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Studies on Nano Cellulose Century Fiber Composites

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ABSTRACT
Nano cellulose composites are used for advanced applications for structural parts and electronic components. Biocompatible water soluble cellulose composites are used in medical applications as well. Nano cellulose fibers are extracted through various chemical and mechanical treatments to separate the cellulose and to further refine it. Composites are made using thermosetting polyester resin and biodegradable poly vinyl (PVA). A research work is proposed in this paper for extraction of lignocellulose and nano cellulose fibers from century plant and developing the composites for evaluation of TGA, DSC, DMA, dielectric, tensile, flexural, impact, hardness and hygroscopic properties. Applications for the composites will be suggested based on the the properties of the composites.

Keyword: *Century fiber composites; nanocellulose fibers; nano cellulose composites; biodegradable composites.*

I. INTRODUCTION
Nano-cellulose composites have drawn the attention of the researchers for development thermosetting and biodegradable materials for automotive, packaging and medical applications [1-10]. Nano cellulose fiber composites are fully biodegradable and biocompatible with excellent mechanical properties. Due to high crystallinity and high aspect ratio and low density of the nano cellulose fibers, there is considerable increase in the stiffness of the composites produced.

II. LITERATURE REVIEW
The comparative study [1-3] of mechanical, thermal, electrical and water absorption properties of different natural fiber composites has identified that the Century fiber has great potential to give better properties. There has been an increasing interest on use of natural fiber composites over the last decade and extensive research has been carried out to explore the best properties for various applications [4, 5]. Nano cellulose fibers are isolated from a variety of natural fibers by chemical and mechanical treatments [5]. Chemical treatments prior to mechanical treatments reduce the size of the fibers before homogenization by [6] and reduce the energy consumption during mechanical treatments. Different chemical treatments include: alkaline treatment coupled with high pressure defibrillation, acid treatment, enzyme-assisted hydrolysis and acid hydrolysis. Mechanical treatments include: high pressure homogenization, ultrasonication, cryocrushing and grinding. Isolation of nano fibers is assisted by oxidation pretreatment by 2-Tetramethylpiperidine-1-oxyl (TEMPO) that facilitates. Other methods include steam explosion and electro- spinning [7-11]. Nano fibrillated cellulose (NFC) reinforced composites are produced using phenolic resin.

Styrene butyl acryl ate amyl pectin, polyurethane, melamine formaldehyde, etc. Nano composites are made by hand layup technique using bio-based epoxy resin and TEMPO oxidized NFC. The specimens are investigated for mechanical, dynamic mechanical, thermal [12] and dielectric properties as well as humidity absorption, morphology and transparency of the composites [6]. Different biodegradable polymers used are: PEO-poly (ethylene oxide), PVA-poly (vinyl alcohol), and PAA- poly (acrylic acid), PCL-poly (ε-caprolactone), PLA- poly (lactic acid), PS-polystyrene, EVOH-ethylene-vinyl alcohol copolymer, PMMA-poly (methyl methacrylate) [12, 14]. Thermoplastic rice straw nano cellulose composites are made using reinforced starch polymer [15]. In the first step, almost all the non-cellulosic components are removed from the straw and a white pulp of cellulosic fibers are obtained. Then a diluted

suspension of fibers was ultra-sonicated to destroy inter molecular hydrogen bonds and nano fiber networks are obtained. The fibers are then used for casting films of the composite. It was found that the yield strength and Young's modulus of the nano composites is due to the reinforcement by cellulose fibers. Humidity Absorption resistance was significantly enhanced.

Recently, modified cellulose has been used as reinforcement for various composites with water soluble polymers. Addition of cellulose increased the viscosity and mechanical properties and accelerated the rate of biodegradation. Chemical modification of cellulose has been an important route for the production of multifunctional materials. High strength biodegradable composite films for membrane and packaging applications are developed by film casting method using modified cellulose with poly(vinyl alcohol) in different compositions [8,15,16]. These films are characterized for mechanical, moisture absorption, gas barrier, and biodegradable properties [16,17]. They have shown good transparency, flexibility, good mechanical and biodegradable properties. These films have exhibited better barrier properties with increase in percentage of modified cellulose. Literature review revealed that reported research has not been found on Century cellulose nano fiber composites. Century fibers will be extracted from Agave Americana plants are abundantly available as border plant. The century fibers are widelyused in the textile and paper industry. The century nano cellulose fibers will be used to cast various structural parts in Automobiles, electronic and packaging industry.

