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Synthesis and Characterization of Cellulose Nanocrystals-Graft-N-Butyl Acrylate / Polyethylene Glycol Blend

K. Rani[1] and P. N. Sudha[1]*
[1, 1*] **Biomaterials research lab, Department of Chemistry, D. K. M. College for Women (Autonomous), Vellore, India.**

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Corresponding Author Email: drparsu8@gmail.com

Abstract
In this paper, Banana fibers were converted to Cellulose Nano Crystals (CNCs) using steam explosion method. The CNCs grafted with n-butyl acrylate monomer and ceric ammonium nitrate as an initiator. The synthesized graft copolymer was blended with poly ethylene glycol (PEG) in (1:1) ratio with gluteraldehyde as a crosslinking agent. The prepared blend was characterized using various analytical techniques such as FTIR, XRD, thermal studies (TGA and DSC) and SEM. The FTIR study clearly indicates that the formation of blend and by XRD, were used to find crystalline and amorphous areas of CNCs and binary blend. The thermal stability and miscibility of the prepared blend were evaluated by TGA and DSC. The surface morphology of the prepared blend was characterized by SEM images. The results are discussed.

Keywords
Cellulose Nano Crystals (CNCs), Polyethylene Glycol (PEG), N-Butyl Acrylate, and Characterization.

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1. INTRODUCTION
Cellulose is the most ancient and important natural polymer on earth which has revived and attracted more attention in the new form of “Nano cellulose” which is used as novel and advanced material. The growth and development of Nano technology, cellulose play a large role in nanotechnology field. Nano cellulose is extracted from native cellulose (found in plants, animals, and bacteria) collected of the Nano scaled structure material. Nano cellulose is divided in three types, (1) cellulose Nano crystals (CNC) with other denominations such as Nano crystalline cellulose. (2) Cellulose Nano fibrils (CNF) with the meaning of Nano-fibrillated cellulose (NFC), micro fibrillated cellulose (MFC), cellulose Nano fibers; and (3) bacterial cellulose (BC), also mentioned to as microbial cellulose. [1, 2] Nano cellulose shows the properties such as high tensile strength, ease of functionalization and biological properties are renewability, biocompatibility, biodegradability and low toxicity. [3, 4, 5] CNCs have possible for several applications in various industrial sectors, such as medicine, electronics, biomaterials and energy production. [6, 7] Cellulose Nano crystals are generally

derived from the sources are wood, cotton, hemp, flax, wheat straw, sugar beet, potato tuber, mulberry bark, ramie, algae, and tunicin. CNC with rod-like morphology with length ranging from 100nm to 700nm and diameter about 5nm to 30nm [from plant cellulose]; animal tunicate length ratio close to 100nm, bacterial cellulose with length ranging from 100nm to several micrometre and diameter about 5nm to 50nm. [8, 9] Cellulose Nano crystals (CNCs) are rigid, rod-like particles with a width of several nanometers and lengths of up to hundreds of nanometers. [10, 11]

The graft copolymerisation is one of the effective methods which was used to improve the intrinsic properties of natural fibers. The graft copolymerization processes the branched copolymer was formed by covalently attaching the side chain grafts to the main chain of the polymer backbone. [12] Now a day's polymer blending has received more attention of researchers. The polymer blending, which results in the preparation of new materials can be observed with better physicochemical and mechanical properties. [13] Polyethylene glycol (PEG) was selected because it is hydrophilic and low toxicity, which blended onto CNCs grafted copolymer, is expected to increase in flexibility of the macromolecule. T. jayaramudu et al [14] reported poly (ethylene oxide)-lignin (PEO-L) blends with CNF through single lap joint tests for CNF films. The PEO-L blends were prepared by blending them with 60% methanol. The prepared blend was characterized by FTIR, DSC and TGA. The blend formation confirmed by hydrogen bond formation between lignin/PEO and CNFs. N.Y. Abou-Zeid and his coworkers [15] reported Cyanoethylchitosan/Cellulose acetate blended flim were prepared successfully by mixing CECS and CA in DMF as co-solvent and impregnated in water. The prepared blended films were good miscibility and good antibacterial activity. S. Rudhziah et al [16] reported on new biopolymer blend based on kappa-carrageenan and cellulose derivatives were prepared using solution casting technique. The cellulose derivative, carboxymethyl cellulose was produced from cellulose extracted from kenaf fibres. The polymer blend was prepared at various ratios, blend system containing 40 wt% of CMCE was found to be the highest conducting amongst all the polymer blend films.

