

# **DEVELOPMENT OF LOW TEMPERATURE CURED NR LATEX NANOCOMPOSITES USING NANOCELLULOSE OBTAINED FROM ISORA FIBERS :- AGEING STUDIES**

**Minor Research Project Report submitted  
To  
University Grants Commission**

**By**

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# Contents

<b>Title</b>	<b>Page No.</b>
Abstract	iii
Chapter I - Introduction and Objectives	1
Chapter II - Experimental	10
Chapter III - Characterization and Mechanical Analysis	14
Chapter IV - Results and Discussion	17
Chapter V - Conclusion	27
References	29

## **ABSTRACT**

Ecological concern now-a-days has resulted in a renewed interest in natural materials and issues such as recyclability and environmental safety are becoming increasingly important for the introduction of new materials and products. Natural fibers do have a number of ecological and economical advantages over synthetic fibers. The combination of interesting mechanical and physical properties together with environmental friendly character has triggered a number of industrial sectors to consider these natural fibres as potential candidates to replace synthetic fibres in environmentally safe products. Natural fibre reinforced polymer composites have attracted substantial interest as a potential structural material as well as in other applications in almost all industrial fields. Interest in using natural fibres as reinforcement in polymer matrices to make composites has grown rapidly in recent years for making low cost building materials, automobile components and other industrial products. This is partly due to the environmental concern and mostly due to their potential in cost reduction. Instead of natural fibers, cellulose obtained from natural fiber can also be used as the reinforcement material. In the present work we prepared natural rubber composites, reinforced with nanocellulose obtained from a natural fiber called 'Isora' and their mechanical properties were analyzed. The quality of a rubber composite is determined mainly by the curing or vulcanization temperature. Linear rubbers like NR, NBR etc get degraded when the temperature is raised. Low temperature curing results in composites of better quality. In this work composites of natural rubber reinforced with nanocellulose obtained from isora fibers were prepared with low temperature curing method and the change in their mechanical properties with soil and thermal ageing were studied.

## CHAPTER I

### INTRODUCTION

#### 1.1. NANOTECHNOLOGY

Nanotechnology literally means any technology on a nanoscale that has applications in the real world. Nanotechnology is the creation of useful/ functional materials, devices and systems through control of matter on the nanometer length scale and exploitation of novel phenomena and properties at that length scale. A nanometer is a billionth of a meter or 80,000 times thinner than human hair.

The term "nano-technology" had been coined in 1974 by Norio Taniguchi to describe semiconductor processes involving control on the order of a nanometer. Today's scientists and engineers are finding a wide variety of ways to deliberately make materials at the nanoscale to take advantage of their enhanced properties such as higher strength, lighter weight, less pollution, and greater chemical reactivity than their larger-scale counterparts. Unusual physical, chemical, and biological properties can emerge in materials at the nanoscale. These properties may differ in various ways from the properties of bulk materials to single atoms or molecules. The advantages of nanotechnology are less pollution, low production cost, mass production in food and consumables etc. Nanotechnology make it possible to manufacture lighter, stronger and programmable materials that require less energy to produce than conventional materials and promise greater fuel efficiency in land transportation, ships, aircraft and space vehicles. Nanotechnology is already in use in many computing, communication, and other electronic applications to provide faster, smaller, and more portable systems that can manage and store larger and larger amounts of information.

#### 1.2 NANOFIBERS

'Fiber' is defined as any single unit of matter characterized by flexibility, fineness and high aspect ratio. It is a slender filament that is longer than 100  $\mu m$  and the aspect ratio is greater than 10. Fibres have a fine hair like structure and they are of animal,

vegetable, mineral or synthetic origin. Fibers are broadly classified as natural and manmade or synthetic. They can be used as components of composite materials.

Natural fibers are one of the major renewable materials in the world. There are about 2000 species of useful fibre plants in various parts of the world and these are used in many applications. Natural fibers like silk, wool, cotton etc have a number of technological advantages over synthetic fibers. Nanofibers are defined as fibers with diameter in the nanometer range. A nanofiber is a continuous fiber which has a diameter in the range of billionths of a meter. The smallest nanofibers made today have diameter between 1.5 to 1.75 nanometers. The major properties of nanofibers are large specific surface area, high porosity, small pore size and diameter range 50 – 1000 nm.

### **1.3 HELICTERES ISORA FIBER**

Helicteres isora, sometimes called the Indian screw tree, is a large shrub found in Asia including Indian Subcontinent, South China, Malay Peninsula, Java Saudi Arabia and Australia. The red flowers are pollinated mainly by birds of the sunbird family. It possesses an impressive range of nutritional and medicinal properties. Fibres from the bark are used to make rope.

Isora fiber is a bast fiber separated from the bark of Helicteres isora plant by retting process. Helicteres Isora (Family-Sterculiaceae) is a shrub or small tree found in forests with stems 1-2 inches in diameter and a height of 5-15 feet. Helicteres isora dried fruit solvent extracts were evaluated for their antioxidant and anticancer activity. It is also used as a cure for snakebite. Isora resembles jute in appearance with comparable strength, better durability and luster. The properties of isora are found to be comparable to some other natural fibers. Hence high performance cost effective composites can be developed by the incorporation of isora fibers into matrices like natural rubber.

Isora fibers have a high proportion (71 %) of cellulose, which can be readily isolated from the other components namely lignin and hemicellulose by chemical treatment. The morphology and properties of the cellulose nanoparticles depend on the source of original

cellulose and the extraction process. Isora fiber due to its adequate tensile strength and good specific modulus enjoys the right potential for usage in composites.



**Figure :** Helicteres isora plant and its seeds

It is a good source of carbohydrates, proteins, fiber, calcium, phosphorus and iron. Different parts of the plant are traditionally used in the indigenous medicine system to cure various ailments. They are useful in griping of bowels, diabetes, diarrhea and dysentery. The root juice and fruits are topically applied to cure snake bite. The fruit powder mixed with some other herbs and spices is given to new mothers in order to cope with post-delivery weaknesses.

Isora fiber is a biodegradable and environmentally friendly crop and has been found to be an important source of fiber for composite and other applications. The successful development of advanced nanocomposites depends primarily on the sophisticated use of well-dispersed fillers and reinforcement such as nanocellulose, nanotubes, nanofibers etc. Such nanofillers can enhance the mechanical, electrical, thermal, flame- retardant properties of corresponding nanocomposites.

## **1.4 NANO CELLULOSE**

Nanocellulose is a light solid substance obtained from plant matter which comprises nanosized cellulose fibrils. Nanocellulose is transparent, electrically conductive and stronger than steel. Nanocellulose boost up the rapidly expanding field of sustainable materials and nanocomposites. Nano sized cellulose fillers have been found to be very promising reinforcing elements for the last few years. There are two types of nano sized cellulose, cellulose whiskers and micro fibrillated cellulose. Depending on the source, the microfibrils of cellulose may be of

several micrometers in length. Each microfibril consists of crystalline domains intermixed with disordered amorphous regions. On acid hydrolysis, the amorphous phase can be removed and obtains highly crystalline cellulose whiskers or nanocellulose. Mechanical treatments can be employed to obtain nanofibrillated cellulose (NFC). As mentioned earlier, crystalline cellulose nanowhiskers can be obtained by simple acid hydrolysis. Cellulose whiskers are also known as cellulose nanocrystals, nanorods or nanowhiskers. It can be prepared from a variety of sources, like microcrystalline cellulose , bacterial cellulose , algal cellulose (valonia) , hemp , tunicin , cotton , ramie , sisal , sugar beet , and wood .

The potential applicability of nanocellulose is widely extended. Applications of nanocellulose are mainly considered to be in paper and packaging construction, automotive, furniture, electronics, pharmacy etc. For companies producing electroacoustic devices, nanocellulose is used as a membrane for high quality sound. Additionally, nanocellulose is applied in membrane for combustible cells (hydrogen); additives for high quality electronic paper (e-paper); ultra-filtrating membranes (water purification) and membranes used to retrieve mineral and oils, and nowadays, nanocellulose has been greatly discussed and researched for a huge variety of applications . The high strength and stiffness as well as the small dimensions of nanocellulose may well impart useful properties to composite materials reinforced with these fibers, which could subsequently be used in wide range of applications.

