International Journal of Engineering & Technology, 7 (3.29) (2018) 604-606

International Journal of Engineering & Technology

Website: www.sciencepubco.com/index.php/IJET

Research paper

Synthesis and characterization of cellulose Nano fibers in enhancing the tensile stress properties of paper composites.

Sushmitha. D [1] *, Srinath. S [2]

[1] *Ph.D. Scholar, NIT Warangal, Telangana, India*
[2] *Associate professor, NIT Warangal, Telangana, India*
**Corresponding author E-mail: sushmitha.devadasu@gmail.com*

Abstract

In this contemporary work, synthesis of Nano cellulose was performed using microwave irradiation and Sonochemical approach. Experiments were conducted to know the synergetic effect on the synthesis of cellulose nanoparticles using a microwave at 700 watts for 1 minute and followed by Sonication having a power of 500W with probes size of 13mm. The Sawdust was first undergone pretreatment steps, then Consecutive treatments of extracted cellulose with combined equipment finally, gives Nano cellulose of particle size 68nm after 2 hours of sonication with 3% HCl. The tensile stress of the paper composites was increased exhibiting excellent mechanical properties when [5] wt% of nano-cellulose is added to the pulp for paper composites.

Keywords: *Ellulose Nano Fibers; PSD; FTIR; Paper Composites; Tensile Stress; Tensile Strain.*

1. Introduction

Nanotechnology has in numerous applications, in enhancing the thermal and mechanical stability of a material. Especially polymers have lot more interesting applications when it is in nano size. Particularly in synthesizing cellulose which is obtained from natural resources, when it is present in nanoscale can act as an insulator, thermosetting polymer, thermoplastic polymers, super capacitor Polyaniline (PANI) based electrode materials as energy storage, and even in sensor applications, and lot more applications.
Cellulose nanofibers can be obtained from, Acid Hydrolysis, Enzymatic Hydrolysis, Oxidation, Electrospinning method, Steam Explosion, High-Pressure Homogenization [5]. Synthesis of cellulose nanofibers should be considered in a cost-effective way to, with a faster rate of preparation.
The following Literature review explains that Several experiments were done for synthesizing nanofibers from different sustainable, renewable and naturally occurring raw materials.
Cao X et al, separated Cellulose from jute fibers, by treating with TEMPO (0.02 g)/NaBr (0.20 g) / NaClO (18 g), by adjusting the ph with sodium hydroxide, and the reaction was stopped by adding ethanol. Cellulose nanowhiskers of size 3-10 nm, with a crystallinity index of 69.7% using an ultrasonicator of power 1000 watt, and 20-25 kHz frequency for 30 minutes [1].
Camarero Espinosa et al, isolated thermally stabled nano-cellulose of size 5-50 nm, were obtained using filter paper with 10.7 M phosphoric acid. The mixture was heated 50-100°C, in an oil bath, and sonicated with 50−60 Hz frequency, and 200 W, power [2].
Anuj Kumar et al synthesized Cellulose nanocrystals by acid hydrolysis of bagasse with low-intensity ultrasound. Finally, average cellulose nanoparticle size of 250-480 nm, with nanotube of length 60 nm and 20 nm diameter, were obtained. They also reported cellulose nano fibers with a higher crystallinity index of 72.5 % [3].
Xie J et al, processed 2-5 nm Cellulose nano fibers by treating with Sulfuric acid (1.75% of solvent weight) using microwave of 1200 W maximum microwave power, and 100 bar maximum operating pressure. The residues from the microwave liquefaction process were further chemically purified. Using acidified NaClO2 solution (0.1% w/v) at 75°C for 1 h. Followed by treatment with (0.4%) NaoH at 75 °C for 30 min. then sonicated with a power of 700 watts and frequency of 25 kHz for 30 minutes [4]
Cellulose Papermaking is one of the most interesting and economical ways to produce a sustainable product from renewable resources. Wood is one of the common raw materials among agricultural wastes that are used from the past years to produce paper. The steps involved in the preparation of paper are 1) Isolation of cellulose fibers from wastes. 2) Addition of chemicals to smooth the fibers. 3) Beating the obtain fiber to get the pulp. 3) Separation and washing the pulp, using fourdrinier machine. 4) The pulp is spread into molds/vats and pressed to make wet sheets. 5) finally drying.
Application of cellulose Nanofibers in Paper composites has many interests in the present years.
Mechanical (grinding and homogenization) of cellulose produces cellulose nanoparticles, incorporation of 4 wt% in papermaking improves, tensile index by 21% [6]. Hassan et al. synthesizes CNF by Enzymatic and TEMPO-oxidization by using unbeaten bleached hard wood and softwood, by mixing 20 wt % of synthesized CNF, increases tensile index to 62% [7].
Kose et al. prepares CNF by mechanical means, and addition of 10 wt % CNF, without Softening pulp, increase its tensile index to 40%.[8].The tensile strength of the paper can be increased by increasing the weight percentage of nano-cellulose in papermaking composites. So in this work an attempt was made to synthesis CNF by synergic process and experiments were done on addition