Based on the literature review, the aim of the present study banana fibers converted to Cellulose Nano Crystals grafted with n-butyl acrylate monomer and ceric ammonium nitrate as an initiator. The grafted copolymer blended with Polyethylene glycol (PEG) in (1:1) ratio with gluteraldehyde as a crosslinking agent. The prepared blend was characterized using various analytical techniques such as FTIR, XRD, thermal studies (TGA and DSC) and SEM.

2.MATERIALS AND METHODS

2.1 MATERIALS

The materials used for the study includes banana fibers (collected from local farms), NaOH (commercial grade), acetic acid (commercial grade), sodium hypochlorite (commercial grade), oxalic acid (commercial grade). Analytical grade butyl acrylate, ceric ammonium nitrate, Polyethylene glycol (PEG) were used.

2.2 Extraction of Cellulose Nano Crystals (CNCs) from Banana fiber by using Steam Explosion Method

30 gms of Banana fibers were chopped into uniform size of approximately 10 cm. The fibers were treated with 2% NaOH (fiber to liquor ratio 1:10) in an autoclave and kept under 20 lb. pressure for a period of 1 h. Pressure was released immediately. The fibers were removed from the autoclave, and the fibers were washed in water till they were rid of alkali. The washed fibers were allowed to drain off free flowing water (Stage – 1). The steam exploded fibers were bleached using a mixture of NaOH and acetic acid (27 and 78.8 g, respectively) and a mixture of 1:3 sodium hypochlorite solution. The bleaching was repeated six times. After bleaching, the fibers were thoroughly washed in distilled water and dried (Stage –2). The steam exploded bleached fibers were treated with oxalic acid (9%) in an autoclave till it attained a pressure of 20 lb. The pressure was released immediately. The autoclave was again set to reach a pressure of 20 lb., and the fibers were kept under that pressure for 15 min. The pressure was released, and the process repeated 8 times. The fibers were taken out, washed till the washings no longer decolorized $KMnO_4$ solution to make sure that the washings were free from acid (Stage – 3). The fibers were suspended in water and under continuous stirring with a mechanical stirrer of type RQ – 1.27 A and 8000 R.P.M. for 4 h. The suspension was kept in an oven at 90 °C till it was dry (Stage – 4).

Stage-1 **Stage-2** **Stage-3** **Stage-4**

Figure-1: Photograph of steam exploded banana fibers at various stages

2.3 Preparation of grafted co-polymer

A required amount of cellulose (0.5 g) was dispersed in water (100 mL) with constant stirring forming a homogeneous solution. 1 mL of butyl acrylate monomer dissolved in 10 mL of ethanol was then added to that homogenous solution. To initiate the polymerization process, the initiator ceric ammonium nitrate (10 mL) was added.

After all the addition was over the above mixture was heated to 70 °C simultaneously the stirring of that mixture was performed using a magnetic stirrer. This solution was then poured into excess 2N sodium hydroxide solution to precipitate the graft copolymer. It was then filtered, dried and weighed.

2.4 Preparation of binary blend

The polyethylene glycol (PEG) weighed in (1g) dissolved in 10ml of water with constant stirring forming a homogeneous solution added in 1g of grafted copolymer (1:1) (CNCs + Butyl acrylate monomer + CAN Initiator) and added a cross linker gluteraldehyde 15ml was added. After all the addition was over the above mixture was stirred well by using a magnetic stirrer. This solution was then poured into Petri dish. It was then dried and weighed.

3. CHARACTERIZATION

3.1 FTIR Spectroscopy

Measurements were performed with Thermo Nicolet AVATAR 330 Spectrometer in 4000 – 400 cm^{-1} ranging using KBr pellet at DKM College for women, Vellore.

3.2 X – ray powder Diffractometer (XRD)

In order to observe the molecular packing of the processed Banana fibers (4 stages) and grafted copolymers X – ray powder diffractometer (XRD – SHIMADZU XD –D1) using a Ni – filtered Cu Kα X – ray radiation source.