Nanocellulose is often being regarded as the next generation renewable reinforcement for the production of high performance biocomposites. The use of natural fibers instead of traditional reinforcement materials provides several advantages. Biodegradable composites are one of the important applications. Aerogels are a new class of materials for catalysis applications and in structures for liquid storage. They have exceptional thermal insulation properties. Cellulose nanofibril suspensions can also be converted to nanopaper structures. Recent developments have shown the possibility to increase paper strength with additive of nanocellulose particles or nanofibrillated cellulose to paper compositions. Nanocellulose can also be used as a reinforcing filler to prepare composites with solutions of water soluble polymers, as well as with acrylic and other latex to change the viscosity and increase mechanical properties of dry composites. Another application is the addition of

nanocellulose to biodegradable polymers, permitting both the improvement of mechanical properties and acceleration in the rate of biodegradation. Nanocellulose in its pure state is harmless for people and is biocompatible. So it can be used for health care applications like personal hygiene products, cosmetics and biomedicine.

## **1.5 NANO COMPOSITES**

Polymer nanocomposites are of growing interest because of their significantly enhanced mechanical properties and thermal stability versus neat polymers or conventional polymer composites. Abundance, high strength and stiffness, low weight and biodegradability are some features which make nanoscale fiber cellulose materials promising candidates for bio-nanocomposite production. Progresses in the field of materials science and technology have given birth to fascinating and wonderful materials known as 'composites'. A composite material can be defined as a macroscopic combination of two or more distinct materials, having a recognizable interface between them. Composites are made up of continuous and discontinuous medium. The discontinuous medium that is stiffer and stronger than the continuous phase is called the reinforcement and the so called continuous phase is referred to as the matrix. The properties of a composite are dependent on the properties of the constituent materials, and their distribution and interaction. A composite material can provide superior and unique mechanical and physical properties because it combines the most desirable properties of its constituents while suppressing their least desirable properties. At present composite materials play a key role in aerospace industry, automobile industry and in other engineering applications as they exhibit outstanding strength to weight and modulus to weight ratio.

Polymer based composites are currently a very important and rapidly expanding field of research. The composite materials are inherently heterogeneous at a micro structural level consisting of two components having different elastic moduli, different strengths, different thermal expansion coefficients and so on. Composites are increasingly being used as a substitute for many of the conventional materials. Composites are broadly classified into natural and synthetic composites. The different components, the reinforcement



and the matrix with the distinct interface in between must have appropriate characteristics and function both individually and collectively to attain the desired superior properties of the composite. Reinforcement provides high strength and modulus. It may be in the form of fibers, particles or whiskers. Particles have no preferred direction. The particles may be metallic, ceramic, manmade or natural materials. The constituent that is continuous and often (but not always) present in greater quantity is termed as matrix. The main role of the matrix is to transmit and distribute stresses along the reinforcement phase. Durability, interlaminar toughness, shear, compressive and transverse strength are also provided by the matrix resin. Commonly used matrix materials include polymers, metals, ceramics and carbon. Most of the composites used in the industry today are based on polymer matrices.

Nanocomposite is a multiphase solid material where one of the phases has one, two or three dimensions of less than 100 nanometers, or structures having nano-scale repeat distances between the different phases that make up the material. In the broadest sense this definition can include porous media, colloids, gels and copolymers, but is more usually taken to mean the solid combination of a bulk matrix and nano-dimensional phase(s) differing in properties due to dissimilarities in structure and chemistry. The mechanical, electrical, thermal, optical, electrochemical, catalytic properties of the nanocomposite will differ markedly from that of the component materials. Nanocomposite have recently attracted significant research interest due to their improved strength and modulus, better thermal and chemical stabilities with enhanced flame retardancies. Having such improved properties make nanocomposites attractive in various applications such as automotive, food, medical etc.

## **1.6 NATURAL RUBBER COMPOSITES**

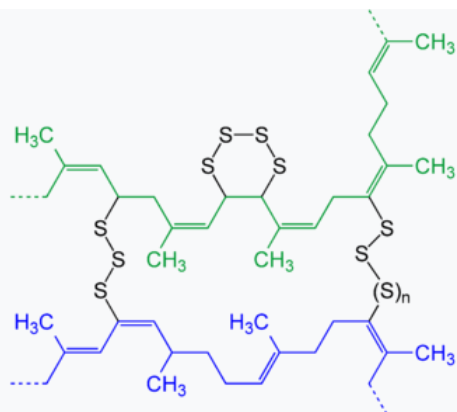
Natural rubber consists of polymers of the organic compound isoprene, water and minor impurities of some organic compounds. Rubber is harvested mainly in the form of the latex from the rubber tree. The latex is a sticky, milky colloid drawn off by making incisions in the bark and collecting the fluid in vessels in a process called "tapping". The latex then is refined into rubber ready for commercial processing. Natural rubber is used extensively in many applications and products, either alone or in combination with other materials. It has a

large stretch ratio and high resilience, and is extremely waterproof. Ammonia solution is usually used to prevent the coagulation of raw latex. Rubber exhibits unique physical and chemical properties. Due to the presence of a double bond in each repeat unit, natural rubber is susceptible to vulcanization and sensitive to ozone cracking. Rubber begins to melt at approximately 180 °C (356 °F). Vulcanization of rubber creates disulphide bonds between chains, which limits the degrees of freedom and results in chains that tighten more quickly for a given strain, thereby increasing the elastic force constant and making the rubber harder and less extensible.



**Figure:** Rubber plant and natural rubber latex

Vulcanization is a chemical process for converting natural rubber or relative polymers into more durable materials by the addition of sulphur or other equivalent accelerators. These additives modify the polymer by forming cross-links (bridges) between individual polymer chains. Vulcanized materials are less sticky and have superior mechanical properties. Uncured natural rubber is sticky, deforms easily when warm, and is brittle when cold. The reason for inelastic deformation of unvulcanized rubber can be found in its chemical structure — rubber is composed of long polymer chains. These chains can move independently relative to each other, which lets the material change shape. Cross linking introduced by vulcanization prevents the polymer chains from moving independently. As a result, when stress is applied the vulcanized rubber deforms, but upon release of the stress it reverts to its original shape.



**Figure:** Schematic presentation of two strands of natural rubber after vulcanization with elemental sulphur.

Vulcanization of rubber is a process of improvement of the rubber elasticity and strength by heating it in the presence of sulphur, which results in three-dimensional cross-linking of the chain rubber molecules (poly-isoprene) bonded to each other by sulphur atoms. Vulcanization process was invented by Charles Goodyear in 1839. Vulcanization technique comprises the following principal stages;

- Mixing of crude rubber with about 5-30% of sulphur (cross-linking agent) and other additives such as activator (commonly zinc oxide or stearic acid), accelerator (guanidines, thiazoles, dithiocarbamates, xanthates, thiurams), coagulants (acetic acid, calcium chloride), anti-oxidants (amines, phenolics, phosphites), color pigments, surfacants, softeners (oils), ant-foaming agents, anti-tack agents (Rosin derivatives, coumarone-indene resins, aliphatic petroleum resins, alkyl-modified phenol-formaldehyde resins). Slow cross-linking starts at this stage. It is necessary to avoid active vulcanization during mixing, which may cause cracks formation at the molding stage.
- Molding (shaping) the rubber mixture. The rubber must be shaped prior to heating stage since cross-linking makes shaping impossible.
- Heating the mixture to 120-200<sup>0</sup>C (250-400<sup>0</sup>F). Increased temperature speeds up the vulcanization process resulting in fast and complete cross-linking. C-S bonds replace C-H bonds linking chains of poly-isoprene molecules. Each link is formed by one to seven sulphur atoms.

By the process of vulcanization the properties of natural rubber like tensile strength, elasticity, hardness, tear strength, abrasion resistance, resistance to solvents get enhanced.

