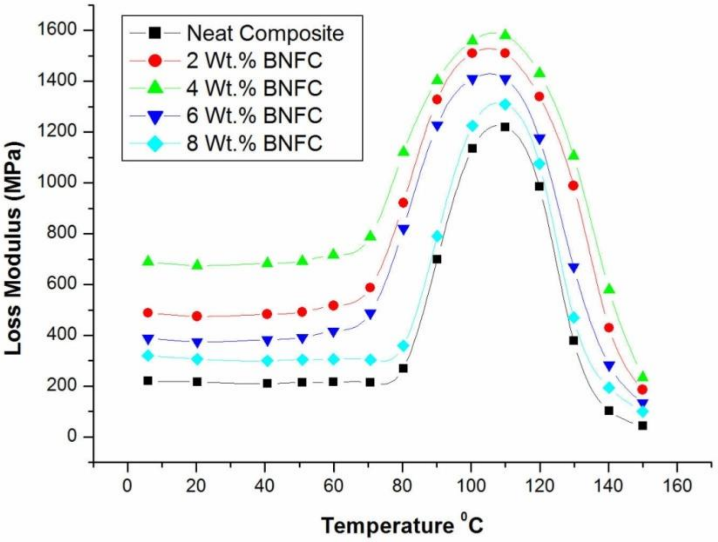

Fig.4.8 Loss modulus vs Temperature of the different weight percentage of banana fibers nanocomposites

The loss factors are sensitive to molecular motions it could mean that the mobility of the polymer molecular chains decreases as the chains were hindered by the nanofibers reinforcement leading to shift of T_g. This might be a trend that as banana nanofiberss weight content increased the mobility of the polymer chain decreased by T_g increased subsequently. Good adhesion of banana nanofibers with the surrounding polymer matrix would additionally benefit the dynamic modulus by hindering the molecular motion to some extent. The loss modulus increases with an increase in the percentage of banana nanocomposite. This may be due to energy losses caused by the rearrangements of the molecules and banana nanofibers as well as the internal friction between the banana nanofibers and the polymer matrix.

The loss factor tan δ is the ratio of the loss modulus to storage modulus. It is a measure of the energy lost, expressed in terms of the recoverable energy, and represents mechanical damping or internal friction in a viscoelastic system. The loss factor tan δ is expressed as a dimensionless number. A high tan δ value is indicative of a material that has a high, nonelastic strain component, while a low value indicates one that is more elastic. The tan δ curve is a popular measurement point for the T_g and it is usually easier to isolate than to determine the onset of the drop in the storage modulus. The tan δ verses temperature curve can be used to determine much more about a system than just its T_g. The width of the tan δ peak can indicate how homogeneous a system. A system has very broad peaks which are generally composed of different polymer chain lengths or structures which gives rise to a boarder temperature range, that initiate significant viscous chain motions for the various components. On the other hand, systems with narrow peaks generally have a more narrow distribution of chain types and molecular weights.

The height or amplitude of the tan δ curve is directly related to a material's ability to dissipate energy through segmental motion. Systems with tan δ peaks have higher ratios of energy absorbing viscous motions and are therefore generally tougher systems than those with lower tan δ amplitudes. It was observed that the intensity of tan δ peak decreased with an increased in banana nanofibers reinforcement as shown Fig.5.9. The maximum damping parameter was observed as 0.220 of 8wt. % banana fiber polymer nanocomposite and its improvement is 36%.This might be due to the decrement of the mobility of polymer molecular chains as hindered by reinforcement which led to a reduction of height and sharpness of the peak in the curves as shown in Fig. 4.9. The experimental values are represented in Table 4.5 (c) in Appendix - I.

The increase in modulus together with a positive shift in tan δ peak position can be attributed to the physical interaction between the polymer and reinforcements that restrict the segmental mobility of the polymer chain [28]. The decline in the elastic properties combined with the increase in damping at higher temperatures is attributed to the damage of the polymer chain structure. In addition, damping in the transition region measured imperfectly in the elasticity and some of the energy used to deform the material was directly dissipated into heat. Thus, the mechanical laws that overcomes the friction of the inter molecular chain was reduced with banana nanofibers additives. It was also indicated in other literature that the reduction in tan δ denotes the improvement in the hysteresis of the system as a reduction in internal friction.

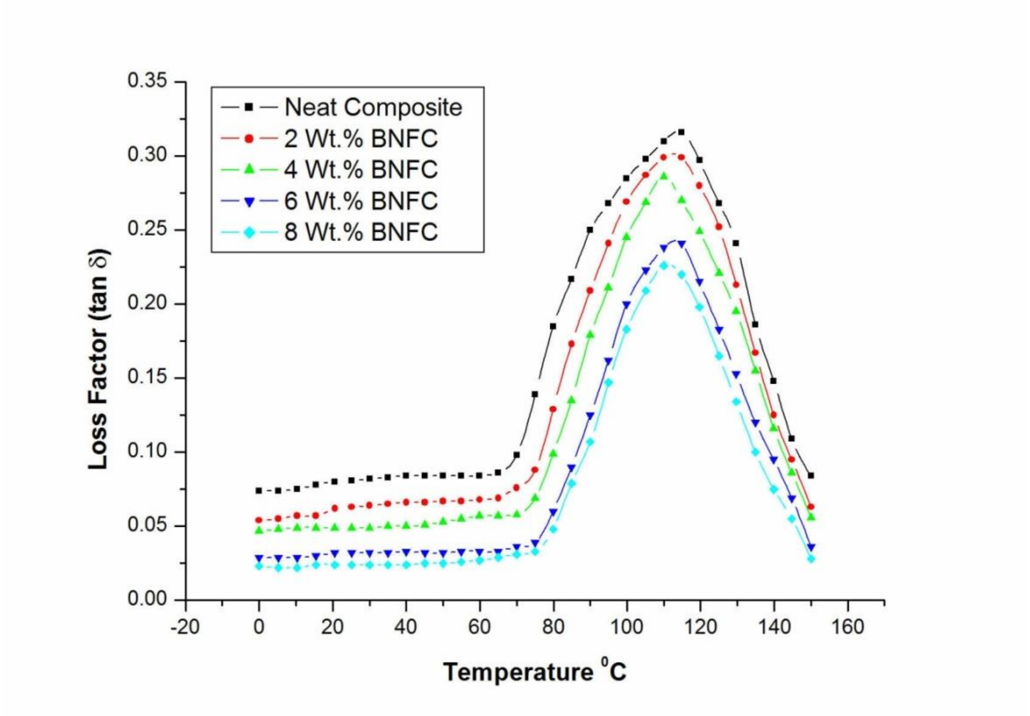

Fig.4.9 Loss tan δ vs Temperature of the different weight percentage of banana fibers nanocomposites

4.4 SUMMARY

In this chapter the thermal properties were analyzed by TGA, DSC and DMA. The incorporation of banana nanofibers in epoxy polymer improves the thermal stability of the composites. TGA curves indicate that the decomposition temperature slightly increases with the addition of nanofibers reinforcement. The glass transition temperature and the melting temperature also increased with the addition of nanofibers in the epoxy polymer composite. nanocomposites reduce the rate of heat release, even when as adding of banana nano fiber is present. There is no linear relationship between the amount of nano banana fiber and the rate of heat release, or with any other calorimetric property. The substituents on the nano fiber can interact with the polymer, have some effect on the compatibility between the polymer and the banana nano fiber reinforcement, but there apparently effects on the thermal stability as measured by the TGA. Thermal degradation behavior was studied in detail by heating rate mode of high resolution TGA. The activation energy values of samples in air were smaller than that in nitrogen, indicating that the thermal degradation mechanism.

This DSC curve indicates that the nucleating effect of banana fibers nanocomposites was strengthened. The nanofibers played the role of a nucleating agent and facilitated crystallization. This is due to a stronger interaction between banana nanofibers surface and chains. The nano scale dispersion of the filler and the matrix are among these factors. All fibers nanocomposites had a higher melting temperature compared with Neat composite. Investigations on polymer crystallization behavior and crystal morphology were carried out on nanocomposites formed by melt processing. These studies shows that the crystallization of the nanocomposites is dramatically increased by filler concentrations. In general, crystallization rates are enhanced more for nanocomposites formed from high molecular weight which provides more filler surface for crystal nucleation.

The dynamic properties of the polymer epoxy resins are temperature dependent. The storage modulus E' decreases with increasing temperature, while the loss modulus E" increases which should facilitate internal damping. The DMA study indicates that the increased modulus, together with the positive shift in tan delta peak position, is attributed to the physical interaction between the polymer and nanofibers that restrict the segmental mobility of the polymer chains.

DMA was found to be a very useful technique for interpreting some of the thermomechanical behavior of banana nano composites based on their viscoelasticity.

Three useful quantities obtained by dynamic testing, namely, storage modulus, loss modulus and tan δ were used to interpret the behavior and performance of the materials.
On the initial storage moduli of the specimens, the highest value, followed by the copolymer, respectively. The high modulus values of the copolymer material were due to their higher initial molecular weights. The high modulus value of the nanocomposite due to strong initial nano fiber/matrix interfacial bonding. The effect of frequency on the storage modulus of the blend, copolymer materials was studied. On comparing the peak tan δ values, the blend had the highest value followed by the copolymer. The tan δ value of the lowest of all the materials and this was probably due to the excessive crosslinking of the phase, resulting in a rigid interface that does not allow high-energy dissipation.

The increase in modulus with due to the increase in crosslinking of the polymer resin and the formation of stronger nanofiber/matrix interfacial bonds. The peak tan δ values and the area under the tan δ curves of the blend materials were found to decrease with the time. This was expected since molecular motions become more restricted because of cross linking and formation of stronger interracial bonds, resulting in a decrease in the energy that can be dissipated throughout the sample.

CHAPTER–5

REGRESSION ANALYSIS, EFFECT OF PROCESS PARAMETERS MECHANICAL AND THERMAL PROPERTIES NANOCOMPOSITES

5.0 INTRODUCTION

These statistical methods for data analysis is used to validate the experimental parameters by using regression analysis. It is used to ensure that the intended constructs can be justified, and to prevent the variables that do not represent the intended to measure are included in the final model. Regression analysis is used to test the established hypotheses. This chapter describes the importance of predicted models of regression results were compared with experimental values. From this, one of the developed equations which are useful for prediction of the trend pattern. In this direction regression analysis is a statistical tool for the investigation of relationships between variables. It includes many techniques for modeling and analyzing several variables, when the focus is on the relationship between a dependent variable and one or more independent variables. An attempt has been made to regression analysis and Analysis of variance (ANOVA) is used to check the validity of the model. The results indicated that the developed models are suitable for prediction of mechanical and thermal properties of banana nanofiber reinforced composite.