3.3 Thermal Analysis (TGA and DSC)

Thermal behaviours of the prepared blend were studied by using a TGA instrument (SDT Q600, TA Instruments USA), at a heating range of 5 to 20 °C/minutes in the temperature range upto 1200^0 C at VIT, Vellore. Glass transition temperature of the specimens were investigated by using a DSC instrument (micro DSC7 evo) at a heating range of 30 to 400^0 C.

3.4 SEM Analysis

Scanning Electron Microscopy (SEM) is a method for high resolution surface imaging. The surface morphology was studied using JEOL Model JSM – 6390LV, with magnification X250. Scanning electron micrograph was taken at voltage of 20 kV.

4. RESULTS AND DISSCUSSION

4.1 FOURIER TRANSFORM INFRARED SPECTROSCOPY

FTIR is mainly used to determine of functional groups in frequency range from 400 cm^{-1} to 4000 cm^{-1}. The FTIR Spectral details of Cellulose Nano Crystals (CNCs) and binary blend were represented in **Fig. 2 and Fig. 3.**

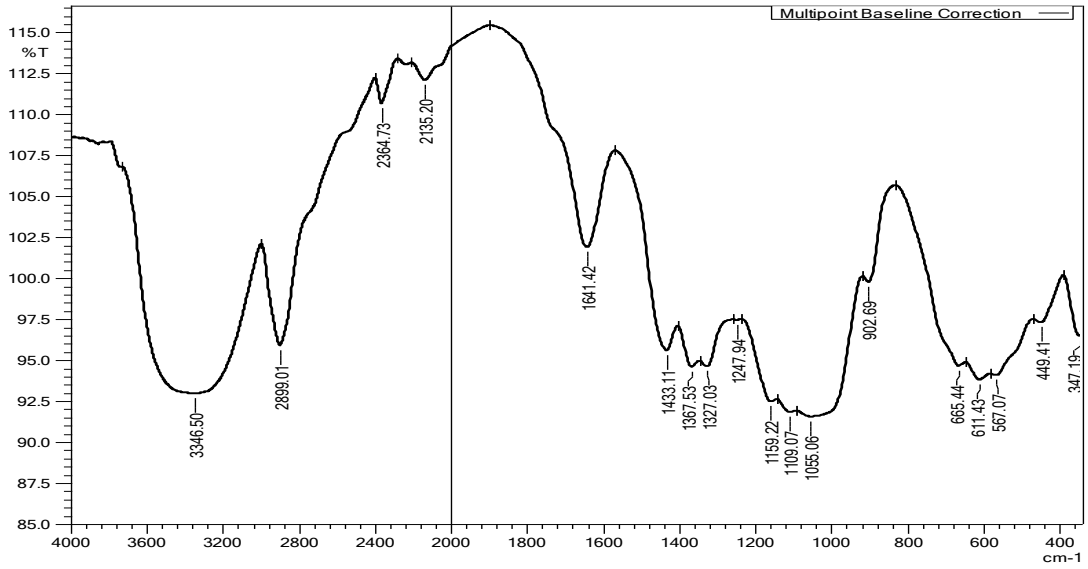

Figure-2: FTIR Spectra of Cellulose Nano Crystals (CNCs)