### **1.7 LOW TEMPERATURE CURING OF NATURAL RUBBER COMPOSITES**

Usually the process of vulcanization of natural rubber is done at a temperature of 120-200<sup>0</sup>C. The novel low temperature curing agents used for natural rubber are accelerators like sodium, potassium or zinc salts of ethyl, isopropyl or butyl xanthates. Literature survey shows that on evaluation of properties such as tensile strength, tear strength, crosslink density, elongation at break, and modulus at 300% elongation it can be seen that all the above mentioned xanthate accelerators are effective for room temperature curing.

### **Objectives of the work:**

The main objectives of the project are:

1. Preparation of nanocellulose from isora fibers by steam explosion process
2. Characterization of nanocellulose using the standard technique of Scanning Electron Microscopy
3. Preparation of nanocomposites by incorporating nanocellulose of variable concentration into natural rubber latex by low temperature curing method
4. Characterization of nanocomposite samples using Scanning Electron Microscopy
5. Degradation or ageing studies of nanocomposites with special reference to soil and thermal ageing using harness, specific gravity, tear strength and tensile strength measurements before and after ageing

## CHAPTER II

### EXPERIMENTAL

#### 2.1 MATERIALS USED

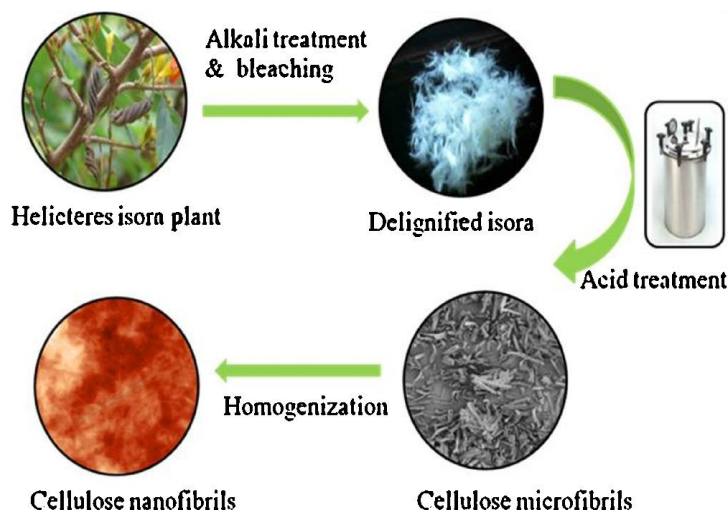
- Fibers of *Helicteres isora* were collected from the regions of Adimali and Thommankuthu, Idukki District, Kerala.
- Sodium hydroxide, glacial acetic acid, sodium hypochlorite, oxalic acid were of laboratory grade and were used without further purification.
- Natural rubber latex (60%), potassium hydroxide (10%), potassium oleate (10%) prepared from potassium hydroxide and oleic acid, sulphur (50%) , zinc diethyl carbamate -ZDC (50%) , zinc oxide (50%) , potassium butyl xanthate (50%) prepared from butanol, potassium hydroxide, and carbon disulphide.

#### 2.2 METHODS & PROCESSING

##### 2.2.1 Preparation of Isora Nanofibrils from Raw Fibers of Isora

Isora fibers were chopped into short pieces and dried in an air oven for 3-5 hours at a temperature range of 50-70<sup>0</sup>C. They are then treated with 2% NaOH in an autoclave kept under 25 psi pressure and 110<sup>0</sup>C temperature for two hours. Then the pressure was released immediately and the fibers were washed free of NaOH using water. The fibers are then kept in an air oven for 10-12 hours. Then the fibers were subjected to bleaching. Bleaching is done with a mixture of NaOH , glacial acetic acid and sodium hypochlorite (a mixture of 27 g NaOH + 73ml glacial acetic acid made up to 1 litre and 250 ml sodium hypochlorite made up to 1 litre). Bleaching was repeated six times in an autoclave for 30 minutes each. After bleaching, the fibers were thoroughly washed with water, filterd and dried. Fibers were then subjected to acid hydrolysis using 10% oxalic acid under a pressure of 25 psi in an autoclave for 15 minutes. The pressure was released immediately and the process was repeated 8 times. The fibers were taken out, washed free of acid, suspended in water and homogenized for 2 hours with a

homogenizer. The suspension was kept in an oven at 90°C till it was dry. Cellulose nanofibers were obtained in this manner and then characterized using Scanning Electron Microscopy(SEM) to study their surface morphology.



**Figure:** Procedure for extraction of isora nanofibrils

## 2.2.2 Nanocomposite preparation

The nanocomposite is prepared in the following manner:

Natural rubber latex (60%), after thorough stirring is sieved and a definite quantity of it was poured into a beaker. Label three 100 ml beakers as 1, 2, 3 respectively. The chemicals for vulcanization and low temperature curing of the composite were taken in the three breakers in the following manner. 10% KOH and 10% Potassium Oleate in Beaker 1, 50% Sulphur, 50% ZDC and 50% Zinc Oxide in Beaker 2, 50% Potassium Butyl Xanthate in Beaker 3. Add the contents in each beaker one by one to the beaker containing the natural rubber latex with thorough stirring. Wash out all the beakers using water containing little ammonia. Keep the mixture for 24 hours and after this filter the mixture using a sieve into another beaker.

Weigh out nanocellulose powders as 0.5, 1.0, 1.5 and 2.0 gram into four different beakers containing 25ml water each and homogenized in order to get 2%, 4%, 6% and 8% nanocellulose solutions respectively.

Compounding is done by first adding the 2% nanocellulose solution into the natural rubber latex containing low temperature curing reagents, with thorough stirring. This

mixture is then poured into different dry glass trays to get thin, uniform sheets. Similarly sheets of 4%, 6% and 8% nanocellulose solutions mixed with the natural rubber latex containing low temperature curing reagents were also prepared. Compounding is also done without nanocellulose solution (neat/blank). Keep the contents of the glass trays for 24 hours for drying and then taken out with the help of talc powder. Low temperature cured natural rubber latex nanocomposites using nanocellulose obtained from isora fibers are hence prepared in this manner. The samples were characterized using Scanning Electron Microscopy. Then studies of mechanical properties of nanocomposites and their ageing by soil and temperature were conducted.

The nanocomposites prepared is shown in the following figure.



**Figure :** Low temperature cured nanocomposites

### **2.2.3 Characterization of samples**

The nanocomposites prepared including the neat sample(without nanocellulose) and the composites with different compositions of nanocellulose, were characterized using Scanning Electron Microscopy (SEM) in order to study their surface morphology and the dispersion of the nanofibers.

## **2.2.4 Ageing studies**

### **1. Soil Ageing**

The nanocomposites prepared by the low temperature curing method were kept buried under soil for a period of one month to study their soil ageing or degradation. The hardness, specific gravity, tear strength and tensile strength of the nanocomposites were analysed before and after the soil ageing.

### **2. Thermal Ageing**

The nanocomposites were kept under a temperature of 170<sup>0</sup>C for one month in order to study their thermal ageing . The hardness, specific gravity, tear strength and tensile strength of the nanocomposites were analysed before and after the thermal ageing.



## **CHAPTER III**

### **CHARACTERIZATION AND MECHANICAL ANALYSIS**

#### **3.1 CHARACTERIZATION OF NANOCELLULOSE AND NANOCOMPOSITES BY SEM**

The scanning electron microscope (SEM) is capable of producing high resolution images of sample surface. In SEM, electrons are thermoionically emitted from a tungsten or lanthanum hexa-boride [LaB<sub>6</sub>] cathode filament towards an anode; alternatively electrons can be emitted via field emission [FE]. The electron beam which typically has an energy range from a few keV to 50 keV is focused by two successive condenser lenses into a beam of very fine spot size. The beam then passes through the objective lens, where pairs of scanning coils deflect the beam either linearly or in a raster fashion over a rectangular area of the sample surface. As the primary electrons strike the surface, they are inelastically scattered by atoms in the sample. Through these scattering events, the primary beam effectively spreads and fills a tear-drop-shaped volume extending about 1 µm to 5 µm into the surface. Interactions in this region lead to the subsequent emission of electrons, which are then detected to produce an image.

The surface morphological studies of nanocellulose and nanocomposites were carried out by the method as described above.