III. PROPOSED WORK

The proposed research work is to produce wealth from waste by developing useful products from desert plants. By extracting nano cellulose fibers from leaves of Century plant. Century nano cellulose fiber sheets are molded using thermoset polyester resin and also poly (vinyl alcohol), a biodegradable resin.

The composites will be tested for various mechanical properties such as: flexural, tensile, fracture, impact, Barcol hardness and water absorption and dielectric breakdown properties. Thermal properties like, glass transition temperature and thermal degradation will be studied using Differential Scanning Calorimetry and Thermogravimetric Analysis respectively. Scanning Electron Microscopy will be done to study the fracture behavior of the composite. Based on the results of the studies, the scope of application of the material will be suggested.

IV. PREPARATION OF NANO CELLULOSE COMPOSITES

A. Century Plant

Century (Agave Americana) plant belongs to Agavaceae family and is native to Mexico and its name is derived referring to the long time it takes to flower. These plants grow in clusters and are generally used for fencing. Their leaves are 2 m long and 25 cm wide. They are located as a rosette without trunk. Century fiber has drawn attention by several researchers because of their large leaf length, leaf biomass, fiber length, fineness, density and high strength.

B. Extraction of Century Fibers

The lignocellulosic fibers from Century plant are produced from leaves by retting process. The leaves are cut and dried to allow the watery substance to evaporate and then soaked in still water for 15 days. The fermented soft greenish substance is washed thoroughly and the fibers are peeled off the leaves and are washed and dried in shady place. The length of the fibers is between 100-123 cm and the size ranges between 150µ m to 300 µm.

C. Extraction of Cellulose [16,17]

Cellulose is extracted by a process called water pre-hydrolysis [10]. The fibers which are of 1-1.25 m long are cut into approximate length of 5-10mm. The chopped fibers are dewaxed where a mixture of Toulene/ethanol (2:1 vol/vol) is poured in flask and the fibers were put in a cloth and placed in the Soxhlet extractor and boiled at a temperature of 70°C for 6 hours. They are washed with ethanol for 30 min and then allowed to dry. The de-waxed fibers are then mixed with 0.1M NaOH in 50% volume of ethanol at 45°C for 3 hours under continuous stirring by keeping the beaker on a magnetic stirrer. Then the fibers are treated with H_2O_2 at pH=10.5(buffer solution) is carried out at 45°C in a solution of $H_2O_2$with different concentrations, viz., (a)0.5% H_2O_2 (b)1% H_2O_2 (c)2% H_2O_2 (d)3% H_2O_2 for 3 hours each under continuous agitation. Then each mixture is treated with 10% w/v NaOH + 1% w/v $Na_2B_4O_7.10H_2O$ at 32°C for 15 hours under continuous stirring. Then, solid obtained will be filtered off. Salt formation will be confirmed by solubility test, since it is freely soluble in water. This

salt will be treated with 2-(Triflurомethyl) benzoylchloride in the presence of pyridine as a base cum Solvent and stirred overnight at 100˚C.

At the end of the process, only cellulose will be left which is then washed with 95% ethanol and then water and dried at 60°C in an oven until the weight remains constant. Then the fibers are ground to obtain in the form of a powder. Important stages are illustrated in Fig. 1.