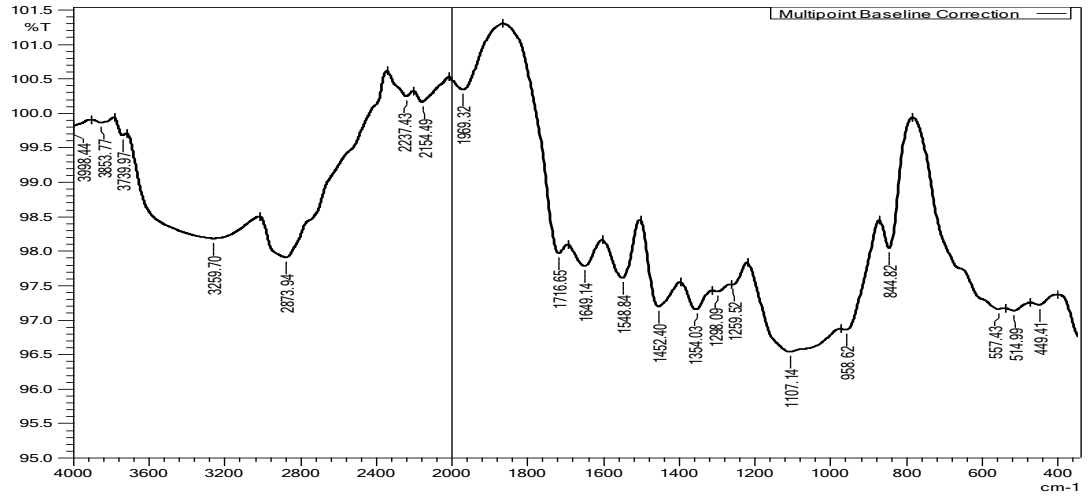

Figure-3: FTIR Spectra of CNCs-g-n-BA/PEG 1:1 Binary Blend

Fig. 2 represents Infrared spectra of Cellulose Nano Crystals and **Fig. 3** shows CNCs-g-n-butyl acrylate/PEG blend. The characteristic peaks at 3346 cm^{-1} in CNCs and at 3259 cm^{-1} in CNCs-g-n-BA/PEG binary blend due to the OH stretching. [17] The peaks at 2899 cm^{-1} and 2873 cm^{-1} for both spectrum corresponds to CH stretching. [18] CNCs sharpening of the peak area 2899 cm^{-1} take place. This is due to the increase in the crystallinity in the fibers, showing the increase in the percentage of cellulose components. Moreover, the absorption bands at CNCs 1641 cm^{-1} the presence of lignin and attributed to the C=C vibration. The peaks in the region 1430 cm^{-1} are due to C-O stretching. The broad peak at 1050 cm^{-1} are assigned to ether linkage (C-O-C) from lignin or hemicellulose. [19]

The CNCs-g-n-BA/PEG blend the new peaks observed at 1548 cm^{-1}, 1354 cm^{-1}, 844 cm^{-1} and 514 cm^{-1} due to (COO^{-}) asymmetrical carboxylate anion, OH bending, C-H out of plane bending and C-C bending. [20] It can be concluded that the comparison between the spectra of CNCs and CNCs-g-n-BA/PEG binary blend OH-stretching vibration band shifted to a lower wave number and broaden. These results indicate that strong inter molecular hydrogen bonding interaction takes place between CNCs and binary blend leading to a good miscibility of the blend.

4.2 X-RAY DIFFRACTION STUDIES

The X-ray diffraction was carried out to study the structure and crystalline nature of CNCs and binary blend. [21] The XRD patterns of CNCs and CNCs-g-n-

BA/PEG binary blend were represented in **Fig. 4** and **Fig.5.**

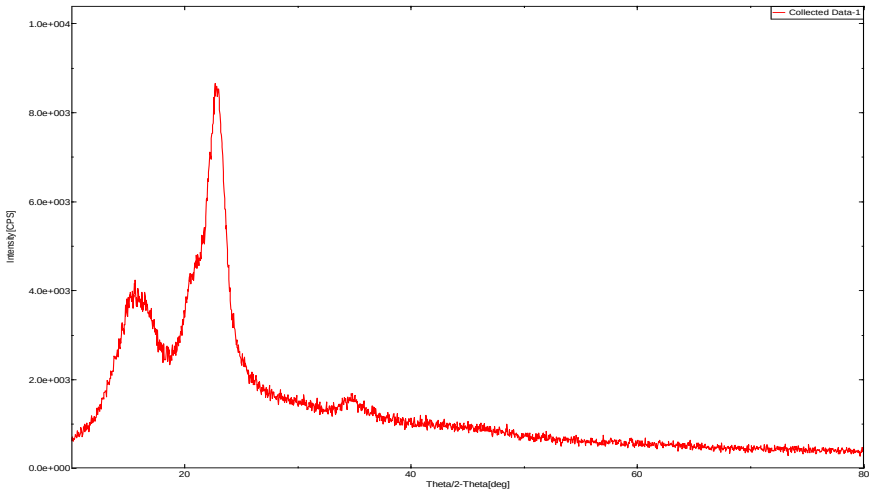

Figure-4: XRD Spectra of Cellulose Nano Crystals (CNCs)