of cellulose nano fiber in fabrication of paper to check its tensile properties.

In this paper, we concentrated on preparing nano-cellulose with microwave and high-intensity ultrasound, and its characterizations. To find the tensile strength properties of paper when cellulose nano fiber were added to the pulp.

2. Materials and methodology

2.1. Materials and methods

Teak Saw dust obtained from a local industrial mill located in Warangal, India. Toluene, ethanol Sodium hydroxide, Potassium hydroxide (himedia chemicals) and hydrogen peroxide (Fisher scientific chemicals), distilled water (deionized water), Ragas scientific microwave system, were used for the experiment. Ultrasound of Vibra-Cell Processors VCX-750.

2.2. Experimental procedure

All experiments are done with Teak sawdust (Tectona grandis). Water, toluene, and ethanol extractives were removed from sawdust using soxhlet extraction. Firstly water extractives were removes for 100 ̊C for 4 hours of extraction followed by toluene: ethanol extraction in 1:4 ratio for 4 hours. Delignification of biomass (Sawdust) was first done with 4wt% sodium hydroxide (NaOH) at 75^{o}C for 1hour and then bleached with 5wt% hydrogen peroxide (H_2O_2) at 30^{o}C for 1h. The dignified sawdust was filtered and washed with distilled water. Leaching of hemicellulose was done with 6 wt% Potassium hydroxide KOH at 80C for 2 hours. Finally, isolated cellulose contents were dried and analyzed for its cellulose content.

2.3. Cellulose nano-fibers preparation

Isolated cellulose is further treated with [3] wt% hydrochloric acid (HCl) using water to extracted cellulose ratio as 1:10. The solution was first treated with microwave (700 watts for 1 minute) and followed by Sonication with a power of 500 Watts, operating conditions of 30% amplitude, with a pulse of 30 seconds on, and 30 seconds off, for 45 minutes.

2.4. Pulp preparation

The isolated cellulose was saturated with water for about 1 hour and made to a homogeneous solution, by a homogenizer to obtain pulp. Further pulp was washed with distilled water. Obtained nano-cellulose of 5 wt % was added and then placed evenly on a rectangular mould (15 x 2.5 cm^2), extra water was drained by pressing the pulp with the help of 3 kg iron rod having even surfaces for 5 hours. The pressed fiber sheets were then cured for 24 hours.

3. Characterizations methods

3.1. Fourier transform infrared spectroscopy (FTIR)

Spectrum 100 Optica FT-IR Spectrophotometer of Perkin Elmer, (NITW, Telangana, India) is used for FTIR analysis of treated biomass. The pretreated and dried sawdust was mixed with spectroscopic grade KBr powder in 1:3 ratio and pelletized like thin 1mm circular disk. This pellet was kept in a pellet slot for FTIR analysis under mid-range from 400 to 4000 cm-1.

3.2. Particle size distribution (PSD)

Particle size analysis was performed using particle size analyzer Malvern. The nano-cellulose power was dispersed in a distilled water solution of a concentration of 0.1g/ml, sonicated for proper mixing using bath sonicator for 15 minutes. A small amount of liquid was transferred to the Disposable sizing cuvette and placed in the allotted slot to get the particle size.

3.3. X-Ray diffraction (XRD)

XRD [X-Ray Diffraction], X-Ray Diffraction (X-RD) patterns were collected from a powder X-ray diffractometer (PANalytical X1pert powder Model) at operating conditions of 45 mV and 30 mA, for a 2theta range of 5-30 degrees at 0.016 degrees step intervals, with step input of 34.925 sec.