5.1 DATA ANALYSIS OF EXPERIMENTAL RESULTS

The data analysis is a model which describes the behavior of the system, statistical methods are used to developed model. It will first describe the chosen statistical methods, the variables that are used and then describe the data collection process. Regression analysis can be used to come up with a mathematical expression for the relationship between the two variables. The primary data was collected from respondents that were identified and exported directly Statistical package for the social science and analyzed.

5.1.1 Regression Analysis

A regression describes and evaluates the relationships between a given dependent variable and one or more independent variables. Earlier research focusing on similar subjects has found significant results using regression analysis. During the regression analysis, important assumptions for a valid regression will be elaborated and tested in order to ensure that the final regression models. However, it is also used to understand which, among the independent variables relate to the dependent variable.

The performance of regression analysis methods in practice depends on the form of the data generating process, and how it relates to the regression approach being used. In the present study the results obtained from the experimental data on mechanical properties, water absorption and Thermal properties are developed by using the regression analysis. The data required for regression analysis are generated by experimental data, the data set is used to build model and the other for validation. The data created is subjected testing and regression analysis is done by passing the input and output data as arguments to the program and the polynomial equations are generated. After the analysis is done the required ‘R-square’ value and coefficients are obtained. The predicted values are then back substituted for finding out error and the error percentage is then calculated.

5.2 EXPRESSION FOR POLYNOMIAL REGRESSION MODEL

The general equation for polynomial equation that can be written in the form of

$$ax^n + bx^{n-1} + \ldots + rx + s = 0, \qquad (5.1)$$

where a, b, . . . , r and s are constants.

The largest exponent of 'x' appearing in a non-zero term of a polynomial is considered as the degree of that polynomial.

5.2.1 Considerations in fitting of polynomial equation for mechanical properties

The polynomial models of second order can be used where the relationship between study and explanatory variables is curvilinear. It is always possible for a polynomial of order n-1 to pass through n points so that a polynomial of sufficiently high degree can always be found that provides a “good” fit to the data. Such models do not enhance the understanding of the unknown function. From the experimental data of the banana nanofibers composite of different weight percentage and their mechanical properties were formulated in polynomial equation by the regression model.

The equations obtained from the regression analysis for the mechanical properties are represented below.

Tensile Strength, $y_t = -6.113x^2 + 49.89x + 78.66$ (5.2)

Impact Strength, $y_i = -0.04321x^2 + 0.4017x + 2.684$ (5.3)

Flexural Strength, $y_f = -3.22x^2 + 26.74x + 114.1$ (5.4)

Hardness, $y_h = -0.2214x^2 + 2.551x + 79.87$ (5.5)

Damping ratio, $y_d = -0.02839x^2 + 0.1906x + 2.467$ (5.6)

5.2.2 Considerations in fitting of polynomial equation for thermal properties

This function fits a polynomial regression model to the powers of a single predictor by the method. Interpolation and calculation under the curve are also given. The thermal properties are analysed by using thermogaravometric analysis, differntial scanning calorimetry and dynamic mechanical analysis have been formulated in polynomial equation by regression models.

(a) Thermogaravometric Analysis

(i) Thermal degradation temperature (T_g) can be expressed as

$$y = -0.7068x^2 + 6.123x + 332.3 \quad (5.7)$$

(ii) Decomposition temperature (T_{decomp}) can be expressed as

$$y = -0.4248x^2 + 4.032x + 533.2 \quad (5.8)$$

(b) Differential Scanning Calorimetry

(i) Crystallization temperature (T_c) can be expressed as

$$y = -0.2666x^2 + 2.242x + 83.36 \quad (5.9)$$

(ii) Melting temperature (T_m) can be expressed as

$$y = -0.5891x^2 + 5.289x + 131.6 \quad (5.10)$$

(c) Dyanmic Mechanical Analysis

(i) Storage modulus (E') can be expressed as

$$y = -45.77x^2 + 419.7x + 569.9 \tag{5.11}$$

(ii) Loss modulus (E'') can be expressed as

$$y = -37.71x^2 + 374.6x + 785.3 \tag{5.12}$$

(iii) Tan deta (δ) can be expressed as

$$y = 0.000071x^2 - 0.01117x + 0.3138 \tag{5.13}$$

The mechanical properties such as tensile strength, impact strength, flexural strength, hardness and damping ratio has been predicted using the above equations and compared with the values obtained from the experiment test data. Thermal Properties like thermal degradation temperature and decomposition temperature from TGA, crystallization temperature, melting point temperature from DSC, and the dynamic behaviours like storage modulus, loss modulus and tan delta values has been predicted using the above equations and compared with the results obtained from the experiment test data. It is observed that the banana nanofibers reinforced composites with different weight percentages show good agreement with regression models, the remarks are found to be capable of predicting the percentage of error.

5.3 R, R^2, ADJUSTED R^2

'R' is a measure of the correlation between the experimental value and the predicted value of the response (criterion) variable. R^2 is called the coefficient of determination indicates the explanatory power of any regression model. Its value lies in between '0' and '+1'. It can be shown that R^2 is the correlation between experimental and predicted value. It will reach a maximum value when the dependent variable is perfectly predicted by the regression. R^2 is the square of this measure of correlation and indicates the proportion of the variance in the response variable, which is accounted for by the model. In essence, this is a measure of how good a prediction of the criterion variable can be made by knowing the predictor variables. However, R^2 tends to somewhat overestimate the success of the model when applied to the real world and so an adjusted R^2 value is calculated which takes into account the number of variables in the model and the number of observations the model is based on. This adjusted R^2 value gives the most useful measure of the success of the model.

5.4 EXPRESSIONS FOR THE OUTPUT VARIABLES - ANALYSIS OF VARIANCE (ANOVA)

Analysis of variance is a powerful statistical technique used to confirm the effect of several simultaneously applied factors on the response variable. A null hypothesis, postulating no dependence of the applied factors and response variables are considered and is checked for its validity. degrees of freedom (df) and the sum of the squares (ss) are computed from the consideration data. f-statistic (variance ratio) is computed as the ratio of sums of squares denoting influence of factors and their interdependence. The computed value of variance ratio (f) is compared with the standard ANOVA table and the hypothesis is significant or not significant.The significance of the parameters on the responses could be found using ANOVA. Analysis of Variance (ANOVA) consists of calculations that provide information about levels of variability within a regression model and form a basis for tests of significance. In this work, a commercially available mathematical software package Statistical Package in Social Science was used for the computation of regression constants and parameters.

5.5 SIGNIFICANCE OF REGRESSION

The significance of a regression coefficient in a regression model is determined by dividing the estimated coefficient over the standard deviation of this estimate. To determine the regression analysis, there is a relationship between response variable 'y' and a subset of regression. The hierarchical model of ANOVA response as shown in Table 6.1, the coefficient of determination R-square is proportion of variability. In this definition, the term “variability” is defined as the sum of squares. There are equivalent expressions for R-square based on analysis of variance decomposition. Adjusted R-square is a modification of R-square that adjusts for the number of terms in a model.

Table 5.1 ANOVA response

Source	**Hierarchical Method**				
Model	**Degrees of freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance Ratio (F)**	**Significant**
Regression	k	SS_R	MS_R	$\frac{MS_R}{MS_E}$	-
Error	n-k-1	SS_E	MS_E	-	-
Total	n-1	$SS_y = SS_R + SS_E$	-	-	-

5.5.1 ANOVA response on mechanical properties

Analysis of variance is similar to regression in that it is used to investigate and model the relationship between a response variable and one or more independent variables. The main purpose of this work is to use a new technique that combines functional data analysis and design of experiments, functional ANOVA for a one way treatment to measure the influence of adding banana fiber nanocomposites. The functional ANOVA uses all the information of each test or functional data. The results obtained using this methodology with the mechanical properties indicates that the percentage weight of nanofibers significantly affects the banana fiber nanocomposites.

Table 5.2 ANOVA response on tensile strength

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	Significant
Model	2	8408.65	4204.33	44.81	0.022	
Error	2	187.64	93.82	-	-	
Total	4	8596.29	-	-	-	

From the table 5.2. analysis of variance, a complete realization of the tensile strength of the nanocomposites and their effects were achieved. The variation of percentage weight nanofibers and tensile strength was mathematically modeled using response surface methodology. Finally the developed model is validated with the set of experiments. The Model F-value of 44.81 implies the model is significant and the R- squared value is obtained as 97.8% and the adjusted R- squared 95.6%. It is observed that developed model is in close agreement with the experimental results.

Table 5.3 ANOVA response on impact strength

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	
Model	2	0.543754	0.271877	34.06	0.029	Significant
Error	2	0.015966	0.007983	-	-	
Total	4	0.559720	-	-	-	

Impact strength of the banana fibers nanocomposites and their effects were achieved by ANOVA. The predicted data from the regression analysis is used for surface roughness with impact properties of different weight percentage of nanofibers. The developed model is validated with the set of experiments. The variance ratio is 34.06 which implies the predicted model is significant as given in the Table.5.3 and the R-squared value is obtained as 97.1% and the adjusted R-squared 94.3%.

Table 5.4 ANOVA response on flexural strength

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	
Model	2	2362.37	1181.19	35.60	0.027	Significant
Error	2	66.36	33.18	-	-	
Total	4	2428.73	-	-	-	

Flexural strength of the banana fiber nanocomposites and their effects were achieved by ANOVA. The predicted data from the regression analysis is used for surface roughness with flexural properties of different weight percentage of nanofibers. The developed model is validated with the set of experiments. The variance ratio is 35.60 which implies the predicted model is significant as given in the table. 5.4 and the R-squared value is obtained as 97.3% and the adjusted R-squared 94.5%.

Table 5.5 ANOVA response on hardness

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	Significant
Model	2	35.3189	17.6594	34.19	0.028	
Error	2	1.0331	0.5166	-	-	
Total	4	36.3520	-	-	-	

From the ANOVA response Table 5.5 the model f-value of 34.19 implies the model is significant and the R- squared value is obtained as 97.2% and the adjusted R-Squared 94.3%. The hardness of the nanofibers composites and their effects were achieved by analysis of variance. The developed model is validated with the set of experiments and the model is suitable.

Table 5.6 ANOVA response on damping ratio

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	Significant
Model	2	0.233869	0.116934	38.90	0.025	
Error	2	0.006011	0.003006	-	-	
Total	4	0.239880	-	-	-	

The stiffness of the nanofiber composites with different weight percentage and their effects werw predicted by regression analysis as given in Table. 5.6 The predicted model is validated with the set of experimental values. These results influence the overall damping effect of the nanofiber composites. The variance ratio 38.90 is significant and the R-squared value is 97.5% and the adjusted R-squared 95.0% is satisfied with the experimental results.

5.5.2 ANOVA response on thermal properties

The thermal properties like thermal degradation temperature and decomposition temperature by TGA. The crystallization temperature, and melting point temperature by DSC, and the dynamic behaviours like storage modulus, loss modulus and tan delta values by DMA are used for ANOVA response.