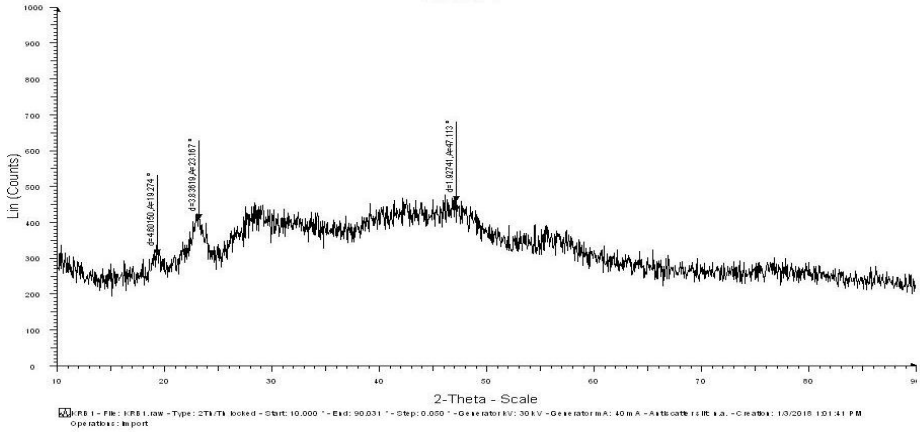

Figure-5: XRD Spectra of CNCs-g-n-BA/PEG 1:1 Binary Blend

The XRD spectra of CNCs in **Fig. 4** shows crystalline nature with characteristic peaks at 2θ =18^0 & 22^0. The CNCs-g-n-BA/PEG binary blend **Fig. 5** broad peak indicating highly amorphous in nature peaks at 2θ = 19^0, 23^0 ,47^0. The highest crystallinity of the CNCs was related to inter and extra molecular hydrogen bonds of cellulose. The CNCs first grafted with n-butyl acrylate the grafted copolymer when treated with NaoH due to breakage of hydrogen bonds in the crystalline part of cellulose chain and results to reduce the crystallinity. [22] In addition, the CNCs-g-n-BA/PEG binary blend, PEG is water soluble which deformation of the crystalline structure in the cellulose. [23] Therefore the binary blend was more amorphous in nature. The percentage degree of crystallinity can be calculated using this formula, Table-1 shows XRD spectral details of CNCs and CNCs-g-n-BA/PEG binary blend given in below,

$$Xc\ (\%) = \frac{Ac}{Ac+Aa} \times 100$$

Xc = % Degree of crystallinity

Ac = Crystalline area on the X-ray diffraction and

Aa = Amorphous area

Table-1 XRD Spectral details of CNCs and CNCs-g-n-BA/PEG (1:1) Binary Blend

Compounds	2θ	Degree of crystallinity (%)
Cellulose Nano Crystals	18^0 & 22^0	73.52
CNCs-g-n-BA/PEG Binary Blend	19^0, 23^0 ,47^0	4.17

4.3 THERMO GRAVIMETRIC ANALYSIS (TGA)

Thermo gravimetric analysis (TGA) measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. TGA is a useful technique to assess the thermal stability of polymer and its blends.

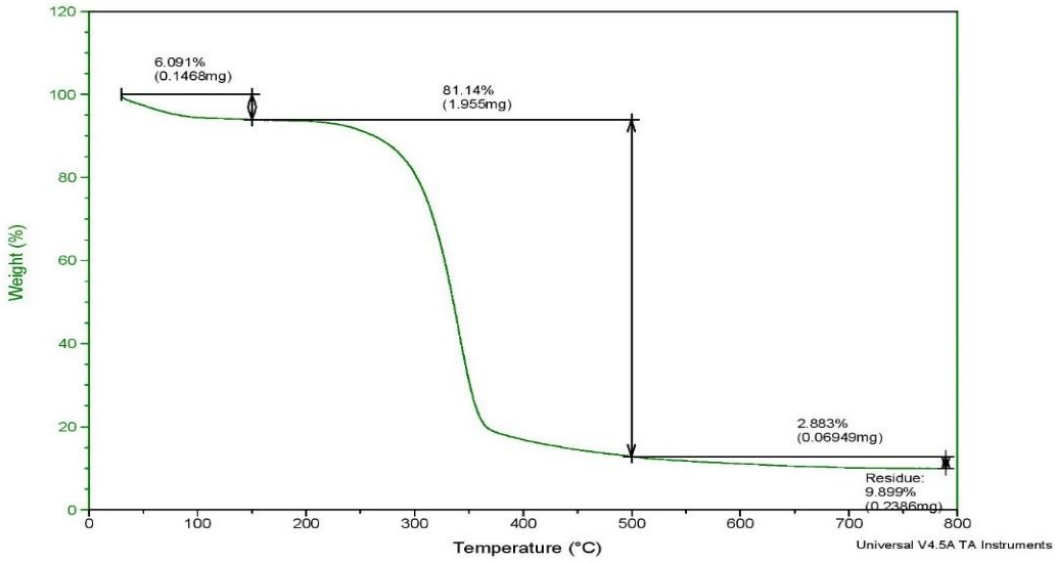

Figure-6: TGA thermogram of Cellulose Nano Crystals (CNCs)