3.4. Tensile strength

Tensile strength was analysed with the universal testing machine, particularly bench top tensile testing machine with a capacity of 30 KN. Tensile properties of paper and paperboard (using constant rate of elongation apparatus) according to Tappi method 494 om-01.

4. Results and discussions

4.1. Fourier transform infrared spectroscopy (FTIR)

Fig1 gives FTIR graphs for sawdust, pure cellulose, and CNF. The CNF peaks show a similar trend with pure cellulose, and the existence of 1737 cm-1and 1255 cm-1peaks for hemicelluloses are due to the C=O stretching vibration of the carbonyl and acetyl groups in the xylan component of hemicellulose are present in the sawdust. The removal of lignin content was observed in CNF.Thepeakat898 indicates C–H vibration corresponding to the cellulose, and nano-cellulose. CNF spectra show a peak at 1616 cm-1 is due to the presence of carboxylic groups in the primary alcoholic groups may be due to the treatment of sawdust with hydrogen peroxide [6].The peak observed at 1642 cm-1 in the sawdust, cellulose, and CNC spectra, associated with the O–H bending vibration of absorbed water, and peak at 1510 cm-1 corresponding to aromatic ring vibrations which signifies the presence of lignin in the sawdust spectrum, these peaks were not found in the cellulose, CNF spectra [5].

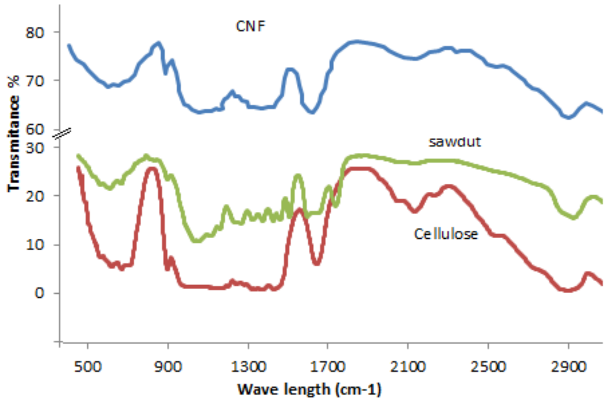

Fig. 1: FTIR Graphs Representing Structural Characterization of Pure Cellulose, Sawdust, CNF.

4.2. Particle size distribution (PSD)

From particle size analysis, cellulose nanoparticle size was obtained as 68 nm. The stability test was done without disturbing the solution for 24 hours, and there was no separation or sedimentation of the particles in the solution.

4.3. X-ray diffraction (XRD)

From XRD graphs it is clearly shown that the area under the graph for sawdust and CNF between the amorphous region and crystallinity region is greater for CNF as compared to the sawdust. That could be a reason for an increase in crystallinity for CNF as compared to the sawdust, is due to the removal of amorphous content during pretreatment step. Crystallinity index can also be calculated by the below-mentioned formula (1), and is defined as the percentage of crystalline material in the biomass, and was calculated with,

$$CrI\ (\%) = (I002 - Iam) * 100 \quad (1)$$

I_{002}

Where I_{002} is the maximum intensity of 002 peaks at 2θ = 23 degrees and I_{am} is the intensity at 2θ = 19 degrees.

The crystallinity index for sawdust was 49 %, and CNF was found to be 78%.

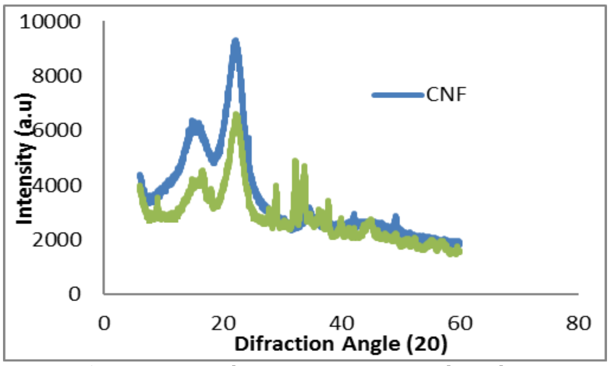

Fig. 2: XRD Graphs Representing CNF and Sawdust.