#### **3.2 MECHANICAL ANALYSIS**

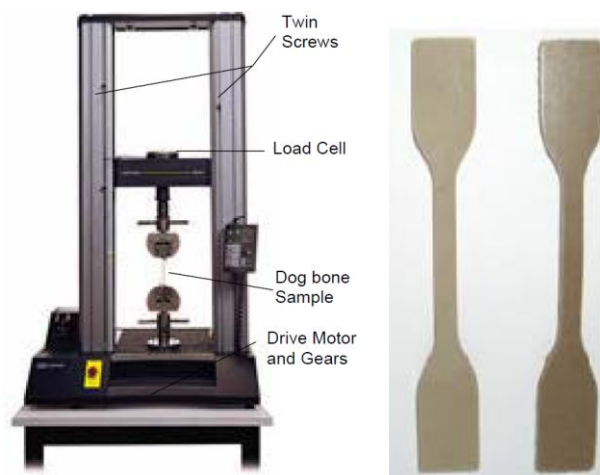
##### **3.2.1 Tensile Strength**

Ultimate tensile strength (UTS), often shortened to tensile strength (TS) or ultimate strength, is the maximum stress that a material can withstand while being stretched or pulled before failing or breaking. The UTS is usually found by performing a tensile test and recording the engineering stress Vs strain. The highest point of the stress- strain curve is the UTS. It is an intensive property; therefore its value does not depend on the size of the test specimen. However it is dependent on other factors, such as the preparation of the specimen, the presence of surface defects, the temperature of the test environment and the material. It is

defined as a stress, which is measured as force per unit area. For homogeneous materials (or for assembled components) it can be reported just as a force or as a force per unit width. In the International System of Units (SI), the unit is the Pascal or  $\text{N/m}^2$ . The measurement of mechanical properties is concerned with load deformation or stress - strain relationships.

Tensile testing, also known as tension testing, is a fundamental material science test in which a sample is subjected to a controlled tension and failure. In tensile testing, a "dog-bone" shaped sample is used. The results from the test are commonly used to select a material for an application, for quality control, and to predict how a material will react under other types of forces. Properties that are directly measured via a tensile test are UTS, maximum elongation and reduction in area. The most common testing machine used in tensile testing is the Universal Testing Machine. The test process involves placing the test specimen in the testing machine and slowly extending it until it fractures.

The stress-strain curve is unique for each material and is found by recording the amount of strain at distinct intervals of tensile or stress. These curves reveal many properties of material including modulus of elasticity. In this work three identical specimens were tested for each composition.



**Figure:** Tensile testing equipment and tensile test specimen

### **3.2.2 Tear Strength**

The tear strength of the nanocomposites was measured using an Instron-4505 (Instron Inc., United Kingdom) Universal Testing Machine at a crosshead speed of 500 mm/min, in accordance to DIN 53507 standard. At least three specimens from each batch were tested at 23°C. The averages of three thickness measurements were taken. The stress-strain graphs were plotted for various samples of different composition.



**Figure:** Tear test specimen before and after the tear test

### **3.2.3 Hardness and Specific Gravity**

Hardness is a measure of how resistant solid matter is to various kinds of permanent shape change when a compressive force is applied. The hardness measurements of the EPDM/OMMT nanocomposites were made by using Vickers method. Hardness values of the specimens were measured using a Shore-A durometer (Bariess, Germany) based on DIN 53505 standard. Hardness values of each specimen were measured at five different part of the each sample and the average of them is taken and also the standard deviations were calculated.

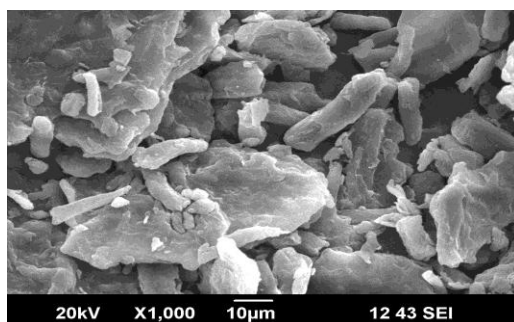
Specific gravity is a dimensionless quantity; that is, it is not expressed in units. Specific gravity is the ratio of the density of a substance to the density of a reference substance; usually the reference substance is water.

## CHAPTER IV

### RESULTS AND DISCUSSIONS

#### 4.1 CHARACTERIZATION OF NANOCELLULOSE AND NANOCOMPOSITES USING SEM

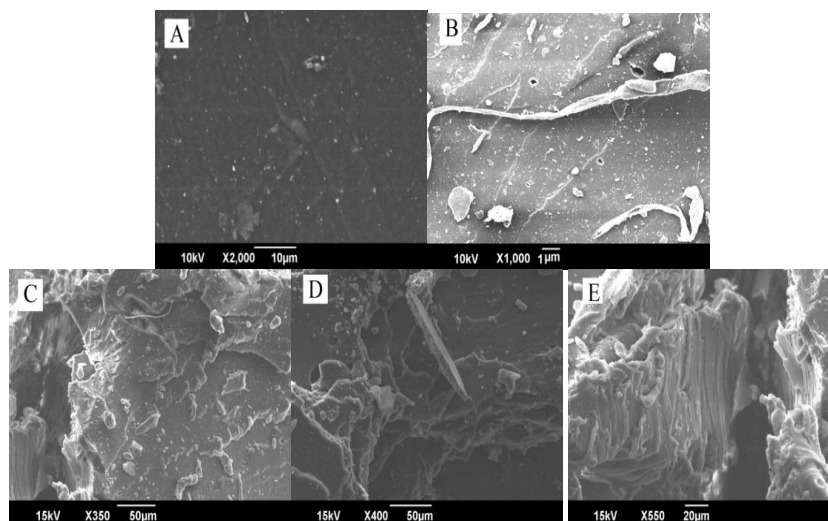
##### 4.1.1 Characterization of nanocellulose using Scanning Electron Microscopy



**Figure :** SEM image of nanocellulose

The figure shows the scanning electron microscopy image of the nanocellulose obtained from isora fibers. From the image it is clear that the obtained nanocelluloses are in cylindrical shape with diameters in the nanometer dimensions. The reduction in size is due to the removal of amorphous portions from cellulose like lignin and hemicelluloses which shows the efficiency of the steam explosion method employed for the synthesis of nanocellulose.

##### 4.1.2 Characterization of nanocomposites Scanning Electron Microscopy

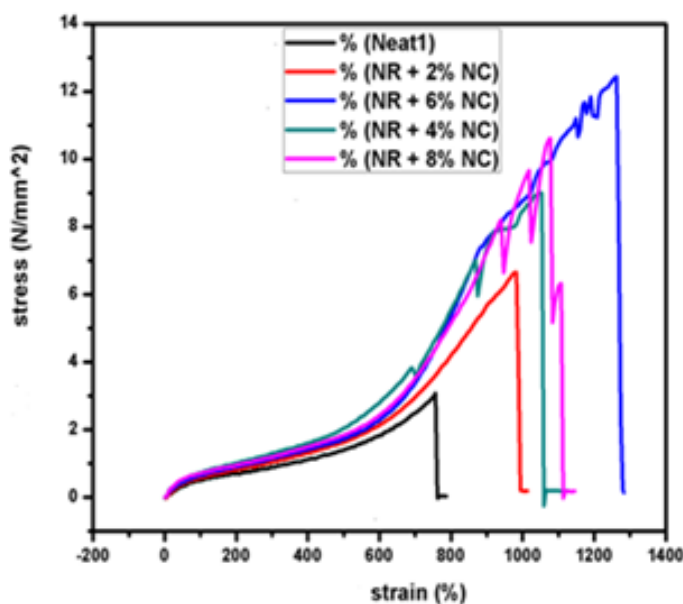


**Figure :** SEM images of composites with (A) 0% nanocellulose (neat sample) (B) 2% nanocellulose (C) 4% nanocellulose (D) 6% nanocellulose (E) 8% nanocellulose