Table 5.7 ANOVA response on thermal degradation temperature (T_d)

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	Significant
Model	2	120.697	60.3484	32.97	0.029	
Error	2	3.661	1.8305	-		
Total	4	124.358	-	-		

The variation of percentage weight nanofibers and surface roughness with thermal degradation was mathematically modeled using response surface methodology. The model variance ratio is 32.97 is significant as shown in Table 5.7, R-squared value is 97.1% and the adjusted R- squared is 94.1%. The prediction provides valuable information that can be used to select banana nanofiber composite for certain end-used application.

Table 5.8 ANOVA response on thermal decomposition (T_{decomp})

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	
Model	2	56.4789	28.2394	34.76	0.028	Significant
Error	2	1.6250	0.8125	-	-	
Total	4	58.1039	-	-	-	

The thermal decomposition of banana fiber nanocomposites was predicted by using analysis of variance as shown in table 5.8. The ANOVA response predicts decomposition information of nanofiber composites and composite life time. The variance ratio is 34.76 and R-squared value is obtained as 97.2% and the adjusted R-squared 94.4%. which is suitable for experimental values.

Table 5.9 ANOVA response on crystallization temperature (T_c)

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	
Model	2	16.4014	8.20069	33.82	0.029	Significant
Error	2	0.4849	0.24245	-		
Total	4	16.8863	-	-		

The variation of percentage weight of banana nanofibers and surface roughness with crystallization temperature was mathematically modeled using response surface methodology is shown in Table 5.9. The developed model is validated with the set of experimental data of crystallization temperature. The model f-value of 33.82 implies the model is significant and the R-squared value is obtained as 97.1% and the adjusted R-squared 94.3%. It is observed that developed model is in close agreement with the experimental results.

Table 5.10 ANOVA response on melting temperature (T_m)

Source	Degrees of Freedom (DF)	Sum of Squares (SS)	Mean Square (MS)	Variance ratio (F)	F-Distrubution (P)	Significant
Model	2	91.0327	45.5163	33.18	0.029	
Error	2	2.7432	1.3716	-		
Total	4	93.7759	-	-		

The thermal behaviors heat flow with temperature variation and thermal stability of the nanofiber composites were predicted by regression analysis. The melting point temperature of the banana fiber nanocomposites and their effects were achieved by ANOVA. The different weight percentage of nanofibers and surface roughness with experimental data was modeled by using response surface methodology, as shown in Table 5.10. The predicted model is validated with the set of experimental values and the model f-value of 33.18 implies the model is significant The R- squared value is obtained as 97.1% and the adjusted R- squared 94.1%. These analyses useful for composite applications and different temperature conditions.

Table 5.11 ANOVA response on storage modulus

Source	Degrees of Freedom (DF)	Sum of Squares (SS)	Mean Square (MS)	Variance ratio (F)	F-Distrubution (P)	Significant
Model	2	583905	291952	36.52	0.027	
Error	2	15986	7993	-	-	
Total	4	599891	-	-	-	

Table 5.11 describes the storage modulus of the banana fiber nanocomposites and their effects by ANOVA. The developed model is validated with the set of experiments and model of variance ratio value is 36.52. This analysis provides glass transition

temperature secondary transition temperatures and yield information about the morphology of the nanofiber composites. It is observed that the model is in close agreement with the experimental results. The R- squared value is obtained as 97.3% and the adjusted R-squared 94.7%.

Table 5.12 ANOVA response on loss modulus

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	Significant
Model	2	530872	265436	34.05	0.029	
Error	2	15589	7795	-		
Total	4	546461	-	-		

The complete realization of the loss modulus of the nanofibers composites and their effects were achieved by analysis of variance. The variation of percentage weight nanofibers and surface roughness with loss modulus was mathematically modeled using response surface methodology. The predicted model f-value of 34.05 implies the model is significant. and the R-squared value is obtained as 97.1% and the adjusted R-squared 94.3% it is observed that developed model is satisfied with experimental results as shown in table 5.12.

Table 5.13 ANOVA response on tan delta

Source	**Degrees of Freedom (DF)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**Variance ratio (F)**	**F-Distrubution (P)**	Significant
Model	2	0.0044955	0.0022478	35.89	0.027	
Error	2	0.0001253	0.0000626	-	-	
Total	4	0.0046208	-	-	-	

The tan delta of the banana fiber nanocomposites and their response was tabulated by ANOVA. The different weight percentage of nanofibers and surface roughness with tan delta was modeled by using response surface methodology. The above table is validated with the set of experimental values and the Model f-value of 35.89 is significant and the R- squared value is obtained as 97.3% and the adjusted R-squared is 94.6% satisfied the experimental results as shown Table 5.13. This result provides the phase lag the displacement composite to the applied force which implies the damping property of banana nanofiber composites.

5.6 COMPARISION OF MECHANICAL PROPERTIES WITH REGRESSION ANALYSIS

The values obtained using the regression equations is represented in Table 5.14 (Appendix - II). The experimental values of mechanical properties are compared with the predicted values. The error percentage is tabulated in Table 5.16 (Appendix - II)

5.6.1 Tensile strength results predicted by regression analysis

The tensile strength values of the predicted by regression model are compared with experimental results with the different weight percentages of nanofiber reinforcement in epoxy resin as shown in Fig.5.1. It is observed that the maximum error 2.67 for regression model when compared to experimental results Which shows better agreement with experimental results for 4wt. % of BNFC.

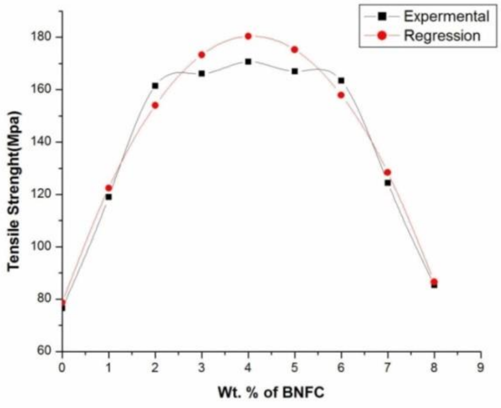

Fig.5.1 Regression with experimental values for tensile strength

5.6.2 Impact strength results predicted by regression analysis

The percentage error observed maximum for regression model is 2.82, regression analysis are presented in Fig.5.2. It is observed that maximum error 2.82 for regression model at 6wt. % of BNFC Composite. These errors are due to the precence of voids in the nanofibr composites.

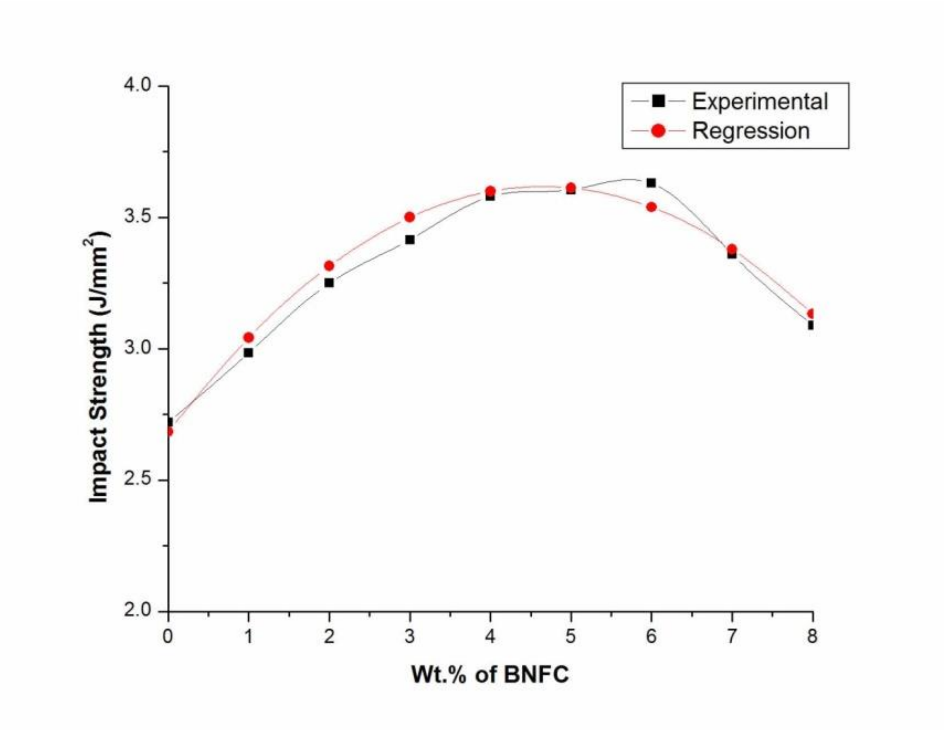

Fig.5.2 Regression with experimental values for impact strength

5.6.3 Flexural strength results predicted by regression analysis

The percentage error observed in regression model is 2.82, regression analysis are presented in Fig.5.3. It is observed that maximum error 2.82 for regression model at 3 wt. % of BNFC Composite. These errors ar due to the precence of voids in the nanofibr composites.

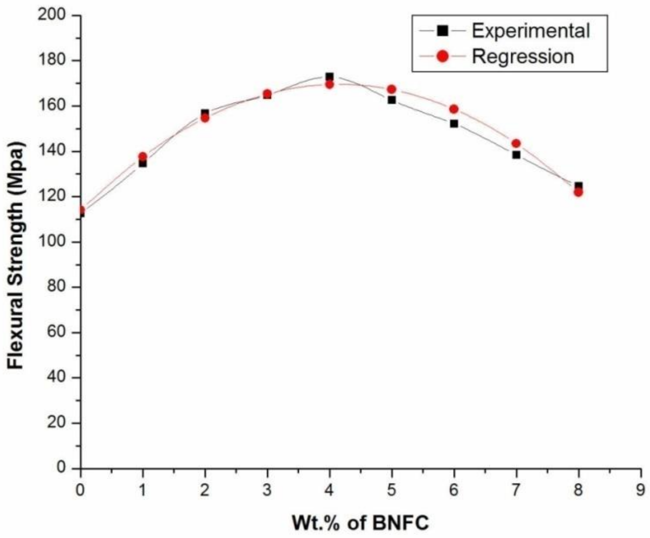

Fig.5.3 Regression with experimental values for flexural strength

5.6.4 Hardness results predicted by regression analysis

Fig.5.4. The maximum error 1.35 for the regression model.The error is observed in 6wt.% nanofiber composites with regression values.The results provide the homogenity of the nanofiber composites.

