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Carbon Nanotube Reinforced Natural Fibers for Biodegradable Nanocomposites

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Abstract: Natural fiber-reinforced nanocomposites (NFRCs) are proved as the best alternative for synthetic composites in view of cost and environmental effects. NFRCs have been produced from agro-waste such as banana tree fiber (BFs), because BF are strong, light-weight, and have smaller elongation. To improve the quality of BF, multiwall carbon nanotubes (MWCNTs) are used as reinforcing filler. MWCNTs are functionalized by an ecofriendly radio frequency oxygen plasma processing method. Cellulose nano-crystals (CNC) are extracted from BFs by double hydrolysis process and a simple dip-drying technique has been used to produce NFRCs. Field emission scanning electron micrographs and transmission electron microscopy conform the well functionalization of MWCNTs and also ensure homogeneous incorporation in the BF matrix. The composites continue thermally stable corresponding to BFs. Mechanical strength of the NFRCs are improved owing to the incorporation of MWCNTs. Functional groups in the BFs, CNC and NFRCs are investigated by Fourier transform infrared spectroscopy. The current density of the sample is increased 1000 times than the raw fibers and conductivity increases up to 17 Sm^{-1}, which increases with temperature under the applied voltage 100 V and shows the linier characterization. Therefore, these light-weight biodegradable NFRCs encourage its ability as cost effective industrial conductive composite as usable in electronic devices.

Keywords: Natural Fiber-reinforced Nanocomposites, Banana Fiber, Multiwall Carbon Nanotubes, Cellulose Nano-crystals

1. Introductions

The use of new materials in modern science and lifestyles has increased significantly due to their scarcity, strength and durability [1]. Plastic materials are now used more than metals used in pubs. The structure of plastic has made it cost-effective and popular for machine components. There are many harmful effects behind the benefits of using plastic that cause extensive damage to the human body and the environment [2]. Plastic is responsible for disturbing the balance of the environment and long-term illness of animal [3]. Therefore, finding alternative to such substances and ensuring their use is the main topic of discussion for scientists now, and the solution may be natural fiber. Natural fiber are ecofriendly and easily perishable [4]. Products made with natural fiber contain maximum cellulose which helps in making biodegradable composite. Recently, cellulose produced from vegetable/plants fiber has been working as an alternative to make synthesis composite [5]. The use of natural fiber to make light and cost-effective composite is increasing worldwide. Furthermore natural fiber acts as a good nanocomposite. Natural fiber is extract from various plant parts like banana, jute, coir, sisal etc. At present natural fibers are used in domestic purpose for making ropes, mats, yarns, handbag, ornaments etc. [6]. But in addition to all these uses natural fiber such as banana expose, jute can be used for industrial purpose. NFRCs with a very thin and tensile strength are made with natural fiber which can be

used as a good electric conductor [7].

Natural fiber is an eco-friendly, biodegradable and recyclable biopolymer as an alternative to polluting the environment with coal, oil, gas and its wastes [8]. The remarkable properties of natural fiber and the nanocomposite prepared with the use of nanofillers could be the next global energy material [9]. Among the natural fibers, banana fiber is a very useful fiber for its remarkable chemical properties and mechanical structure. Banana fiber has innumerable thick walled cell tissues like acrylic natural gums. The main constituents of banana fiber are cellulose and up to 43.6% which form strongly nanocomposite with nanofillers. In addition to cellulose, is contains 31.45 pectin, 14% hemicellulose, 11% lignin and wax etc [10]. The main reasons for the use of BFs are low density and high tensile modulus [11]. Banana trees growing are very worthy for warm weather in Bangladesh. After production of banana fruits banana trees are discarded as agro-waste. It is possible to create an eco-friendly NFRCs by separating BFs from banana trees and processing. BFs are converted to micro range in a molting machine; a double acid hydrolysis process helps to remove hemicellulose and lignin and converted to cellulose nanocrystals (CNC) [12, 13]. By adding conductive nanofiller to this CNC, resistivity is reduced and conductive NFRCs is created [14]. CNTs are the most effective reinforcement element in conductive nanofiller which is thousands of time more conductive to copper [15]. CNTs have flexible mechanical properties, high aspect ratio and large surface area which have made it a promising material. The nano diameter and functional group of these nanofiller is very important to increase the conductivity of the polymer [16, 17]. In order to use CNTs as a good reinforcement element, it is necessary to create a functional group in its surface to volume ratio and there are many methods for processing such as oxidative treatment using strong acid, plasma processing techniques etc. But functional group creation in acid treatment process is so difficult, also removal of surfactant and is not eco-friendly [18].

Based on this fact, CNTs are functionalizing by using plasma processing technique which is safer and ecofriendly. After this processing surface of CNTs creates functional group which contains oxygen containing group which is easier disperse in water and make easy reinforcement filler with CNC. In plasma processing technique is cost-effective, faster and contamination-free so it is a very promising method for the dispersion of CNTs [19].

In this research MWCNTs are safely modified and carboxyl (-COOH) groups and Hydroxyl (-OH) groups functionalized in the MWCNTs surface which is easily dispersed in water. Here the combination of CNC and CNTs new NFRCs are created which properties and applications are promising [20]. The aim in the research enhances the electrical conductivity, thermal stability and mechanical properties in natural fibers. After those NFRCs is usable in the multipurpose electronics devices such as electrical sensors, super capacitors, pH sensors, energy stores devices and health monitoring devices [21-23]. Finally, modifying natural fiber and using it as a timely biodegradable NFRCs will reduce the environmental impact.