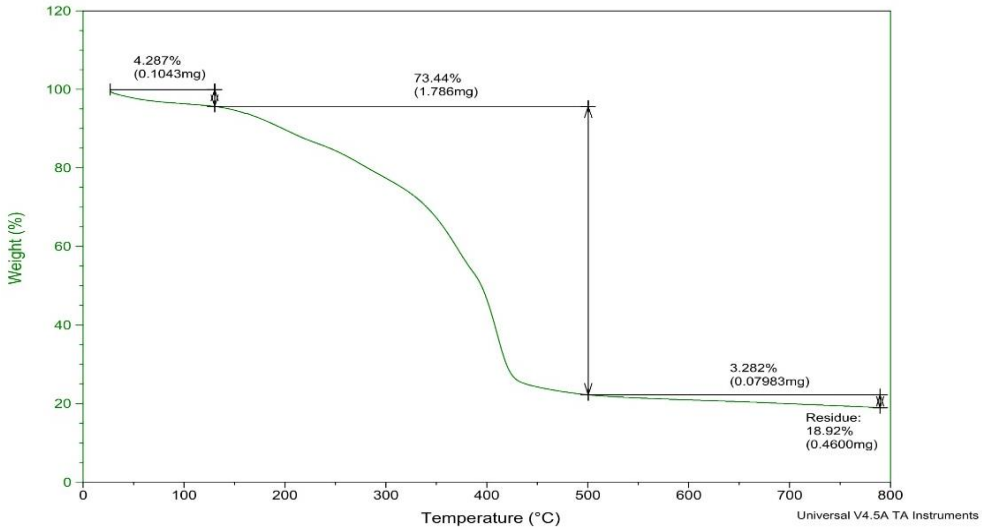

Figure-7:TGA thermogram of CNCs-g-n-BA/PEG (1:1) binary blend

Table-2: TGA thermogram Studies of CNCs and CNCs-g-n-BA/PEG (1:1) blend

Percentage Decomposition (%)	Decomposition Temperature (^{0} C)	
	Cellulose Nano Crystals	CNCs-g-n-BA/PEG (1:1) Binary Blend
10	280	220
20	310	300
30	330	350
40	340	390
50	350	410
60	370	430
70	390	450