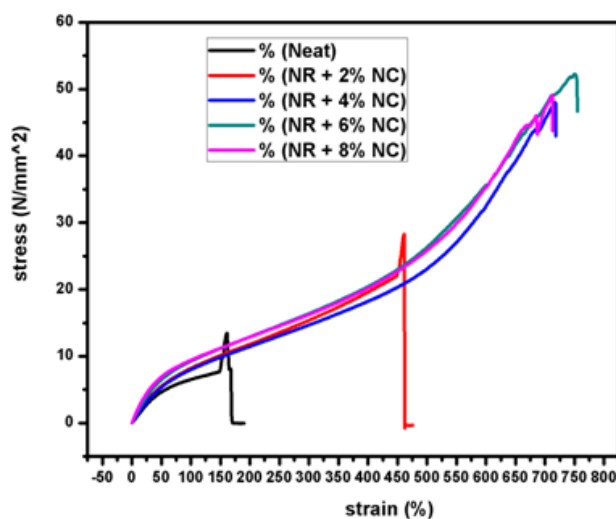
The scanning electron microscopy images of the nanocomposites with different loadings of nanocelluloses as well as that of the neat sample (composite without nanocellulose loading) are shown above. From the above images it is clear that the nanocomposite with 6% nanocellulose [SEM image 2(C)] has the better dispersion of the fillers. This is responsible for the fact that that 6% composition showed better properties like tensile strength, tear strength, hardness and specific gravity on later studies. The improvement in the property of 6% nanocomposites is due to the better dispersion of nanocellulose in the natural rubber as observed by the SEM image 2(C). The decrease in the property of 8% composition is due to the agglomeration of nanocellulose fillers as evident by the SEM image 2(D)

## 4.2 MECHANICAL ANALYSIS

### 4.2.1 Tensile Strength before ageing



#### 4.2.2 Tear Strength before ageing

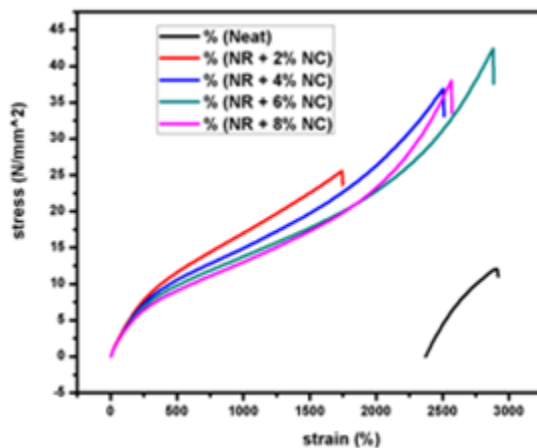


#### 4.2.3 Hardness and Specific Gravity before ageing

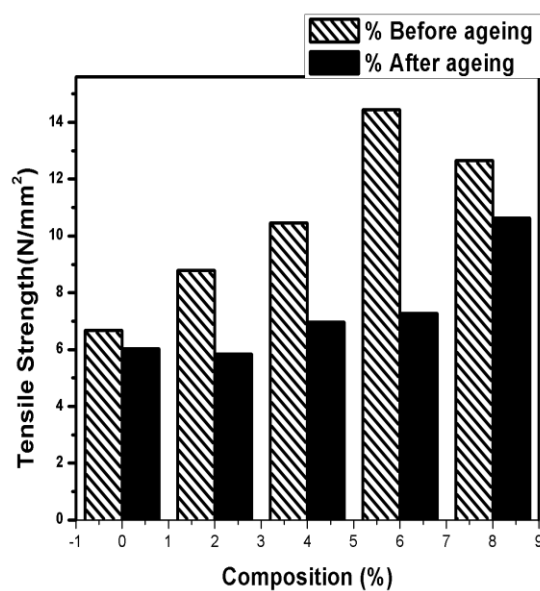
Sl. No.	Sample Name (Nanocomposites before contact with soil)	Name of Test	
		Hardness (shore A)	Specific gravity
1	Neat	49	0.918
2	NR + 2% Nano Cellulose	46	0.930
3	NR + 4% Nano Cellulose	46	0.938
4	NR + 6% Nano Cellulose	51	0.991
5	NR + 8% Nano Cellulose	50	0.945

## 4.3 SOIL AGEING STUDIES

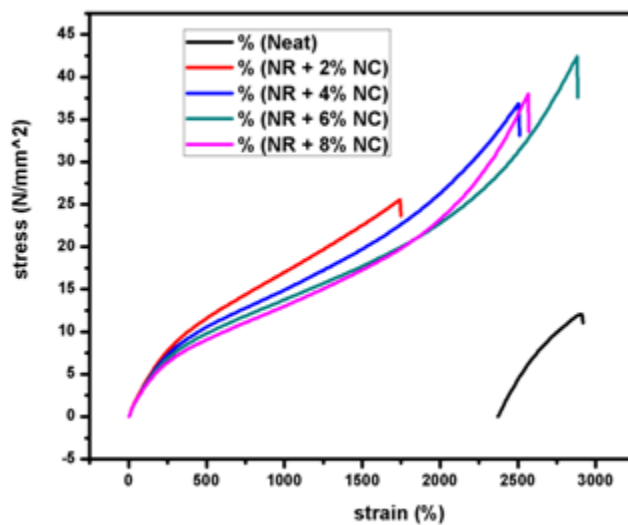
### 4.3.1 Tensile strength after soil aging



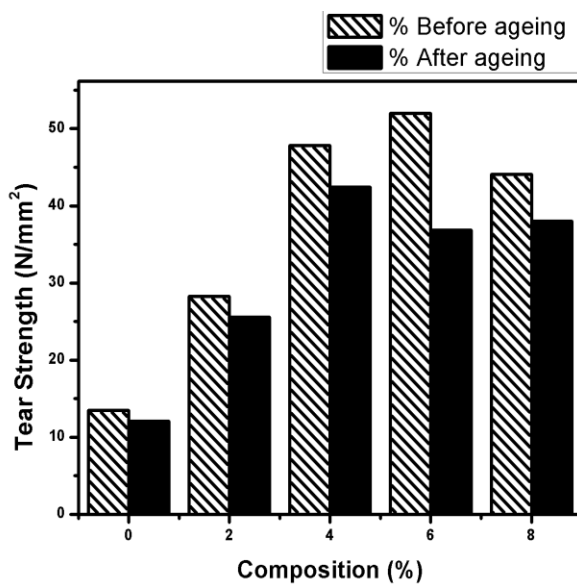
### 4.3.2 Comparison of tensile strength before and after soil ageing



### 4.3.3 Tear strength after soil aging



### 4.3.4 Comparison of tear strength before and after soil ageing





#### 4.3.5 Hardness and Specific Gravity after soil aging

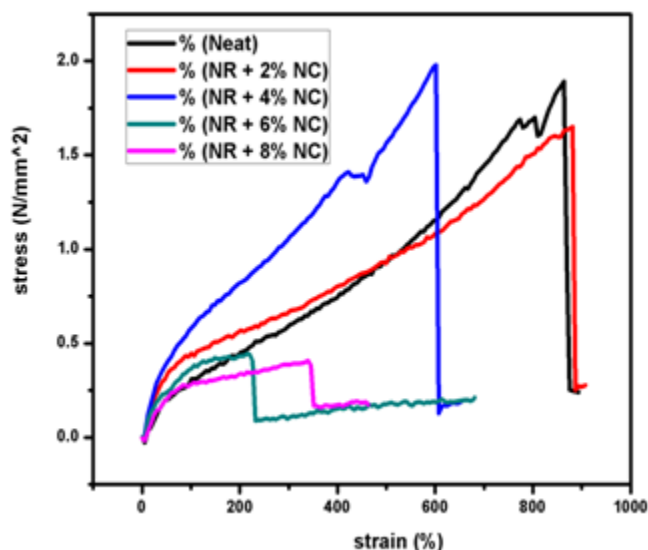
Sl. No.	Sample Name (Nanocomposites after contact with soil)	Name of Test	
		Hardness (shore A)	Specific gravity
1	Neat	22	0.914
2	NR + 2% Nano Cellulose	23	0.925
3	NR + 4% Nano Cellulose	35	0.925
4	NR + 6% Nano Cellulose	40	0.950
5	NR + 8% Nano Cellulose	37	0.935