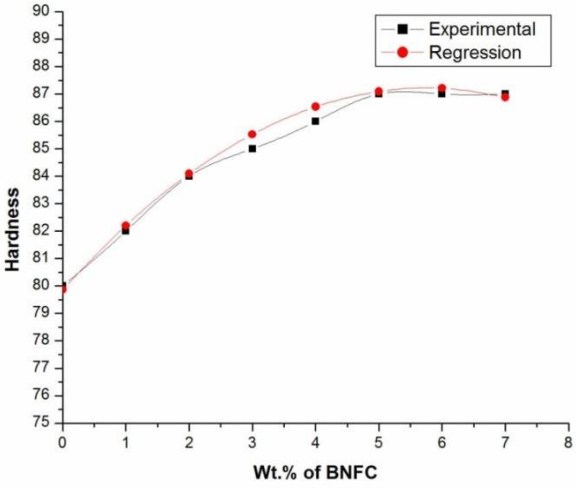

Fig.5.4 Regression with experimental values for hardness

5.6.5 Damping property results predicted by regression analysis

The damping ratio error percentage predicted by regression model is compared with experimental results with the weight percentages of nanofiber reinforcement in epoxy resin as shown in Fig.5.5. It is observed that the maximum error 2.44 for regression model for the 2wt. % of BNFC.

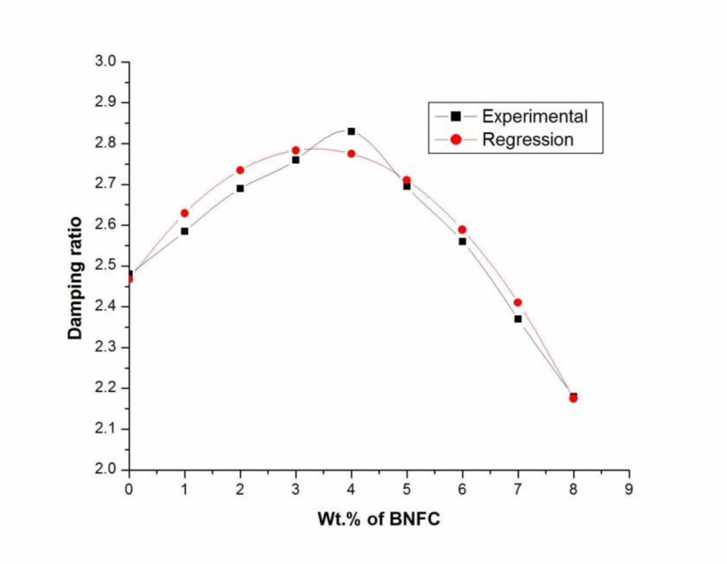

Fig.5.5 Regression with experimental values for damping ratio

5.7 COMPARISION OF THERMAL PROPERTIES WITH REGRESSION ANALYSIS

The thermal properties results obtained by the predicted models of regression were compared with experimental results of BNFC composite with different wt.% of nanofiber reinforcement.The values obtained from the regression equations are tabulated in Table 5.15 (Appendix -II). The error percentage between experimental values and the values obtained experimentally for both mechanical and thermal properties are represented in Table 5.17 shown in Appendix - II.

5.7.1 Thermal degradation temperature

The error percentage provides the thermal stability of nanofibers composites with change in temperature. It is also useful to determine the weight loss of the composites at specified temperature. The maximum error of regression models is 0.35 for 3wt. % nanofiber composites as shown in Fig.5.6.

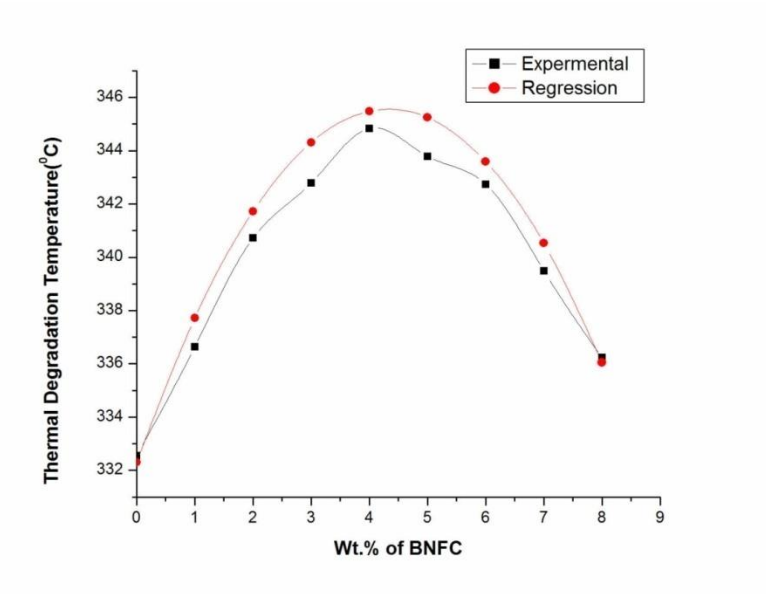

Fig.5.6 Regression with experimental values for thermal degradation temperature

5.7.2 Decomposition temprature

The decomposition temperature values of the predicted by regression model are compared with experimental results with the different weight percentages of nanofiber reinforcement in epoxy resin as shown in Fig.5.7. It is observed that the maximum error 2.07 for regression were observed in 3wt. % nanofibers composites. These error values are useful to predict the composite life.

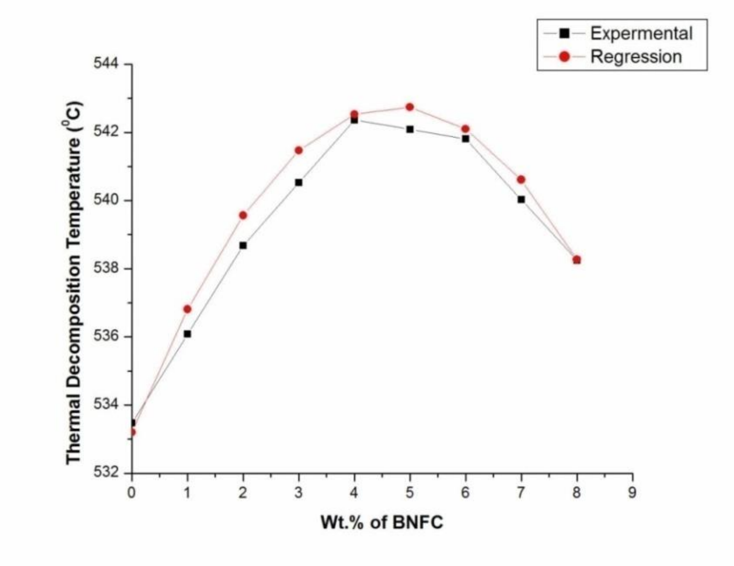

Fig.5.7 Regression with experimental values for decomposition temprature

5.7.3 Crystallization temperature

The effect of banana nanofiber reinforcement alters the crystallization temperature of nanofiber composites. The values predicted by regression model, compared with experimental results with the nanofiber reinforcement in epoxy resin as shown in Fig.6.8. The percentage error is less. These error percentage results are useful to adjust the processing parameters of reinforced polymer nanofibers composites.The maximum error in a regression analysis is 0.9, for 5wt. % banana fiber nanocomposites.

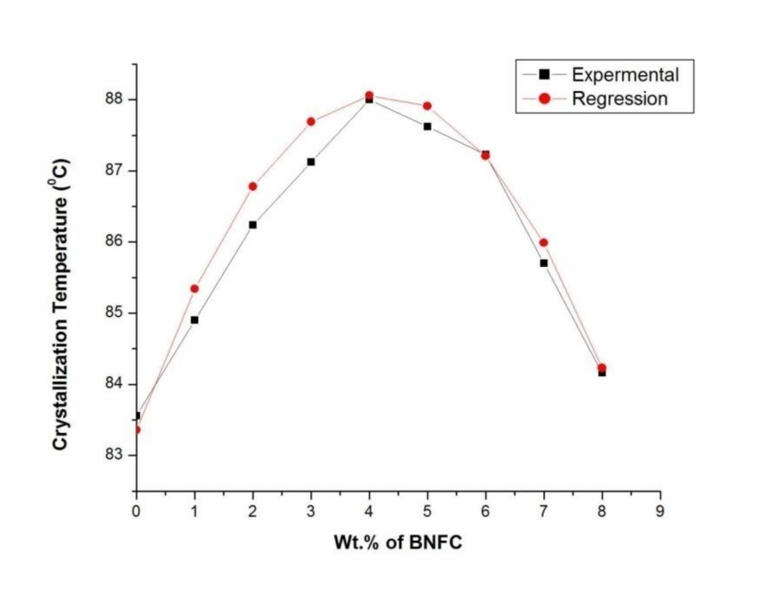

Fig.5.8 Regression with experimental values for crystillization temperature

5.7.4 Melting point temperature

The melting point temperature values of the predicted by regression model and compared with experimental results with the different weight percentages of nanofiber reinforcement in epoxy resin as shown in Fig.5.9. It is observed that the maximum error 0.08 for regression model for 3wt. % nanofibers composites.

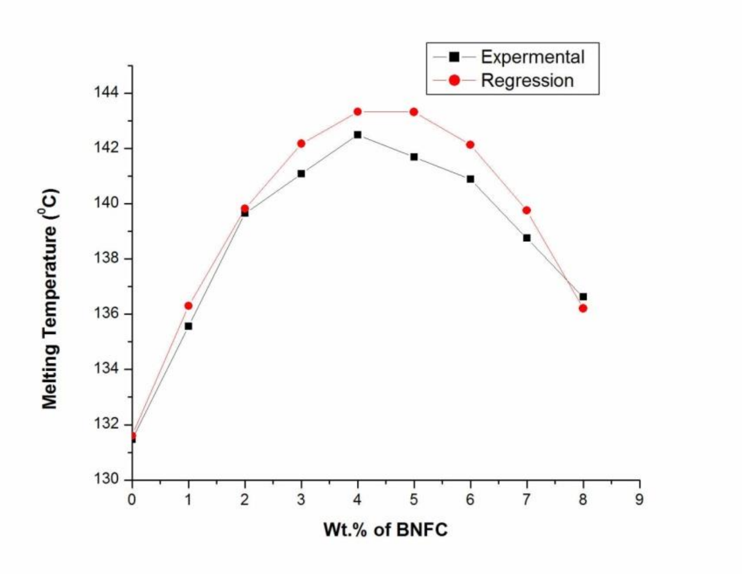

Fig.5.9 Regression with experimental values for melting point temperature

5.7.5 Dynamic Mechanical Analysis

The predicted results obtained by regression are compared with experimental values and the comparison graphs are plotted for storage modulus,loss modulus and tanδ.

5.7.5.1 Storage Modulus

The storage modulus values obtained by DMA were predicted by regression model are compared with experimental results with the different weight percentages of nanofiber reinforcement in epoxy resin as shown in Fig.5.10. It is observed that the maximum error 3.42 for regression model for 2wt. % banana nanofiber composites.