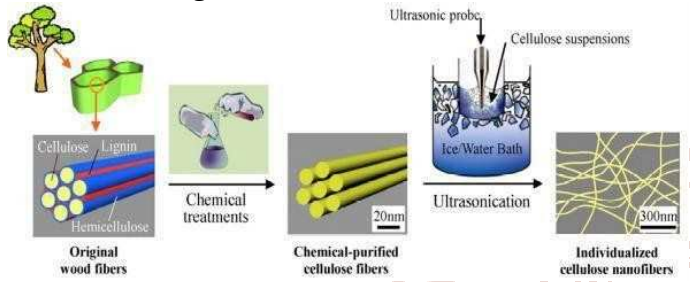

Fig.1. Different stages of extraction of nano cellulose fibers

D. Casting of Biodegradable Composite Films[9]

Modified cellulose of 2-(Triflurомethyl) benzoylchloride will be taken in water along acetonitrile as purifying solvent and Poly(vinyl alcohol) as matrix in different proportions as: 10 : 90, 20 : 80, 30 : 70, 40 : 60, 50 : 50, 60 : 40, 70 : 30, 80 : 20, 90 : 10, and 95 : 05 ratios. The reaction mixture was heated to 100◦C for 24 hrs. After 24 hrs, the reaction mass will turn to viscous state, it will be allowed to reach room temperature and spread on the Teflon mold of one square feet with 3mm depth. The mold will be coated with mold releasing spray and dried under vacuum oven at 100˚C to remove water contents completely. After complete drying of the cast films, they are stored in moisture free environment.

V. EVALUATION OF PROPERTIES

A. Preparation of Specimens and Testing

Preparation of specimens for flexural test tensile test Impact test hardness, moisture absorption test will be done as per corresponding ASTM standards. For morphology studies, Scanning Electron Microscope (SEM) was used.

B. Flexural test, flexural strength, flexural modulus

The flexural strength (N/m^2) of a material is defined as the ability to resist deformation under transverse loads. Flexural properties will be evaluated as per ASTM D-790-03 through three-point bend test on compression testing machine supplied by Hydraulic and Engineering Instruments, New Delhi, at a cross head speed of 1.25 mm/minute, at standard laboratory atmosphere of 23°C ± 2°C (73.4°F ± 3.6°F) and 50 ± 5 percent relative humidity. Specimens for flexural test are cut from laminas as per ASTM D790.

Flexural Strength: Flexural strength is the maximum stress in the outer surface of the specimen at the moment of break. When the homogeneous elastic material is tested with three- point system, the maximum stress occurs at the midpoint.

Flexural Modulus: Flexural modulus (N/m^2) is the ratio of flexural stress to the strain in flexural deformation.It is a measure of the stiffness during the initial part of the bending process. Flexural modulus is the ratio of stress to corresponding strain within the elastic limit.

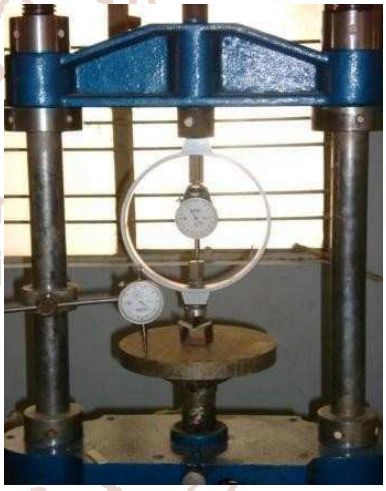

Fig.2. Flexural Test Setup

Fig.3. Universal Testing Machine Zwick/Roell Z010

C. Tensile test, tensile strength and tensile modulus

Tensile Strength: It is the maximum stress (N/m^2) that the material can withstand before failure. It is also called as ultimate tensile strength.

Tensile Modulus: It is also known as Young's modulus (N/m^2) or modulus of elasticity and is a measure of the stiffness of the material. It is given by the ratio of the uniaxial stress to the uniaxial strain.

Tensile test will be conducted on an electronic tensile testing machine Zwick/Roell Z010-10KN-UTM at a cross head speed of 3 mm/min and gauge length of 50 mm. Standard Type IV dumb bell shaped specimens are used as per ASTM D-638-03. The values of tensile strength and tensile modulus are obtained from the load-deflection values by taking the maximum load resisted by the specimen up to the point of fracture and the corresponding strain. Loading of the specimen on the Tensile Testing Machine and the grips used for holding the specimen are shown in Figs. 3.