#### 4.3.6 Comparison of Hardness and Specific Gravity before and after soil aging

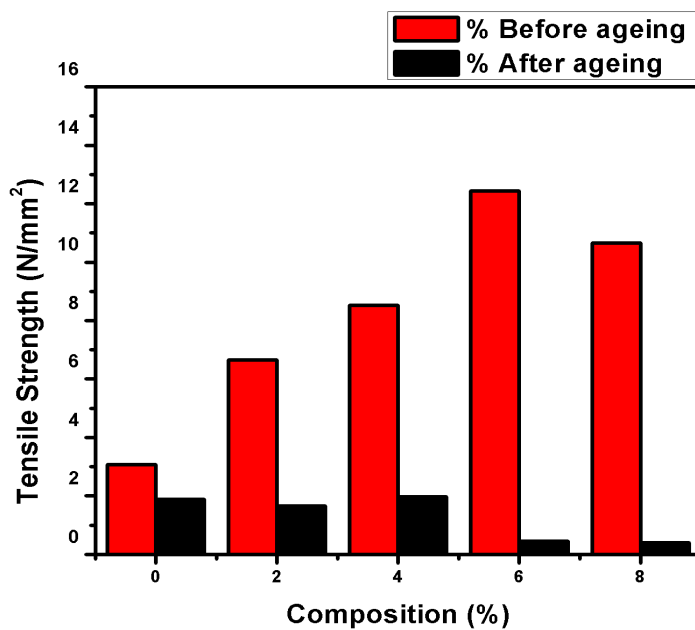
Sl. No.	Sample Name	Name of Test			
		Hardness (shore A)		Specific gravity	
		Before soil ageing	After soil ageing	Before soil ageing	After soil ageing
1	Neat	43	22	0.918	0.914
2	NR + 2% Nano Cellulose	46	23	0.930	0.925
3	NR + 4% Nano Cellulose	46	35	0.938	0.925
4	NR + 6% Nano Cellulose	51	40	0.991	0.950
5	NR + 8% Nano Cellulose	50	37	0.945	0.935

## 4.4 THERMAL AGEING STUDIES

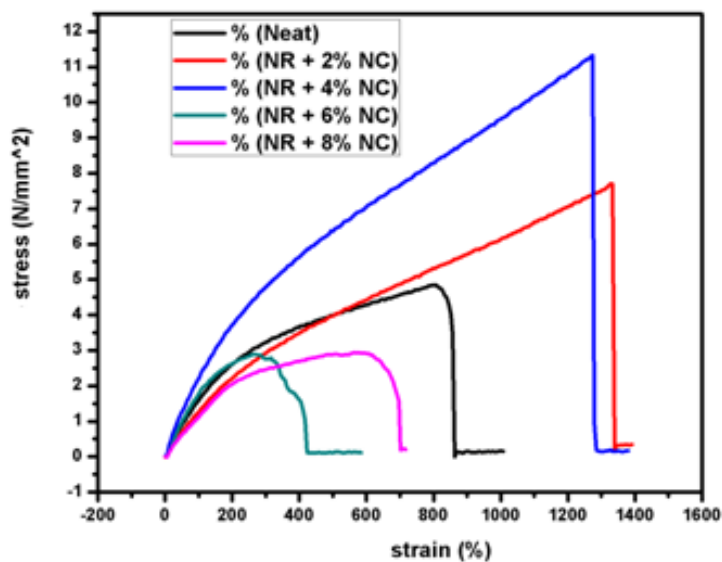
### 4.4.1 Tensile strength after thermal aging



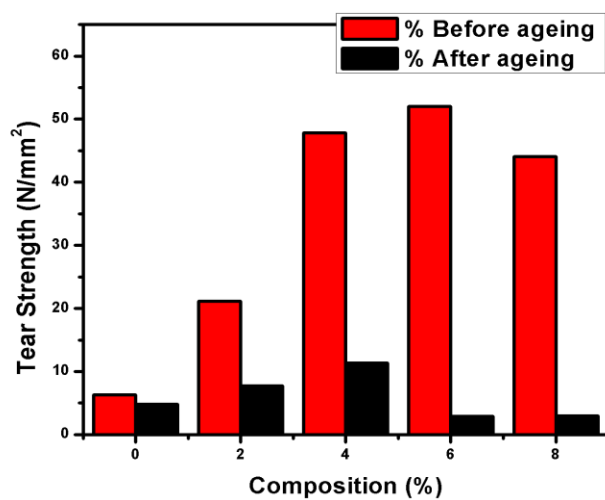
### 4.4.2 Comparison of tensile strength before & after thermal ageing



#### 4.4.3 Tear strength after thermal aging



#### 4.4.4 Comparison of tear strength before & after thermal ageing



#### 4.4.5 Hardness and Specific Gravity after thermal aging at 170 °C

Sl. No.	Sample Name (Nanocomposites after thermal ageing)	Name of Test	
		Hardness (Shore A)	Specific Gravity
1	Neat	15	0.910
2	NR + 2% Nano Cellulose	14	0.923
3	NR + 4% Nano Cellulose	16	0.928
4	NR + 6% Nano Cellulose	13	0.944
5	NR + 8% Nano Cellulose	13	0.933

#### 4.4.6 Comparison of Hardness and Specific Gravity before & after thermal ageing

Sl. No.	Sample Name	Name of Test			
		Hardness (shore A)		Specific gravity	
		Before thermal ageing	After thermal ageing	Before thermal ageing	After thermal ageing
1	Neat	43	12	0.918	0.910
2	NR + 2% Nano Cellulose	46	14	0.930	0.923
3	NR + 4% Nano Cellulose	46	16	0.938	0.928
4	NR + 6% Nano Cellulose	51	13	0.991	0.944
5	NR + 8% Nano Cellulose	50	13	0.945	0.933

As the natural rubber (NR) is reinforced with nanocellulose, there occurs some interactions between the nanocellulose fillers and the matrix also with the cross linking agents

that consequently increase the mechanical properties of the composites. A three dimensional network of nanocellulose will be formed within the composite entity. Due to this three dimensional network, there is an increase in the mechanical properties of the composite at all mixing ratios compared to that of neat. It is evident that mechanical properties of nanocomposites increases as the composition increases from 2% to 6%, but when the composition is 8% the mechanical properties shows a decrease in values. The improvement in the property of 6% nanocomposites is due to the better dispersion of nanocellulose in the natural rubber as observed from the SEM analyzis and the decrease in the property of 8% composition is due to the agglomeration of nanocellulose fillers as evident by the SEM analysis.

The nanocomposites after being kept buried under soil for a period of one month, shows decrease in the values of all the mechanical properties like hardness, specific gravity, tear strength and tensile strength than those before soil ageing. Also the composites with nanocellulose loadings show much more decrease in the values than the neat composite (without nanocellulose fillers). This indicates that the nanocomposites shows much improved degradation property than the neat composite (without nanocellulose fillers) for a long time exposure to soil and hence they proved to be ecofriendly biodegradable materials.

By analyzing the mechanical properties like hardness, specific gravity, tear strength and tensile strength of the nanocomposites after being kept at a temperature of 170<sup>0</sup>C for a period of one month, shows decrease in values than for those before thermal ageing. This indicates that the nanocomposites have undergone degradation. It may be due to the decomposition of xanthate (the accelerator as well as the low temperature curing agent used in the preparation of composites) which is usually stable at low temperatures. The decreased mechanical properties of composites at high temperatures might also be due to the breakage of S-S cross linking between the polymer chains. It was noted that the composites with nanocellulose loadings do not show much notable decrease in the values of mechanical properties than the neat composite (without nanocellulose fillers).