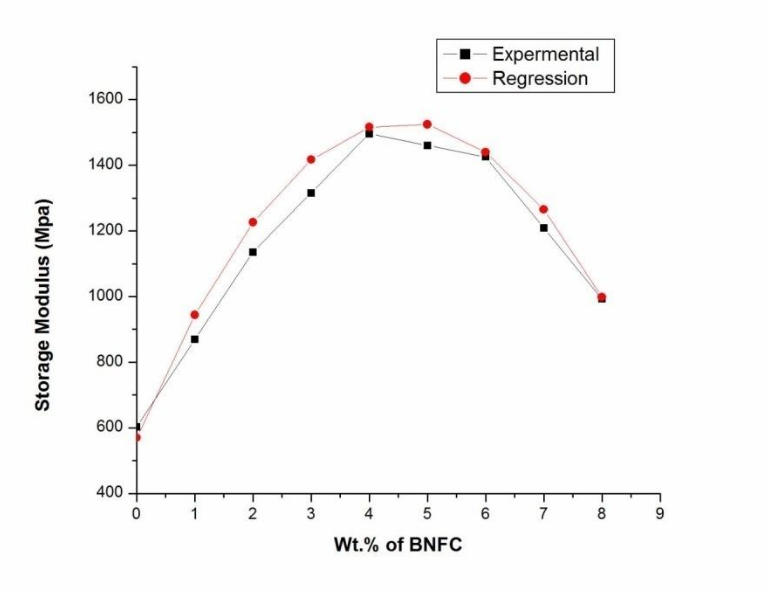

Fig.5.10 Regression with experimental values for storage modulus

5.7.5.2 Loss Modulus

The Loss Modulus values of the predicted by regression model compared with experimental results with the different weight percentages of nanofiber reinforcement in epoxy resin as shown in Fig.5.11. The maximum error 2.75 for Regression model and 3.05 for 2wt. % nanofibers composites.

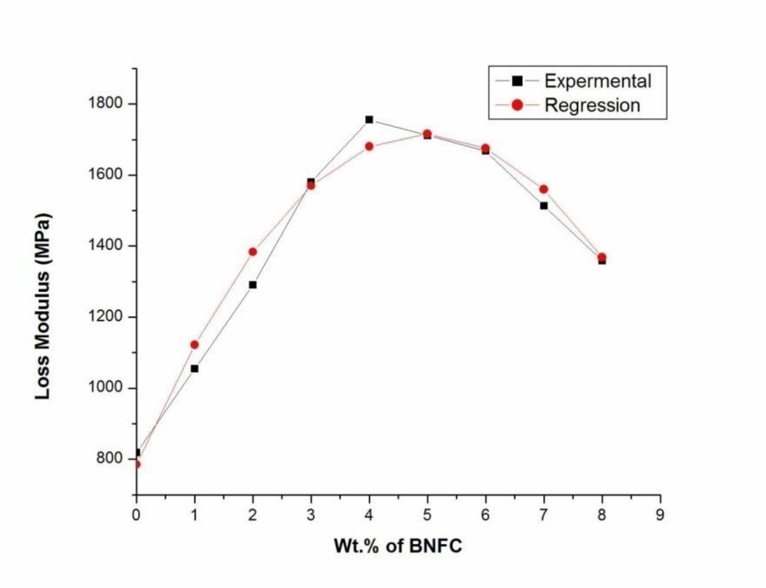

Fig.5.11 Regression and experimental values for loss modulus

5.7.5.3 Tan δ

The Tan δ error percentage values are predicted by regression model compared with experimental results with the different weight percentages of nanofiber reinforcement in epoxy resin as shown in Fig.6.12. It is observed that the tan delta decreased with the increased nanofibers reinforcement, at 3wt. % maximum error 3.85 for Regression model.

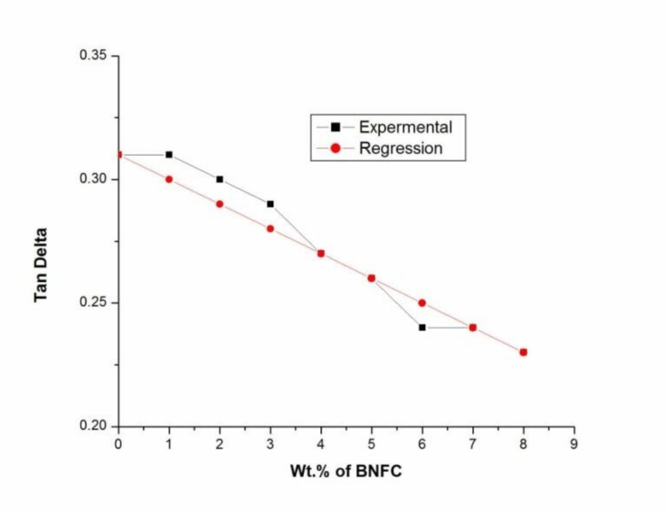

Fig.5.12 Regression and experimental values for tan δ

5.8 SUMMARY

The mechancial properties of the nanofibers composites such as tensile strength, impact strength,flexural strength, hardness, damping and thermal properties like thermal degradation temperature, decomposition temperature, crystallization temperature, melting temperature and the dynamic behaviours like storage modulus, loss modulus and tan delta prediction models are developed using regression.The experimental data have been used to develop these models. Then models are used to predict the mechanical, thermal properties at different weight percentage of banana fiber nanocomposite. The comparison of predicted values with experimental values in all tested cases indicates that the error is less, than 5% in the regression model when compare to experimental results. From this it is observed that this model is found to be capable of predicting the above parameters with accuracy.

CHAPTER–6

CONCLUSIONS AND FUTURE SCOPE

6.1 CONCLUSIONS

The present work investigates the performance of banana fiber polymer nanocomposites. Different parameters which affect the mechanical, thermal and water absorption properties are studied. Most of the investigations include experiments and analytical modelling. From all these investigations, it is observed that there are several common conclusions besides those remarks at the end of each chapter for the banana fiber nanocomposites.

Physical and Tensile Properties of the banana fiber nanocompoaites exhibit superior advantages over the synthetic fibers especially in cost, environmental aspects and high specific modulus compared to synthetic fibers. The physical and mechanical properties of the banana fiber nanocompoaites are observed. The results found good agreement within the range in the literature. The tensile strength of nanocomposites was increased from 50% to 96% with an increasing Banana nanofiber neat composite, 2, 4, 6 and 8wt. % contained. The maximum tensile strength improvement was noticed as 96% for the 4wt. % banana fiber nanocomposites, and then decreased when the content of fibers is 6wt. % and 8wt. %. The increase in tensile strength is attributed to strong interaction between the polymer and banana nanofibers. The interaction has a large impact in nanocomposites due to the large interfacial area between the nanofibers and the resin.The improvement in tensile strength appears to be promising in structural applications irrespective of the direction of the applied load. However, the drawbacks of natural fibers include wear, low shear strength and compression limiting the potential of natural fiber composites in structural use. The drawbacks can be partially overcome by introducing chemical treatment.

The mechanical properties of fiber reinforced composites are dependent upon the stability of the interfacial region. Thus, the characterization of the interface is of great importance. Alkali treatment increases the impurities of fiber surface and increased the interface between fiber and matrix, and also decreases the fiber pullout which enhanced tensile, flexural, impact and hardness properties. Different parameters affected the mechanical properties of natural fiber composites in this investigation, namely curing process, nanofiber weight fraction and fiber treatment. SEM analysis showed that the interaction between the hybrid fibers and polymer matrix is poor such that fiber debonding, fiber pull-out, matrix fracture and fibers fracture occurred. The experimental data of mechanical properties results was compared with mathematical predictions and found to be in good agreement with some of these models.

Effects of Water Absorption, The results of this part discussed the water absorption characteristics and environmental affects of alkali treated banana fiber nanocomposites at immersion times, and fiber weight fraction and temperature surrounding played a role in the rate of moisture uptake. The natural fiber nanocomposites were found to be dependent on the fiber length and fiber weight fraction.

Thermal properties of composites and thermal stability of banana fiber reinforced polymer nanocomposites are considerably dependent on nanofiber weight fraction. The results exposed that incorporation of the nanofibers gave rise to a considerable increase of the E', Tg and a decrease in the tan delta values. The loss modulus peak got broadened emphasizing the improved fiber/matrix adhesion. The Tg temperature shifted positively.

All the nanocomposites had a higher melting temperature compared to neatcomposites. The TGA curves indicate that the thermal degradation of pure epoxy composite started at 344°C and 100% degradation was noticed at 559°C. However, with the incorporation of banana nanofibers, there was a substantial enhancement in the thermal stability of the nanocomposites with an initial degradation temperature at 370°C and final decomposition at 584°C. This indicates that a significant increase in the Banana nanofibers content of fiber reinforced composites plays an important role in controlling its rate of thermal degradation. DSC curves show that the addition of banana nanofibers increased the crystallization temperature Tc by up to 1-4°C compared to the Neat composites. This result indicates that the nucleating effect of Banana nanofibers composites was strengthened. The banana nanofibers played the role of a nucleating agent and facilitated crystallization due to the strong interaction between banana nanofibers and polymer resin. The nucleating effect of banana nanofibers could also explain the increase of crystalline. The nano dispersion of the filler and its orientation in the matrix are among these factors. DMA results banana nanofiber composites indicates the storage modulus E' decreases with increasing temperature, while the loss modulus E" increases. This indicates that the increased modulus, together with a positive shift in the tan delta peak position.

6.2 FUTURE SCOPE

In this research work, the mechanical and Thermal behaviour of a nanocomposite was evaluated.

- The Potential applicability of nanofiber composites is widely extended by studying its electrical and magnetic properties like dielectric strength and permeability that investigated to know the electrical strength of the material as an insulator.
- To investigate the rheological behaviour of the nanocomposites, which provides an insight into the processability of the materials, polymer molecular architecture, morphology, chain branching in polymer micro-structural development, temperature dependent of properties and degradation and stability.
- The dynamic mechanical analysis in shear and tension, fatigue failure and creep failure with oscillating frequencies. Since these materials are suitable for automotive, aerospace, marine, military, structural and other applications. The nanocomposites can be fabricated by using other natural fibers and investigate the mechanical and thermal properties for suitable applications.
- The tribological behaviour of the composite will be of immense importance because when components are made from natural fibers, their wear behaviour is crucial, when subjected to working environments.
- The application of banana nanofiber composites can be used for further review so as to make it compatible to be used in various fields.
- The experimental values of the static and dynamic analysis can be further validated by using various other emperical rules and techniques.
- Testings can be carried out in the component level which are manufactured using nanocomposite material in order to satisfy wide applications.
- Static and dynamic structural analysis can be extended to consolidate the experimental results.