D. Impact strength

It is the ability of the material to withstand shock loading. It is measured by the work done (kJ/m^2) in fracturing the material under shock loading. Izod impact test is used in the present case. Notched impact performance of the composite is evaluated as per ASTM D-256-05 using Pendulum impact tester, model IT 30 Izod impact supplied by PSI Sales Pvt. Ltd., New Delhi, shown in Fig. 4. The size of the samples is 12 mm X 60mm. The test specimen is supported by a vertical cantilever beam and the specimen was broken by the swing of the pendulum. The energy absorbed in doing so is measured as difference between the height of drop before rupture and the height of rise after rupture of the test specimen.

E. Barcol Hardness

Barcol hardness test characterizes the indentation hardness of the materials through the depth of penetration of an indenter, often used for composite materials. Standard test method ASTM D-2583-75 is used to find indentation hardness of the composite through Barcol impresser model
no. 934-1 shown in Fig. 5.

Fig.4. Pendulum Impact Tester

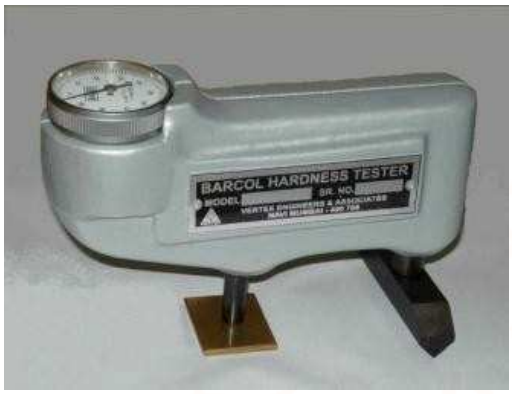

Fig.5. Barcol Hardness Tester

F. Scanning Electron Microscopy(SEM)

The morphology of fractured surfaces of the composites will be studied by Scanning Electron Microscopy (SEM) using EVOMA15 Smart SEM shown in Fig. 6. The fracture behavior of the material indicates the amount of energy absorbed before fracture, which is a measure of toughness of the material. The SEM reveals the nature of the bond between the fibers and the matrix.

Before performing SEM, The fractured specimens will be placed on a stub as shown in Fig. 7 coated with platinum and inserted into the scanning barrel. The inter-condition of the scanning barrel is vacuumed to prevent interference of scanning picture due to the presence of air. Magnification, focus, contrasts and brightness of the result is adjusted to produce the best micrographs.

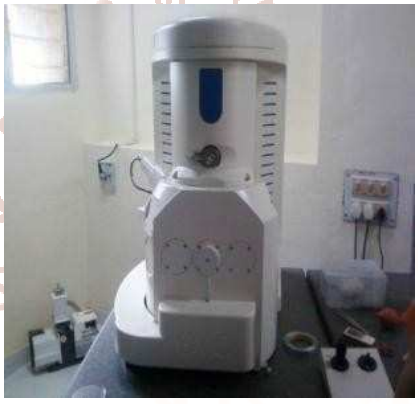

Fig.6. EVOMA15 Smart SEM

Fig.7. Stub of the EVOMA15 Smart SEM

G. Differential Scanning Calorimetry(DSC)

Glass transition temperature Tg of a non-crystalline material is the critical temperature at which the material changes its behavior from hard and brittle to rubbery state. This is less than the melting temperature (Tm). Differential Scanning Calorimetry is used for finding the Tg of the composites. DSC is performed with the help of Mettler using Star SW 8.1 analyzer to measure Tg. The temperature is programmed in the range of 25°-300°C with a heating rate of 10°C/min in nitrogen atmosphere with a flow rate of 30 ml/min. The Mettler DSC instrument and the silver pan used for conducting the test are shown in Figs. 9.

Figure 8 Mettler DSC Instrument

Fig.9. Thermo gravimetric Analyzer Perkin-Elmer

H. Thermo gravimetric Analysis (TGA)

Thermal degradation or weight loss due to heating is a measure of the thermal stability of the material under high temperature. Thermo gravimetric analysis (TGA) curves are used to determine the thermal degradation and thermal stability of the polymeric material. Thermal decomposition is observed as per ASTM E 1131 in terms of loss of global mass using TA Instrument TGAQ50 V20.10 Build 36 thermo gravimetric analyzer shown in Fig.9. The sample area is enclosed by a cylinder inside of the quartz tube. This energy-absorbing cylinder absorbs radiation from the lamps and heats the sample, pan, and thermocouple. Temperature is measured and controlled by a thermocouple assembly under the sample pan.