4.4. Tensile strength

Tensile Strength of paper was increased on addition of CNF content in papermaking composites. Figure 3 shows the percentage of strain developed in the fabricated paper (test specimen) to that of applied tensile stress. The paper was cut into (180x2.5 cm^2) for testing, grammage the specimen is calculated. The test specimen is clamped to the two ends with the aid of jaws, then the stress was applied in a vertical direction, and the corresponding strain is recorded. For zero weight percent loading of CNF in pulp, the tensile strain % obtained was 2.78% for tensile stress of 96 MPa. However, by increasing the CNF loading weight percent in pulp, the tensile stress was increased to 140 MPa with a strain % of 3.80.

The tensile index is known as the tensile strength in Newton per meter divided by grammage. There was an increase in the tensile index for CNF loading of 5 wt% as compared to a fabricated paper without CNF. Table 1 explains about tensile stress, tensile strain percentage, grammage and tensile index of fabricated paper with and without cellulose nano fiber loading.

Table 1: Tensile Stress, and Tensile Strain percentage, Grammage, and Tensile Index of Fabricated Paper (with and without CNF loading)

CNF Wt % in Pulp	Tensile stress(MPa)	Strain %	Grammage (g/m^2)	Tensile index (Nm/g)
0	96	2.78	17.333	$5.5385x10^6$
5	140	3.80	17.377	$8.0566x10^6$

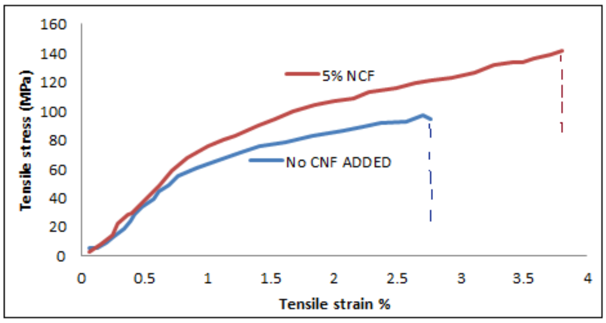

Fig. 3: Tensile Stress Verses Tensile Strain percentage Curves for Fabricated Paper (With and without CNF).

5. Conclusion

Nano Cellulose obtained from successive treatment with microwave irradiation, and high power ultrasonicator gives a particle size of 68 nm, with a crystallinity index of 78 %. Incorporation of cellulose nano fibers in the paper composites enhances the tensile stress from 96 to 140 MPa for CNF loading percentage of zero, to 5 wt%.

References

[1] Cao X, Ding B, Yu J, Al-Deyab SS "Cellulose Nano-whiskers extracted from TEMPO-oxidized jute fibers," Carbohydrate Polym 90:1075–1080, 2012.

[2] Camarero Espinosa S, Kuhnt T, Foster EJ, weder C "Isolation of thermally stable cellulose Nano-crystals by phosphoric acid hydrolysis," Bio macromolecules 14:1223–1230, 2013.

[3] Anuj Kumar, Yuvraj Singh Negi, Veena Choudhary, "Characterization of Cellulose Nanocrystals Produced by Acid-Hydrolysis from Sugarcane Bagasse as Agro-Waste," Journal of Materials Physics and Chemistry, 2 (1), pp 1-8, 2014.

[4] Xie J, Hse CY, De Hoop CF, Hu T, Qi J, "Isolation and characterization of cellulose nanofibers from bamboo using microwave liquefaction combined with chemical treatment and ultrasonication." Carbohydrates Polymer. Oct 20 151: 725-734, Epub 2016 Jun 32016.

[5] Usmani, Amil Khan, Imran Nayeem, Mondal, Dibyendu Bhat, "Biomass nano-fibrillar Cellulose-Reinforced Nanofibre Composites Production, Properties, and Applications". 305-326, 2017.

[6] Eriksen, Syverud, K., & Gregersen, "The use of micro-fibrillated cellulose produced from kraft pulp as strength enhancer in TMP paper," NordicPulp & Paper Research Journal, 23(3), 299–304, 2008.

[7] Hassan, M. L., Bras, J., Mauret, E., Fadel, S. M., Hassan, E. A., "Palm rachis micro-fibrillated cellulose and oxidized-micro fibrillated cellulose for improving paper sheets properties of unrefined softwood and bagasse pulps," Industrial Crops and Products, 64, 9–15, 2015.

[8] Kose, R., Yamaguchi, K., Okayama, T. "Influence of addition of fine cellulose fibers on physical properties and structure of the paper," Sen'I Gakkaishi, 17(2), 85–90, 2015.

[9] Tensile properties of paper and paperboard (using constant rate of elongation apparatus) (Revision of T 494 om-01).