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APPENDIX – I

TABLE 4.3: Experimental data of mechanical properties of alakali treated banana fiber nanocomposites

Reinforcement (%)	Tensile Strength (MPa)	Impact Strength (J/m)	Flexural Strength (MPa)	Hardness	Damping Ratio (%)
Neat Composite	76.54 ±0.82	2.72 ±1.2	112.56 ±0.8	80 ±0.6	0.019 ±0.2
2wt.% BNFC	161.48 ±0.75	3.25 ±1.5	156.71 ±1.2	84 ±0.8	0.032 ±0.4
4wt.% BNFC	170.65 ±0.54	3.58 ±1.8	172.89 ±1.4	86 ±0.6	0.036 ±0.6
6wt.% BNFC	163.45 ±0.68	3.63 ±2.4	152.15 ±1	88 ±0.5	0.028 ±0.4
8wt.% BNFC	85.42 ±0.78	3.14 ±2.1	124.56 ±0.9	85 ±0.4	0.021 ±0.4

TABLE 4.4: Experimental data of damping behavior of the banana fiber nanocomposites with various natural frequency

Natural Frequency (Hz)	Neat Composite	2wt. % BNFC	4wt. % BNFC	6wt. % BNFC	8wt. % BNFC
0.0	6.81E-01	5.78E-01	3.08E-01	6.02E-02	2.18E-02
10.0	5.52E-01	2.87E-01	2.10E-01	1.26E-01	5.22E-02
20.0	2.47E-01	1.76E-01	2.35E-01	9.37E-02	7.56E-02
30.0	5.15E-01	8.00E-01	1.50E+00	4.18E-01	1.97E-01
40.0	8.17E-01	6.82E-01	7.03E-02	4.25E-01	4.82E-01
50.0	1.41E-01	2.44E-01	3.78E-01	4.86E-02	6.05E-02
60.0	1.96E-01	4.07E-01	8.14E-01	3.25E-01	2.80E-01
70.0	5.64E-01	3.82E-01	1.15E+00	5.75E-01	5.92E-01
80.0	9.84E-01	8.68E-01	1.37E+00	5.60E-01	3.26E-01
90.0	1.47E+00	1.43E+00	2.04E+00	8.63E-01	6.00E-01
100.0	2.00E+00	2.01E+00	2.81E+00	1.24E+00	8.93E-01
110.0	2.61E+00	2.69E+00	3.76E+00	1.69E+00	1.20E+00
120.0	3.34E+00	3.58E+00	4.94E+00	2.20E+00	1.55E+00
130.0	3.97E+00	4.69E+00	6.59E+00	2.85E+00	1.98E+00
140.0	4.70E+00	5.84E+00	8.75E+00	3.71E+00	2.53E+00
150.0	6.31E+00	7.21E+00	1.21E+01	4.71E+00	3.20E+00
160.0	8.30E+00	9.77E+00	1.81E+01	5.97E+00	4.06E+00
170.0	1.08E+01	1.37E+01	3.28E+01	7.82E+00	5.21E+00
180.0	1.61E+01	2.17E+01	8.98E+01	1.04E+01	6.95E+00
190.0	2.70E+01	4.00E+01	4.89E+01	1.60E+01	9.65E+00
200.0	5.47E+01	8.31E+01	2.26E+01	2.85E+01	1.48E+01
210.0	1.00E+02	5.62E+01	1.42E+01	7.94E+01	2.72E+01
220.0	4.66E+01	2.62E+01	9.67E+00	4.85E+01	5.11E+01
230.0	2.81E+01	1.74E+01	6.74E+00	2.18E+01	2.91E+01
240.0	2.03E+01	1.23E+01	4.59E+00	1.40E+01	1.47E+01
250.0	1.55E+01	9.22E+00	2.92E+00	1.02E+01	9.54E+00
260.0	1.20E+01	6.96E+00	1.35E+00	7.44E+00	6.79E+00

270.0	9.68E+00	5.22E+00	6.90E-01	5.68E+00	5.11E+00
280.0	7.88E+00	3.68E+00	1.93E+00	4.31E+00	3.77E+00
290.0	6.35E+00	2.31E+00	3.29E+00	3.17E+00	2.76E+00
300.0	5.04E+00	1.17E+00	4.70E+00	2.16E+00	1.93E+00
310.0	3.84E+00	6.20E-01	6.44E+00	1.25E+00	1.25E+00
320.0	2.76E+00	1.71E+00	8.16E+00	4.82E-01	6.86E-01
330.0	1.97E+00	3.04E+00	1.01E+01	7.41E-01	5.97E-01
340.0	1.14E+00	4.36E+00	1.16E+01	1.56E+00	1.10E+00
350.0	3.82E-01	5.92E+00	1.42E+01	2.22E+00	1.74E+00
360.0	1.33E+00	7.68E+00	1.75E+01	3.28E+00	2.33E+00
70.0	2.63E+00	8.54E+00	2.06E+01	4.38E+00	3.05E+00
380.0	4.26E+00	9.79E+00	2.31E+01	5.50E+00	3.95E+00
390.0	6.03E+00	1.19E+01	2.72E+01	6.63E+00	4.35E+00
400.0	8.06E+00	1.51E+01	3.39E+01	7.90E+00	5.29E+00
410.0	1.07E+01	1.84E+01	3.97E+01	9.53E+00	6.41E+00
420.0	1.31E+01	2.03E+01	4.79E+01	1.19E+01	7.62E+00
430.0	1.46E+01	2.31E+01	6.00E+01	1.39E+01	8.64E+00
440.0	1.65E+01	2.68E+01	7.62E+01	1.51E+01	9.61E+00
450.0	1.86E+01	3.14E+01	1.03E+02	1.74E+01	1.14E+01
460.0	2.14E+01	3.68E+01	1.60E+02	2.05E+01	1.33E+01
470.0	2.69E+01	4.43E+01	2.91E+02	2.41E+01	1.53E+01
480.0	3.43E+01	5.48E+01	4.05E+02	2.82E+01	1.77E+01
490.0	4.27E+01	6.87E+01	2.23E+02	3.37E+01	2.07E+01
500.0	5.19E+01	8.79E+01	1.67E+02	4.09E+01	2.43E+01
510.0	6.75E+01	1.24E+02	1.44E+02	5.04E+01	2.88E+01
520.0	1.02E+02	2.02E+02	1.27E+02	6.36E+01	3.45E+01
530.0	1.66E+02	3.81E+02	1.17E+02	8.33E+01	4.19E+01
540.0	2.46E+02	4.59E+02	1.07E+02	1.23E+02	5.28E+01
550.0	2.06E+02	2.68E+02	9.20E+01	2.08E+02	6.98E+01
560.0	1.50E+02	2.06E+02	7.69E+01	3.66E+02	9.61E+01
570.0	1.19E+02	1.64E+02	6.88E+01	2.66E+02	1.44E+02
580.0	9.92E+01	1.30E+02	6.09E+01	1.50E+02	2.29E+02

590.0	8.01E+01	1.12E+02	5.40E+01	1.06E+02	2.40E+02
600.0	6.48E+01	9.25E+01	4.87E+01	8.31E+01	1.34E+02
610.0	5.71E+01	8.36E+01	4.44E+01	6.69E+01	9.10E+01
620.0	4.87E+01	7.97E+01	4.09E+01	5.86E+01	7.04E+01
630.0	4.03E+01	7.58E+01	3.78E+01	5.20E+01	6.05E+01
640.0	3.79E+01	6.84E+01	3.54E+01	4.97E+01	5.17E+01
650.0	3.60E+01	6.19E+01	3.30E+01	4.39E+01	4.47E+01
660.0	3.40E+01	5.67E+01	2.94E+01	3.96E+01	3.87E+01
670.0	3.49E+01	5.19E+01	2.64E+01	3.63E+01	3.40E+01
680.0	3.49E+01	4.82E+01	2.41E+01	3.41E+01	3.09E+01
690.0	3.45E+01	4.50E+01	2.18E+01	3.20E+01	2.84E+01
700.0	3.46E+01	4.32E+01	1.93E+01	3.06E+01	2.66E+01
710.0	3.57E+01	4.13E+01	1.68E+01	2.96E+01	2.52E+01
720.0	3.87E+01	3.97E+01	1.40E+01	2.88E+01	2.37E+01
730.0	4.13E+01	3.73E+01	1.17E+01	2.84E+01	2.22E+01
740.0	4.51E+01	3.32E+01	9.46E+00	2.74E+01	2.09E+01
750.0	4.51E+01	3.01E+01	7.19E+00	2.77E+01	1.99E+01
760.0	3.70E+01	2.86E+01	5.07E+00	2.88E+01	1.93E+01
770.0	2.95E+01	2.73E+01	4.76E+00	3.03E+01	1.91E+01
780.0	2.47E+01	2.63E+01	7.61E+00	2.28E+01	1.90E+01
790.0	1.93E+01	2.52E+01	1.20E+01	1.95E+01	1.93E+01
800.0	1.69E+01	2.38E+01	1.63E+01	1.96E+01	1.85E+01

TABLE 5.1: Experimental data of TGA curves of the banana fiber nanocomposites with various weight percentages

Temperature (°C)	Neat Composite	2wt. % BNFC	4wt. % BNFC	6wt. % BNFC	8wt. % BNFC
29.849	76.818	77.312	77.812	78.312	79.312
100.049	79.697	83.439	86.439	89.439	91.439
200.942	77.627	82.219	85.219	88.219	90.219
300.972	73.886	77.994	80.994	83.994	85.994
400.967	41.232	49.388	52.388	55.388	57.388
500.885	30.914	39.180	42.180	45.180	47.180
600.915	18.634	24.369	27.369	30.369	32.369
700.978	19.280	23.470	26.470	29.470	31.470
745.682	18.990	23.396	26.396	29.396	31.396

TABLE 5.2: Experimental values thermogravimetric analysis of of alakali treated banana fiber nanocomposites

Reinforcement (%)	Thermal Degradation Temperature (°C)	Decomposition Temperature (°C)
0	332.55	533.47
2	340.72	538.68
4	346.84	543.36
6	342.74	541.81
8	336.23	538.24

TABLE 5.3 : Experimental data of DSC curves of the banana fiber anocomposites with various weight percentages