## CHAPTER V

### CONCLUSION

Interest in natural fiber reinforced polymer composites has grown rapidly in recent years for making low cost building materials, automobile components and other industrial products due to their potential in cost reduction and due to the environmental concern. The development of cellulose nano fibers (CNFs) as reinforcement in nanocomposites has attracted significant interest in these decades due to their unique characteristics like abundance, high strength and stiffness, low weight and biodegradability. The morphology and properties of the cellulose nano fibers depend on the source of the original cellulose and the extraction process. In this work, nanocellulose was obtained from a natural fiber called 'Isora' through the process of steam explosion. Isora is a bast fiber separated from the bark of *Helicteres Isora* plant, a shrub found in forests. Isora is a biodegradable and eco-friendly fiber that can be used as fillers in composites as well as a source of nanocellulose. Polymer nanocomposites are of growing interest due to their enhanced mechanical and thermal stability compared to the neat polymers or conventional polymer composites. Composites based on natural rubber are used extensively in many applications. Natural rubber composites are made more durable by a process called vulcanization. Vulcanization prevents the polymer chains from moving independently by introducing three dimensional cross-linking between the polyisoprene chains. As a result, when stress is applied, even though the rubber deforms, it reverts to its original shape once the stress is released. The quality of a rubber composite is determined mainly by the curing or vulcanization temperature. Usually the process of vulcanization is done at a temperature of 120-200<sup>0</sup>C. But linear rubbers like NR, NBR etc get degraded at high temperatures. Hence recent researches are behind reducing the curing temperature of composites as low temperature curing can result in composites of better quality. In the present work the process of vulcanization is done by a room temperature curing method using suitable

stabilizers, surface active agents, vulcanizing agents, fillers , activators and a special curing agent potassium butyl xanthate.

Nanocellulose was prepared from isora fibers by steam explosion process and was characterised using Scanning Electron Microscopy which confirmed the formation of cellulose fibers with diameter in the range of nanometres. Nanocomposites of natural rubber with different concentration of fillers like 0% nanocellulose (neat), 2% nanocellulose, 4% nanocellulose, 6% nanocellulose and 8% nanocellulose were prepared using the low temperature curing method that uses potassium butyl xanthate as the curing agent. The nanocomposite samples were characterized using Scanning Electron Microscopy to analyse the dispersion of nanocellulose in the natural rubber matrix. The SEM analysis showed that the nanocomposite with a composition of 6% nanocellulose has better dispersion of nanocellulose in the natural rubber matrix. Mechanical properties of the nanocomposite samples like hardness, specific gravity, tear strength and tensile strength were also analysed. All the properties showed maximum value for the composition of 6% nanocellulose. The decrease in values for the 8% composition may be due to the agglomeration of fillers as evident from the SEM image. The biodegradability of the prepared nanocomposites was studied by subjecting one set of samples to soil degradation and another set to thermal degradation at 170<sup>0</sup>C, both for a period of one month and then their mechanical properties like hardness, specific gravity, tear strength and tensile strength were analyzed again. It was noted that the composites with nanocellulose loadings do not show much notable decrease in the values of mechanical properties than the neat composite (without nanocellulose fillers).

## **Acknowledgement:**

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## REFERENCES

1. Chirayil, C. J., Joy, J., Mathew, L., Mozetic, M., Koetz, J., & Thomas, S. (2014). Isolation and characterization of cellulose nanofibrils from *Helicteres isora* plant. *Industrial Crops and Products*, 59, 27-34.
2. Lovely Mathew, P., Thomas, S., & Joy, J. Development of polymer bio nanocomposites using nanocellulose obtained from isora fibres.
3. Chirayil, C. J., Joy, J., Mathew, L., Koetz, J., & Thomas, S. (2014). Nanofibril reinforced unsaturated polyester nanocomposites: morphology, mechanical and barrier properties, viscoelastic behavior and polymer chain confinement. *Industrial Crops and Products*, 56, 246-254.
4. Mathew, L., Joy, J., Thomas, S., & Pilla, S. Development and characterization of cellulose nanofiber reinforced poly (butylene succinate) bio nano composites.
5. Joy, J., Mathew Lovely, P., Rojith, G., Singh, I. B., & Thomas, S. (2013, November). Development and Characterization of high performance of Natural Rubber nanocomposites using biologically prepared Isora nanofibers. In *First International Conference on Resource Efficiency in Interorganizational Networks-ResEff 2013* (p. 143).
6. Abraham, E., Pothan, L. A., & Thomas, S. (2012, June). Preparation and characterization of green nanocomposites based on cellulose nanofibre and natural rubber latex. In *15th European Conference on Composite Materials, Venice, Italy*.
7. Abraham, E., Deepa, B., Pothan, L. A., John, M., Narine, S. S., Thomas, S., & Anandjiwala, R. (2013). Physicomechanical properties of nanocomposites based on cellulose nanofibre and natural rubber latex. *Cellulose*, 20(1), 417-427.
8. Thomas, S., Chan, C. H., Pothan, L. A., Rajisha, K. R., & Maria, H. (Eds.). (2013). *Natural Rubber Materials: Volume 1: Blends and IPNs* (Vol. 7). Royal Society of Chemistry.
9. Jacob, M., Thomas, S., & Varughese, K. T. (2004). Mechanical properties of sisal/oil palm hybrid fiber reinforced natural rubber composites. *Composites Science and Technology*, 64(7), 955-965.
10. Ismail, H., Edyham, M. R., & Wirjosentono, B. (2002). Bamboo fibre filled natural rubber composites: the effects of filler loading and bonding agent. *Polymer testing*, 21(2), 139-144.
11. Ismail, H., Shuhelmy, S., & Edyham, M. R. (2002). The effects of a silane coupling agent on curing characteristics and mechanical properties of bamboo fibre filled natural rubber composites. *European Polymer Journal*, 38(1), 39-47.
12. Palaty, S., & Joseph, R. (2006). Low temperature curing of NBR for property improvement. *Journal of elastomers and plastics*, 38(3), 199-209.



13. Sasidharan, K. K., Joseph, R., Palaty, S., Gopalakrishnan, K. S., Rajammal, G., & Pillai, P. V. (2005). Effect of the vulcanization time and storage on the stability and physical properties of sulfur-prevulcanized natural rubber latex. *Journal of applied polymer science*, 97(5), 1804-1811.
14. Sasidharan, K. K., Palaty, S., Gopalakrishnan, K. S., George, K. E., & Joseph, R. (2004). Room temperature prevulcanization of natural rubber latex using xanthate. *Journal of applied polymer science*, 94(3), 1164-1174.
15. Palaty, S., Devi, P. V., & Joseph, R. (2011). Use of sodium and potassium butyl xanthate as accelerator for room temperature prevulcanization of natural rubber latex. *Journal of Applied Polymer Science*, 122(2), 1325-1332.
16. Shelley, J. S., Mather, P. T., & DeVries, K. L. (2001). Reinforcement and environmental degradation of nylon-6/clay nanocomposites. *Polymer*, 42(13), 5849-5858.
17. Sinha Ray, S., Maiti, P., Okamoto, M., Yamada, K., & Ueda, K. (2002). New polylactide/layered silicate nanocomposites. 1. Preparation, characterization, and properties. *Macromolecules*, 35(8), 3104-3110.
18. Shang, S. W., Williams, J. W., & Söderholm, K. J. (1994). How the work of adhesion affects the mechanical properties of silica-filled polymer composites. *Journal of materials science*, 29(9), 2406-2416.
19. Rong, M. Z., Zhang, M. Q., Pan, S. L., Lehmann, B., & Friedrich, K. (2004). Analysis of the interfacial interactions in polypropylene/silica nanocomposites. *Polymer International*, 53(2), 176-183.
20. Jordan, J., Jacob, K. I., Tannenbaum, R., Sharaf, M. A., & Jasiuk, I. (2005). Experimental trends in polymer nanocomposites—a review. *Materials science and engineering: A*, 393(1), 1-11.
21. Rajkumar, K., Kumari, N., Ranjith, P., Chakraborty, S. K., Thavamani, P., Pazhanisamy, P., & Jeyanthi, P. (2011). High temperature resistance properties of NBR based polymer nanocomposites. *Int. J. Chem. Tech. Res*, 3(3), 1343-1348.
22. Reynaud, E., Jouen, T., Gauthier, C., Vigier, G., & Varlet, J. (2001). Nanofillers in polymeric matrix: a study on silica reinforced PA6. *Polymer*, 42(21), 8759-8768.
23. Wu, C. L., Zhang, M. Q., Rong, M. Z., & Friedrich, K. (2002). Tensile performance improvement of low nanoparticles filled-polypropylene composites. *Composites Science and Technology*, 62(10), 1327-1340.