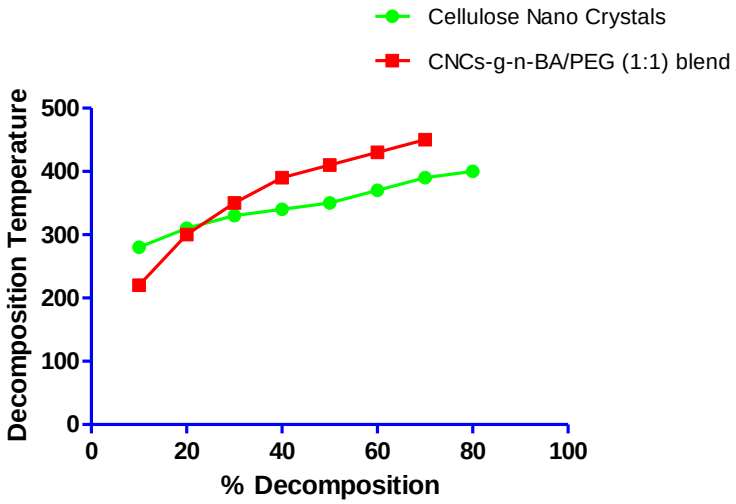

Figure-8: TGA thermogram details of CNCs and CNCs-g-n-BA/PEG (1:1) blend

Fig. 6 and **Fig. 7** shows TGA thermograms of CNCs and CNCs-g-n-BA/PEG binary blend. The CNCs thermal degradation consist of three stages. The first stage is weight loss up to 6.091% with a maximum temperature range at 150^0 C which is related to evaporation of water molecules. The second stage is weight loss of CNCs 81.14% with maximum temperature range at 450^0 C which is related to the cleavage of glycoside linkage in the cellulose backbone, this is due to maximum weight loss of the CNCs. The third stage starts at 450^0 C with maximum at 800^0 C there is only linear shallow decrease in weight with increse in temprature. [24] The CNCs-g-n-BA/PEG binary blend thermal degradation consist of three stages. The first stage is weight loss up to 4.29% with a maximum temperature range at 150^0 C which is related to evaparation of water molecules. The second stage is weight loss of binary blend 73.44% with maximum temperature range at 500^0 C which is related to depolymerization of CNCs-g-n-BA/PEG blend chains, this is due to maximum weight loss of the blend. The third stage starts at 500^0 C with maximum at 800^0 C there is only linear shallow decrease in weight with increse in temprature. [25] On comparing the TGA results of CNCs and CNCs-g-n-BA/PEG binary blend was found to be highly thermally stable and this was conformed from the higher amount of residue remained at the end of the experiment.

4.4 DIFFERENTIAL SCANNING CALORIMETRY (DSC)

DSC is an effective and well-established technique that can determine T_g (Glass transition temperature) of the blended composites. Glass transition temperature is an important physicochemical parameter that explains miscibility or immiscibility of the blended composites. [26]

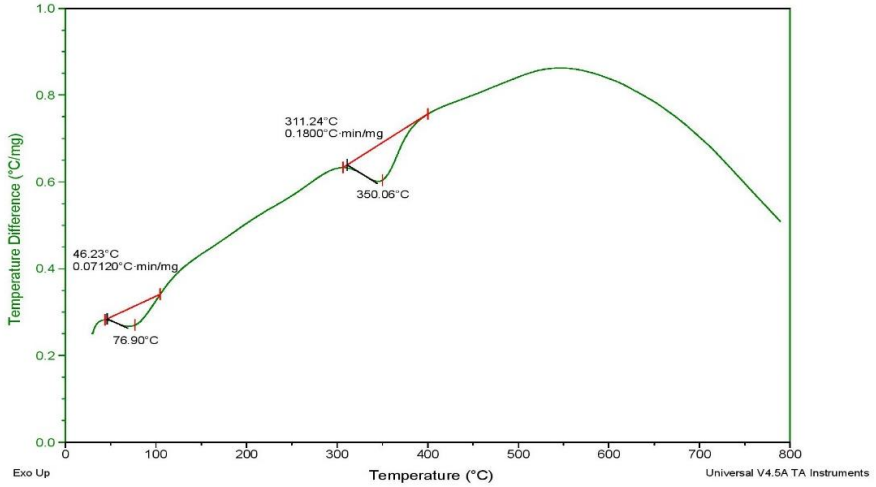

Figure-9: DSC Thermogram of Cellulose Nano Crystals (CNCs)