Temperature (^{o} C)	Neat Composite	2wt. % BNFC	4wt. % BNFC	6wt. % BNFC	8wt. % BNFC
30.025	-0.016	-0.018	-0.013	-0.006	-0.016
40.026	-0.043	-0.241	-0.265	-0.307	-0.413
50.013	-0.056	-0.266	-0.284	-0.332	-0.440
60.984	-0.067	-0.295	-0.298	-0.348	-0.461
70.978	-0.110	-0.346	-0.314	-0.398	-0.519
80.991	-0.096	-0.339	-0.351	-0.386	-0.507
90.971	-0.084	-0.321	-0.329	-0.376	-0.493
100.100	-0.080	-0.299	-0.317	-0.366	-0.492
110.995	-0.073	-0.282	-0.309	-0.360	-0.483
120.971	-0.073	-0.279	-0.314	-0.367	-0.487
130.013	-0.078	-0.278	-0.323	-0.385	-0.500
140.020	-0.080	-0.276	-0.328	-0.402	-0.513
150.031	-0.079	-0.270	-0.331	-0.413	-0.524
160.008	-0.077	-0.263	-0.335	-0.418	-0.529
170.020	-0.075	-0.260	-0.340	-0.420	-0.527
180.030	-0.073	-0.260	-0.342	-0.420	-0.528
190.006	-0.070	-0.259	-0.347	-0.420	-0.529
200.017	-0.066	-0.251	-0.348	-0.420	-0.529
210.030	-0.061	-0.235	-0.348	-0.420	-0.526
220.007	-0.052	-0.218	-0.343	-0.418	-0.518
230.023	-0.040	-0.209	-0.331	-0.411	-0.501
240.002	-0.022	-0.217	-0.309	-0.397	-0.476
250.019	0.004	-0.197	-0.276	-0.375	-0.440
260.004	0.039	-0.113	-0.226	-0.342	-0.393
270.025	0.082	-0.073	-0.161	-0.294	-0.328
275.015	0.109	0.249	0.287	0.233	0.309
274.999	0.102	0.253	0.291	0.237	0.316
270.991	0.234	0.508	0.615	0.639	0.758
263.800	0.223	0.465	0.572	0.627	0.734
255.971	0.188	0.421	0.518	0.583	0.683
240.704	0.138	0.366	0.449	0.522	0.607
220.237	0.100	0.323	0.394	0.472	0.547
208.538	0.088	0.308	0.376	0.454	0.524
200.874	0.082	0.300	0.367	0.444	0.513
192.009	0.076	0.292	0.359	0.435	0.503
190.009	0.075	0.291	0.357	0.434	0.501
180.012	0.071	0.284	0.350	0.425	0.493
170.017	0.068	0.279	0.344	0.418	0.486
164.019	0.066	0.276	0.341	0.415	0.483
150.891	0.064	0.270	0.335	0.408	0.476

TABLE 5.3 : Experimental data of DSC curves of the banana fiber anocomposites with various weight percentages (contd.)

140.527	0.062	0.266	0.331	0.402	0.472
130.131	0.060	0.259	0.326	0.397	0.467
110.030	0.046	0.246	0.310	0.361	0.436
80.600	0.026	0.230	0.275	0.325	0.397
73.134	0.023	0.226	0.269	0.319	0.391
73.101	0.023	0.226	0.269	0.319	0.391

TABLE 5.4: Experimental values differential scanning calorimetry of alakali treated banana fiber nanocomposites

Reinforcement (%)	Crystallization Temperature (°C)	Melting Temperature (°C)
0	83.56	131.47
2	86.24	139.65
4	88.48	142.53
6	87.23	140.88
8	84.16	136.62

TABLE 5.5: Experimental data of dynamic mechanical analysis of the banana fiber nanocomposites with various weight percentages

(a) STORAGE MODULUS (E') - MPa

Temperature (^{o} C)	Neat Composite	2wt. % BNFC	4wt. % BNFC	6wt. % BNFC	8wt. % BNFC
5.200	10209.300	13905.200	14887.700	13209.300	11709.300
20.600	10120.400	13957.200	14829.300	13120.400	11620.400
30.000	10104.500	13965.100	14808.400	13104.500	11604.500
40.000	10140.900	13982.200	14782.200	13140.900	11640.900
50.000	10078.000	13977.000	14630.000	13078.000	11578.000
59.900	10020.600	13896.500	14543.100	13020.600	11520.600
70.000	8483.800	12912.400	13709.800	11483.800	9983.800
79.900	5698.160	10853.430	11695.200	8698.160	7198.160
90.000	3828.020	8322.050	9552.400	6828.020	5328.020
99.900	1843.970	6339.190	7539.080	4843.970	3343.970
110.000	1050.360	4581.660	5578.350	3652.220	2152.220
119.700	968.000	3397.870	3953.830	2957.680	1958.659
129.700	1056.000	2919.460	3598.400	2523.170	1823.478
139.900	1001.130	2848.900	3220.420	2486.520	1712.312
150.000	939.740	2879.960	3072.380	2435.130	1608.258

(b) LOSS MODULUS (E") - MPa

Temperature (^{o} C)	Neat Composite	2wt. % BNFC	4wt. % BNFC	6wt. % BNFC	8wt. % BNFC
5.921	220	489	689	389	320
20.225	217	475	675	375	307
40.654	210	483	683	383	300
50.845	215	492	692	392	305
59.925	216	517	717	417	306
70.654	214	588	788	488	304
80.269	270	921	1121	821	360
90.147	700	1328	1404	1228	790
100.365	1135	1510	1560	1410	1225
109.925	1220	1510	1581	1410	1310
119.914	986	1340	1431	1176	1076
129.825	380	989	1106	670	470
140.165	104	430	580	284	194
149.945	45	186	235	135	100

(c) TAN DELTA

Temperature (^{o} C)	Neat Composite	2wt. % BNFC	4wt. % BNFC	6wt. % BNFC	8wt. % BNFC
0.000	0.074	0.054	0.047	0.029	0.023
5.200	0.074	0.055	0.048	0.029	0.022
10.300	0.075	0.057	0.049	0.029	0.022
15.400	0.078	0.057	0.049	0.030	0.024
20.600	0.080	0.062	0.049	0.032	0.024
25.100	0.081	0.063	0.049	0.032	0.024
30.000	0.082	0.064	0.049	0.032	0.024
35.000	0.083	0.065	0.050	0.032	0.024
40.000	0.084	0.066	0.050	0.033	0.024
45.000	0.084	0.066	0.051	0.032	0.025
50.000	0.084	0.067	0.053	0.032	0.025
54.900	0.084	0.067	0.055	0.033	0.026
59.900	0.084	0.068	0.057	0.033	0.027
65.000	0.086	0.069	0.057	0.033	0.029
70.000	0.098	0.076	0.058	0.036	0.031

(c) TAN DELTA (contd.)

75.000	0.139	0.088	0.069	0.039	0.033
79.900	0.185	0.129	0.099	0.060	0.048
84.900	0.217	0.173	0.135	0.090	0.079
90.000	0.250	0.209	0.179	0.125	0.107
95.000	0.268	0.241	0.211	0.162	0.147
99.900	0.285	0.269	0.245	0.200	0.183
105.100	0.298	0.287	0.269	0.223	0.209
110.000	0.310	0.299	0.286	0.238	0.226
114.800	0.316	0.299	0.270	0.241	0.220
119.700	0.297	0.280	0.249	0.215	0.198
125.000	0.268	0.252	0.221	0.183	0.165
129.700	0.241	0.213	0.195	0.153	0.134
134.900	0.186	0.167	0.155	0.120	0.100
139.900	0.148	0.125	0.116	0.095	0.075
144.800	0.109	0.095	0.086	0.069	0.055
150.000	0.084	0.063	0.056	0.036	0.028

APPENDIX – II

TABLE 6.14: Mechanical properties of alakali treated banana fiber nanocomposites by regression analysis

Reinforcement (%)	Tensile strength (MPa)	Impact Strength (J/m)	Flexural Strength (MPa)	Hardness	Damping Ratio (%)
0	78.660	2.684	114.134	79.871	2.467
1	122.437	3.042	137.619	82.268	2.629
2	153.988	3.315	154.696	84.086	2.735
3	173.313	3.561	165.331	85.512	2.783
4	180.412	3.599	169.524	86.532	2.775
5	175.285	3.612	167.275	87.09	2.710
6	157.932	3.539	158.584	87.206	2.589
7	128.353	3.379	143.451	86.878	2.410
8	86.548	3.132	121.876	86.108	2.175

TABLE 6.15: Thermal properties of alakali treated banana fiber nanocomposites by regression analysis

(a) THERMOGRAVIMETRIC ANALYSIS		
Reinforcement (%)	**Thermal Degradation Temperature (°C)**	**Decomposition Temperature (°C)**
0	332.304	533.472
1	337.716	536.075
2	341.719	538.682
3	344.308	541.024
4	345.483	543.362
5	345.245	542.585
6	343.593	541.812
7	340.528	540.025
8	336.049	538.242

(b) DIFFERENTIAL SCANNING CALORIMETRY		
Reinforcement (%)	**Crystallization Temperature (°C)**	**Melting Temperature (°C)**
0	83.360	131.473
1	85.335	135.562
2	86.778	139.652
3	87.687	141.075
4	88.062	142.523
5	87.905	141.691
6	87.214	140.885
7	85.991	138.753
8	84.234	136.627

(c) DYNAMIC MECHANICAL ANALYSIS			
Reinforcement (%)	**Storage Modulus (E') - MPa**	**Loss Modulus (E")-Mpa**	**Tan Delta**
0	569.912	819.261	785.300
1	943.830	1054.955	1122.190
2	1226.220	1290.652	1383.660
3	1417.070	1580.324	1569.710
4	1516.380	1755.423	1680.340
5	1524.150	1711.492	1715.550
6	1440.380	1667.561	1675.340
7	1265.070	1513.415	1559.710
8	998.220	1359.275	1368.660

TABLE 6.16: Error percentage between experimental and regression of mechanical properties

Reinforcement (%)	Tensile strength (MPa)	Impact Strength (J/m)	Flexural Strength (MPa)	Hardness	Damping Ratio (%)
0	2.770	1.324	1.368	0.162	0.527
1	2.880	1.926	2.216	0.243	1.681
2	4.640	1.986	1.285	0.103	1.632
3	4.365	2.495	0.322	0.624	0.837
4	4.720	0.543	1.947	0.618	1.976
5	4.930	0.201	2.926	0.682	0.563
6	3.376	2.517	4.229	0.236	1.103
7	3.149	0.554	3.683	0.148	1.663
8	1.321	1.364	2.155	0.254	0.237

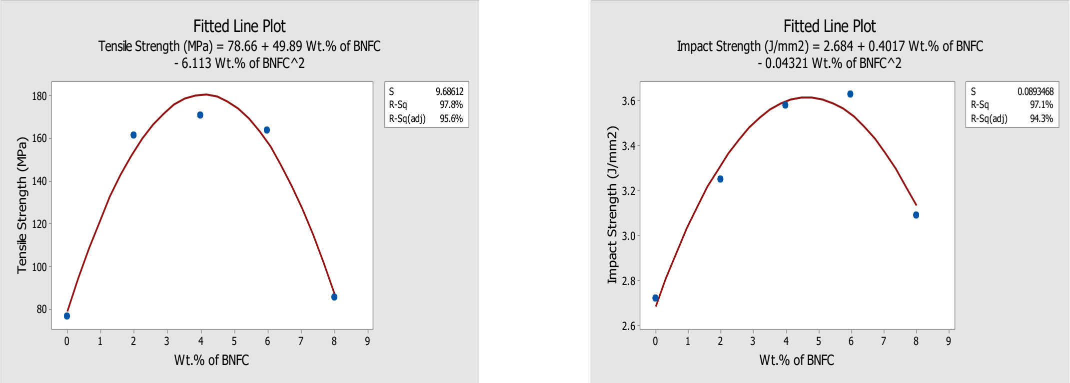
TABLE 6.17: Error percentage between experimental and regression of thermal properties