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## Synthesis, Characterization and Nano filtration Performance Studies of Nanocellulose Filter Paper

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### Abstract

Nano cellulose is a light solid substance obtained from plant matter which comprises nanosized cellulose fibrils. Nano cellulose is transparent, electrically conductive, and stronger than steel. For the preparation of nanocellulose, Isora fibers can be used. Isora is a bast fiber presented in the bark of Helicteres Isora plant. Preparation of the nanocellulose includes processes like alkali treatment with 2% NaOH in magnetic stirrer), bleaching (using sodium hypochlorite, glacial acetic acid and NaOH in autoclave), oxalic acid treatment, and homogenization. The characterization of the fiber has been done by SEM and XRD analysis. Nano filter paper is prepared as thin films. The dye adsorption studies of the nanocellulose filter papers were done by colorimetry and uv-visible spectrophotometry using dye solutions of crystal violet, methyl orange and Eriochrome black T. Nanocellulose filter paper prepared will show high dye adsorption properties than Whatman No.40 filter paper and hence it is concluded that nanocellulose filter papers could be effectively used for the removal of dye pollutants from polluted water.

### Reference

- 1, Rheological behavior of nanocellulose reinforced unsaturated polyester nanocomposites. Chirayil, Cintillose ; Mathew Lovely ; Hassan PA ; Mozetic Miran ; Thomas, Sabu 2014-08-01.
- 2, Isolation and characterization of cellulose Nano fibrils from Helicteres isora plant . CJ chirayil, J joy, L Mathew, M Mozetic, J Koetz, S Thomas, Industrial



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### Antimicrobial Studies of Zn (II) Complexes of Few 2-Methoxy-4-Chromanone Derivatives

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3-Formylchromanones (4-oxo-4H-1-benzopyran-3-carboxaldehyde) are frequently for the synthesis of various heterocyclic derivatives ever since its convenient synthesis was reported by Nohara et al. Metal complexes bearing benzopyranone moiety like chromones, flavanoids, chromanones etc are of considerable interest due to its applications in wide range of fields. However, less literature is available about the 4-chromanone metal complexes. In the present study we report the synthesis of a series of Zn (II) complexes incorporating 2-alkoxy-4- chromanones which were derived from 3-formylchromanones and 2-aminopyridines. In vitro anti bacterial and anti fungal activities of the ligand and its complexes were tested against bacterial species *Staphylococcus aureus* (MTCC 96), *Bacillus subtilis* (MTCC 121), *Pseudomonas aeruginosa* (MTCC 741) and *Escherichia coli* (MTCC 1652) and *Candida albicans* (ATCC 10231). The results indicate that activity of the compounds were enhanced by complexation.

**Key words:** Antimicrobial studies, heterocyclic, flavanoids, 4-chromanone

### Mechanical Analysis and Ageing Studies of Room Temperature Cured Nanocellulose Reinforced Natural Rubber Composites

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Nanocellulose has gained considerable attention in recent years due to its outstanding mechanical and chemical properties. The development of cellulose nanofibers (CNFs) and nano composites has attracted significant interest in the last few decades due to their unique characteristics. Because of increasing



### Performance of CdTe Quantum Dot Sensitized TiO<sub>2</sub> Solar Cells in Polysulfide and Cobalt Complex electrolytes

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The present paper gives a comparative study on the performance of TiO<sub>2</sub>-CdTe quantum dot (QD) system in polysulfide and cobalt complex electrolytes for photo voltaic applications. Electrolytes for quantum dot sensitized solar cells (QDSSCs) have been a major issue which is still not resolved despite promising theoretical efficiencies offered by quantum dot-based solar cells. TiO<sub>2</sub> nanoparticles and CdTe QDs were synthesized by spray pyrolysis deposition (SPD) and hydrothermal route, respectively. CdTe QDs were deposited on TiO<sub>2</sub> films by heat-assisted drop-casting method. The TiO<sub>2</sub> nanoparticles and the CdTe QDs were characterized by spectroscopy and microscopy, respectively. Under standard 1 Sun illumination (100 mW/cm<sup>2</sup>, AM 1.5G condition), efficiencies of cells of area 0.20

environmental awareness and the international demand for green technology, bio nano composites have the potential to replace present petrochemical based materials. In the present work, nanocomposites were prepared by incorporating nanocellulose prepared from natural isora fiber into natural rubber latex. The process of vulcanization is done by room temperature curing using suitable stabilizers, surface active agents, vulcanizing agents, accelerators, fillers and activators. The biodegradability of the prepared nanocomposites was studied by keeping the samples buried in soil for one month. While comparing with neat composite, nanocomposite reinforced with nanocellulose were more eco-friendly and they showed much improved degradation property. Low temperature curing method offers a much more convenient way of synthesizing vulcanized composites.

**Keywords:** room temperature curing, nanocellulose, isora

**Minor Research Project Report submitted  
To  
University Grants Commission**

**"Development of low temperature cured NR latex nanocomposites  
using nanocellulose obtained from isora fibers :- ageing studies"  
[MRP(S)-333/12-13/KLMG037/UGC-SWRO Dated 23-Sep-2013]**

**Summary of Findings**

Interest in natural fiber reinforced polymer composites has grown rapidly in recent years for making low cost building materials, automobile components and other industrial products due to their potential in cost reduction and due to the environmental concern. The development of cellulose nano fibers (CNFs) as reinforcement in nanocomposites has attracted significant interest in these decades due to their unique characteristics like abundance, high strength and stiffness, low weight and biodegradability. The morphology and properties of the cellulose nano fibers depend on the source of the original cellulose and the extraction process. In this work, nanocellulose was obtained from a natural fiber called 'Isora' through the process of steam explosion. Isora is a bast fiber separated from the bark of Helicteres Isora plant, a shrub found in forests. Isora is a biodegradable and eco-friendly fiber that can be used as fillers in composites as well as a source of nanocellulose. Polymer nanocomposites are of growing interest due to their enhanced mechanical and thermal stability compared to the neat polymers or conventional polymer composites. Composites based on natural rubber are used extensively in many applications. Natural rubber composites are made more durable by a process called vulcanization. Vulcanization prevents the polymer chains from moving independently by introducing three dimensional cross-linking between the poly-isoprene chains. As a result, when stress is applied, even though the rubber deforms, it reverts to its original shape once the stress is released. The quality of a rubber composite is determined mainly by the curing or vulcanization temperature. Usually the process of vulcanization is done at a temperature of 120-200<sup>0</sup>C. But linear rubbers like NR, NBR etc get degraded at high temperatures. Hence recent researches are behind reducing the curing temperature of composites as low temperature curing can result in composites of better quality. In the present work the process of vulcanization is done by a room temperature curing method using suitable stabilizers, surface active agents, vulcanizing agents, fillers, activators and a special curing agent potassium butyl xanthate.



Nanocellulose was prepared from isora fibers by steam explosion process and was characterised using Scanning Electron Microscopy which confirmed the formation of cellulose fibers with diameter in the range of nanometres. Nanocomposites of natural rubber with different concentration of fillers like 0% nanocellulose (neat), 2% nanocellulose, 4% nanocellulose, 6% nanocellulose and 8% nanocellulose were prepared using the low temperature curing method that uses potassium butyl xanthate as the curing agent. The nanocomposite samples were characterized using Scanning Electron Microscopy to analyse the dispersion of nanocellulose in the natural rubber matrix. The SEM analysis showed that the nanocomposite with a composition of 6% nanocellulose has better dispersion of nanocellulose in the natural rubber matrix. Mechanical properties of the nanocomposite samples like hardness, specific gravity, tear strength and tensile strength were also analysed. All the properties showed maximum value for the composition of 6% nanocellulose. The decrease in values for the 8% composition may be due to the agglomeration of fillers as evident from the SEM image. The biodegradability of the prepared nanocomposites was studied by subjecting one set of samples to soil degradation and another set to thermal degradation at 170<sup>0</sup>C, both for a period of one month and then their mechanical properties like hardness, specific gravity, tear strength and tensile strength were analyzed again. It was noted that the composites with nanocellulose loadings do not show much notable decrease in the values of mechanical properties than the neat composite (without nanocellulose fillers).

**SONA JOHN**

**PRINCIPAL INVESTIGATOR**