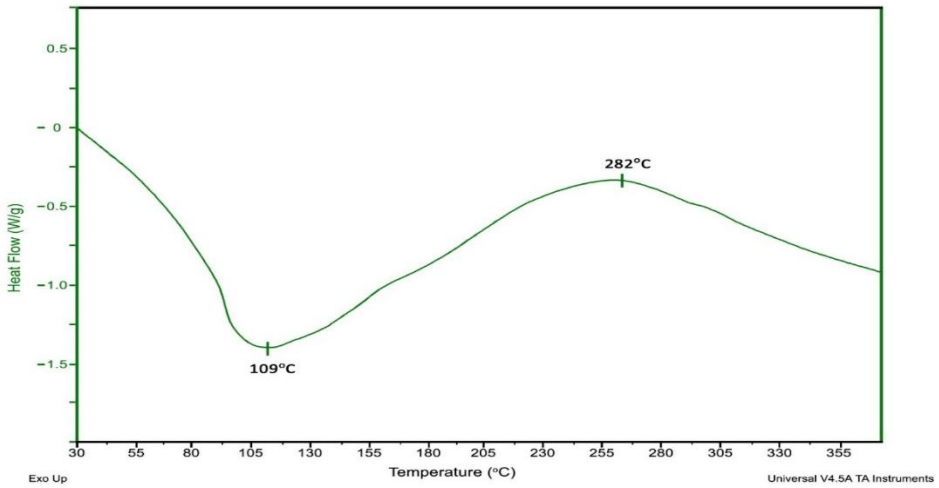

Figure-10: DSC Thermogram of CNC-g-n-BA/PEG (1:1) Binary Blend

Fig. 9 and **10** shows DSC thermogram of CNCs and CNCs-g-n-BA/PEG binary blend. **Fig. 9** shows two peaks (76.90^0 C and 350.06^0 C) which are associated with the glass transition and melting temperatures of CNCs. By blending with PEG **Fig. 10** shows two peaks 282^0 C one exothermic peak (Tm) showing the melting of the sample at this temperature and a one endothermic peak 109^0 C (Tc) shows the crystallization of the sample at a lower temperature. The DSC thermogram of binary blend shows single glass transition temperature (190^0 C) due to good miscibility of the blend. [27]

4.5 SCANNING ELECTRON MICROSCOPY (SEM)

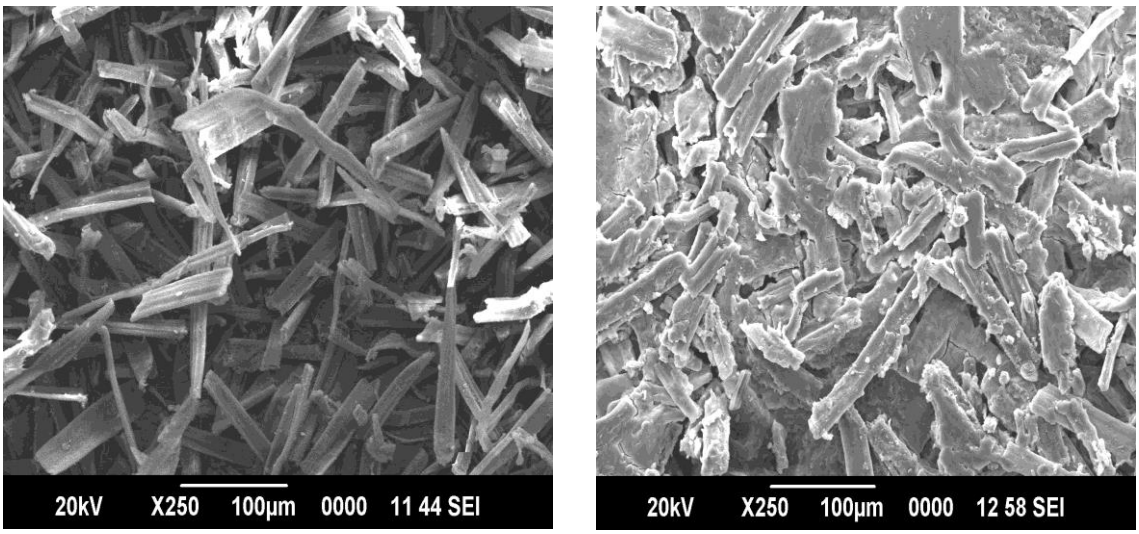

Figure-11
SEM images of CNCs and CNCs-g-n-BA/PEG binary blend

Scanning electron microscopy is an extremely useful method for visual confirmation of surface morphology and the physical state of the surface. **Fig. 11** shows Scanning Electron Microphotographs of Cellulose Nano Crystals and Cellulose Nano Crystals-g-n-butyl acrylate/Polyethylene glycol (1:1) binary blend respectively. On comparing Cellulose Nano Crystals (CNCs) surface was very smooth, ordered and homogeneous. [28] But, the Cellulose Nano Crystals-g-n-butyl acrylate/Polyethylene glycol blend shows uneven and rough surface. [29]

5. CONCLUSION

In this study, Cellulose Nano Crystals have been successfully isolated from banana fiber by steam explosion method. The CNCs grafted with butyl

acrylate monomer and ceric ammonium nitrate initiator. The grafted copolymer blended with polyethylene glycol (PEG) in (1:1) ratio with gluteraldehyde as a crosslinking agent. The FTIR results indicate that strong inter molecular hydrogen bonding interaction takes place between CNCs and binary blend. The XRD of CNCs shows more crystalline in nature. But the prepared blend was more amorphous in nature. TGA and DSC results of the prepared CNCs and CNCs-g-n-BA/PEG binary blend exhibited highly thermally stable and good miscibility of the blend. SEM micrographs of CNCs smooth, ordered and homogeneous. But the binary blend shows uneven and rough surface.

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