I. Dynamic Mechanical Analysis(DMA)

This analysis gives the storage modulus, loss modulus and damping property of the materials. Material‘s response for stress, temperature and frequency is determined through this test. Dynamic Mechanical Analysis, otherwise known as DMA, is a technique where a small deformation is applied to a sample in a cyclic manner. This allows the materials response to stress, temperature, frequency and other values to be studied. The term is also used to refer to the analyzer that performs the test. DMA is also called DMTA for Dynamic Mechanical Thermal Analysis. Specimens for dynamic mechanical analysis are prepared as per ASTMD 4065.

The Pyris Diamond DMA equipment by Perkin-Elmer Instruments is shown in Fig. 10. DMA yields information about the mechanical properties of a specimen placed under minor sinusoidal oscillating force and temperature.

Fig.10.. Pyris Diamond DMA

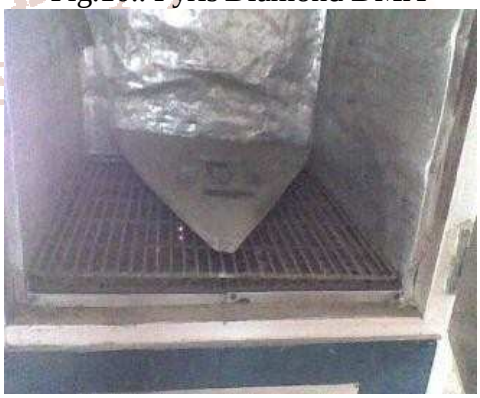

Fig.11. Hot air oven

J. Dielectric Strength

It is a measure of the electrical resistance of the material given by the maximum voltage that an insulating material can withstand before it

decomposes or becomes a conducting material by losing its insulation property. The test is conducted as per ASTM D149: Standard Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies. Voltage at a commercial power frequency 60 hertz is applied to a test specimen. The voltage is increased from zero, until the dielectric failure of the test specimen occurs. Hipotronics 715-1-A AC Dielectric Test set up will be used.

K. Water Absorption

Moisture absorption, soluble matter lost and long term immersion properties are evaluated. Water absorption tests are conducted on rectangular specimens of 76.2 x 25.4 mm^2 size shown in Fig. 18 as per ASTM D-570-98. This test method covers the determination of the relative rate of absorption of water by fiber when immersed. The moisture content of a fiber is very intimately related to such properties as electrical insulation resistance, dielectric losses, mechanical strength, appearance, and dimensions.

Short Time Immersion: The samples are conditioned by heating in an oven at 50±3 ^{o}C for 24 hours and then cooling to room temperature. The weights of the samples will be taken by Shimadzu Electronic Balance (AY 220) that has a readability of 0.001g. All the samples are immersed in double distilled water for 24 hrs at room temperature. Reconditioning is done by keeping them once again in the oven (Fig.11) for 24 hrs at 50±3 ^{o}C. Percentage increase in weight of the specimen during immersion is obtained by the ratio of increase in average weight of the conditioned specimen after immersion in water for 24 hrs and the average weight of reconditioned specimen is calculated nearest to 0.01%. The amount of soluble matter lost is given by the decrease in weight of the specimen after reconditioning. The percentage of water absorbed is the sum of the % increase in weight and the soluble matter lost.

Long Term Immersion: The total water absorbed by the conditioned specimens upon immersion for about 1200. The average % increase in weight of the specimen is calculated. The percentage increase in thickness or length or width swelling is obtained by the ratio of the increase in the respective dimension and the initial dimension.

CONCLUSION

The proposed research work on development of Century nano cellulose polyester and PVA composites can be of great value as new structural materials for automobiles, electronic devices, and packaging materials. The biodegradable PVA composites will find applications as biocompatible materials. Development of composites from these fibers will contribute for the development of Green Technology. Extraction of nano cellulose fibers from the plant fibers can grow as a cottage industry.

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