(a) THERMOGRAVIMETRIC ANALYSIS		
Reinforcement (%)	**Thermal Degradation Temperature (°C)**	**Decomposition Temperature (°C)**
0	0.075	0.051
1	0.321	0.137
2	0.293	0.164
3	0.154	0.084
4	0.391	0.153
5	0.132	0.029
6	0.249	0.053
7	0.307	0.108
8	0.054	0.005

(b) DIFFERENTIAL SCANNING CALORIMETRY		
Reinforcement (%)	**Crystallization Temperature (°C)**	**Melting Temperature (°C)**
0	0.239	0.099
1	0.513	0.546
2	0.623	0.123
3	0.650	0.773
4	0.071	0.583
5	0.331	1.149
6	0.018	0.885
7	0.345	0.726
8	0.087	0.300

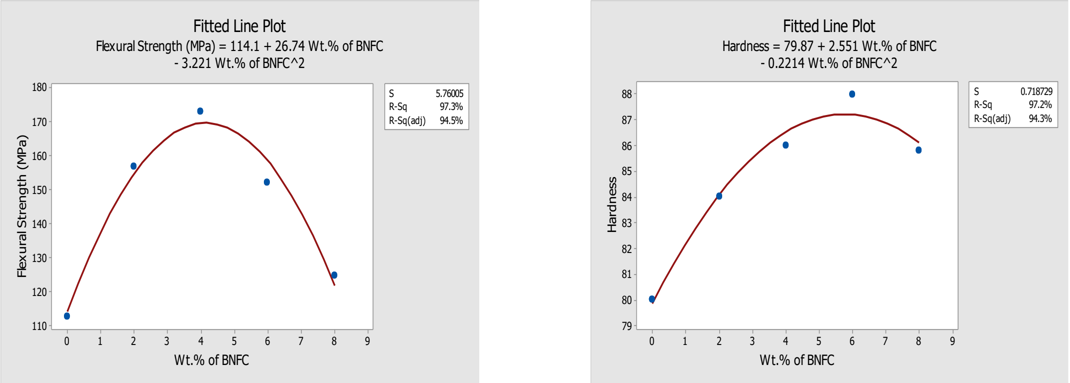
(c) DYNAMIC MECHANICAL ANALYSIS			
Reinforcement (%)	**Storage Modulus (E')- MPa**	**Loss Modulus (E")-Mpa**	**Tan Delta**
0	5.364	4.145	1.211
1	8.653	6.373	0.759
2	8.025	7.206	2.830
3	7.716	0.651	1.449
4	1.362	4.277	0.095
5	4.353	0.237	1.819
6	1.069	0.467	3.744
7	4.641	3.059	1.710
8	0.548	0.691	0.444

APPENDIX – III



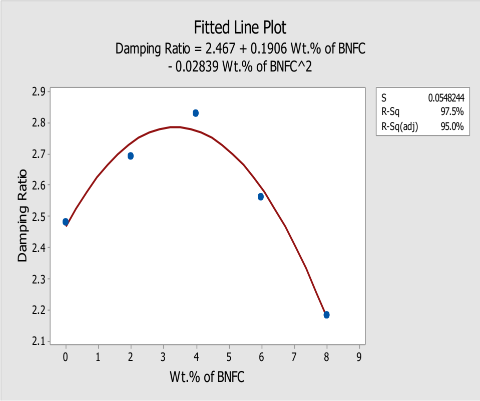
(a) Regression plot between wt.% of BNFC and tensile strength

(b) Regression plot between wt.% of BNFC and impact strength



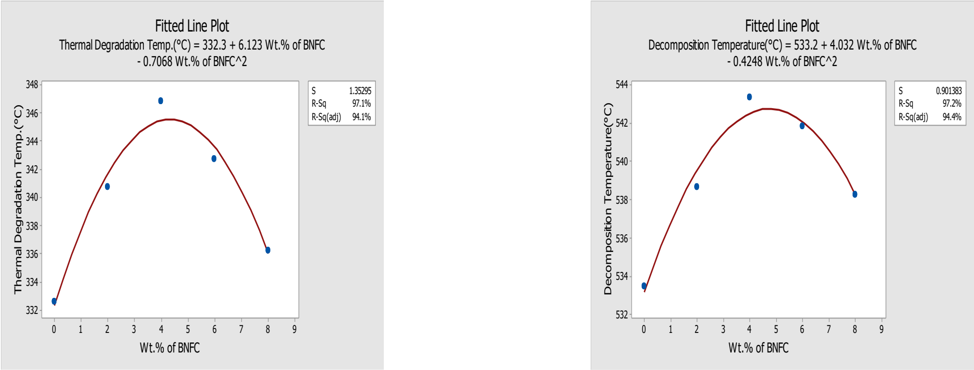
(c) Regression plot between wt.% of BNFC of flexural strength

(d) Regression plot between wt.% of BNFC of hardness



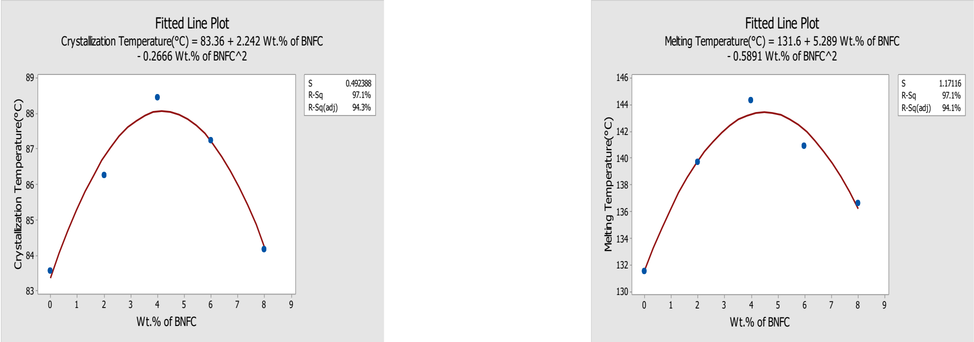
(e) Regression plot between wt.% of BNFC of damping ratio

Fig. 6.13 Regression Plots for Mechanical Properties



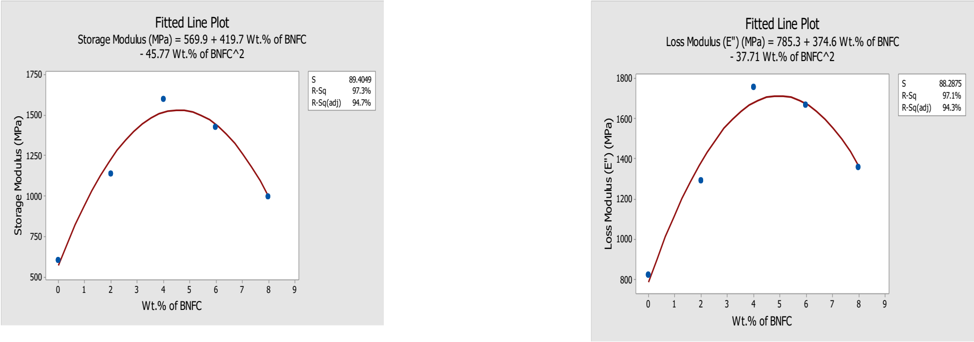
(a) Regression plot between wt.% of BNFC of Thermal Degradation Temperature

(b) Regression plot between wt.% of BNFC of Decomposition Temperature



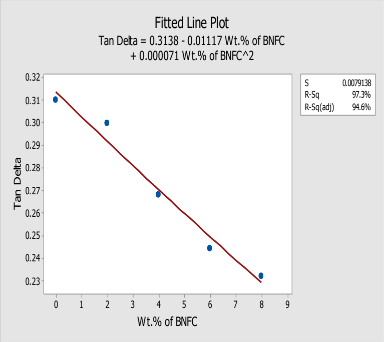
(c) Regression plot between wt.% of BNFC of Crystallization Temperature

(d) Regression plot between wt.% of BNFC of Melting Temperature



(e) Regression plot between wt.% of BNFC of Storage Modulus

(f) Regression plot between wt.% of BNFC of Loss Modulus



(g) Regression plot between wt.% of BNFC of Tan Delta

Fig. 6.14 Regression Plots for Thermal Properties

Papers Publications in Journals:

1. P.Surya Nagendra, VVS Prasad, Koona Ramji, Gangadhara Pusty " Synthesis of Bio-Degradable Banana Nano fibers" International Journal of Innovative Technology and Research, Vol. No.2, Dec - Jan 2014, 730-734.

2. P. Surya Nagendra, VVS Prasad, and Koona Ramji "Fabrication and Mechanical behavior of Banana Nano Fibre Reinforced Epoxy Composite" Journal of Material Science and Mechanical Engineering (JMSME), Vol.2, No.5, April-June, 2015, 483-485.

3. P. Surya Nagendra, VVS Prasad, and Koona Ramji "Experimental Studies of Nano Banana Fiber Various Mechanical Properties of Laminated Epoxy Composite" Advances in Polymer Science and Technology: An International Journal 2015; 5(4): 44-50.

4. P. Surya Nagendra, VVS Prasad, and Koona Ramji "Alkali Treated Water Absorption of Banana Nano Filler/E-Glass Fiber Composites" Andhra University Journal of Engineering, Volume 2 - No.1, Jan-2016, pp no: 50-54.

Papers Published in Conferences:

1. P. Surya Nagendra, VVS Prasad, K, Ramji "Fabrication and Testing of Raw Banana Fiber as Reinforcement Material in Polyester Composites" National Conference on Advances in Materials Engineering (MATERIAUX - 2014)" Metallurgical Engineering, AUCE(A), Andhra University, 06-07 March 2014.

2. P. Surya Nagendra, VVS Prasad, K, Ramji "Effect on Mechanical Properties of Banana Nano Fibre Reinforced Polymer Composite" Twenty Eighth of Metallurgical and Materials Engineers, National Conference on "Frontiers in Material Processing" JNTU-V, January 23&24, 2015.

3. P. Surya Nagendra, VVS Prasad, and Koona Ramji "A Study on Dynamic Mechanical Analysis of Natural Nano Banana Fiber Filled Epoxy Composites" International Conference on Advancements in AeroMechanical Materials for Manufacturing (ICAAMM-2016), Elsevier Journal Materials Today Procedia. 7th-9th July 2016.