2. Methods

2.1. Materials

Banana fibers are collected from local banana garden of Bangladesh. Multiwall carbon nanotubes (MWCNTs) diameter 3-10 nm, length ≤10 μm and purity more than 95% are used to prepare CNC/CNTs nanocomposite and MWCNTs are collected from CNano Technology Ltd. Others chemical such as benzene, sodium hydroxide, sodium hypochlorite, Sodium acetate, Sodium meta-bi-sulphite, citric acid, acetic acid and ethanol are collected from Merck Germany. The functionalization of CNTs is done by pure oxygen gas which is manufactured by Linda, Bangladesh.

2.2. Preparation of CNC

Banana fibers were extracted from banana stem by hand lay-up method. Banana stem was soaked in the water 15 to 20 days than the fibers were extracted by hand and wased by clean water. After drying at 40°C temperatures the fiber is storage in a desiccator. Soda and detergent is a surface-active agent which helps to remove biodegradable fibers impurity such as fatty, waxy and gummy substances. 6g Na_2CO_3 and 4gm detergent mixed with 100 ml of distilled water and per 30 gm solution 1gm of fiber was socked at 60°C temperatures in 2h to remove impurity. After scouring banana fiber is washed out several times and dries at 40°C for farther application [24].

Scoring BFs are molted by planetary ball-milling (Denchtop-FM1) machine as powder in micro range [25]. Than the BFs powder is treated by 5% alkali solution in 2h at 60°C temperatures in 1:30 ratio (1g powder to 30gm water) which remove lignin and hemicellulose and termed alpha-cellulose [26]. Alkali treatment also improves mechanical properties [27]. After drying at 40°C temperature, then alpha-celullose powder bleached by 4% NaOCl with Ph 5 at 70°C temperature in 2 hours and the powder ratio is 1:30 (1g powder to 30ml solution). Completing double bleaching, the powder is dried at 40°C temperatures. The final stage of CNC preparation is double acid hydrolysis where H_2SO_4 solution (60wt%) is used to formation of CNC [28]. The ratio on solution is 1:15 where 1gm BFs powder to 15ml of sulfuric acid solution. After 2 hours treatment at 40°C temperatures in a magnetic hot platr it become terns to CNC from micro range cellulose. The solution is centrifuged and washed in destilled water until the Ph of 7.0 is recorded. Then the CNC extract is dried at 40°C temperature and preserve in an airtight biker with ethanol solution.

2.3. Functionalization of CNTs

Pristine CNT (*p*-CNTs) powder is weighted in balance machine in 30 gm is included into 20ml pure ethanol to functionalize and sonicated at 30°C temperature using 150VT ultra-sonic homogenizer (BioLogics Inc., USA, f=20

kHz, 6.0 mmϕ probe) at an input power of 20 W for 1 hour. After finising sonication, the suspention is dried in room temperature and soaked in 0.15 M (5 ml) of citric acid solution for more than 48 h [19]. The soaked CNTs are placed on lower electrode (SUS, 90 mmϕ) of a plasma reactor (18 cmϕ × 15 cm) in a petri dish. A rotary pump is used to reduce air the plasma chamber and the inner pressure is ca. 0.01 Torr. The schematic of the plasma setup is shown in Figure 1. Finaly very slow oxygen gas flow is included in the plasma reactor and an RF (13.56 MHz) input power of 100 W is used to generate oxygen plasma and the estimated time is 20 min, keeping pressure in this reactor is 0.13 Torr [29]. An RF power functionalized the CNTs to estimation all of element such as oxygen molecules, the evaporated water and citric acid molecules in the plasma reactor. After the treatment, the functionalized CNTs (*f*-CNTs) are washed at least three times using distilled water and dried under reduced pressure at room temperature.

2.4. Preparation of CNC/ f-CNTs Nanocomposite

This is the final step to prepare CNC/ *f*-CNTs composite and the dip-drying process are used. 2 gm BFs powder is mixed will disperse 10 mg *f*-CNTs in 10 ml distilled water for 30 min sonication. The composite is made on this solution in compressed by 3 ton pressure in a hydraulic pressed machine which creates its surface smoother and strong. In the same way, 2 gm with 10 mg *f*-CNTs in 10 ml distilled water for 30 min sonication is mixed well and it is compressed by same pressure. The CNC/ *f*-CNTs nanocomposite combines the functional groups of *f*-CNTs with with CNC to from a strong conductive network.

3. Characterization

The *f*-MWCNTs were characterized by field emission scanning electron microscopy (FESEM) (JEOL JSM-7600F) and transmission electron microscopy (TEM; HITACHI High Technology Co., H-7500). Nanofibers particle size, shape and surface morphology also studies by the use of FESEM. Surface functional groups of MWCNTs and *f*-MWCNTs were characterized by using Fourier Transform infrared spectroscopy (SIMADZU, FTIR-8400 spectrophotometer, Japan) instrument. FTIR samples were prepared by eternal dehydrated MWCNTs together with potassium bromide (KBr) to make a pellet. The thermogravimetric analysis of composites was measured by using TG-DTG instrument (TG-DTA 6300). In N_2 the measurement of the films (weight 7-11 mg) was carried out from 30°C to 650°C at a heating rate of 20°C/min. Mechanical mm/minute and gauge length was 33 mm in length were prepared from each batch and tested according properties were carried out on the samples using UTM (Instron UTM 50kN) testing speed 1 to ASTM D412. The current density-voltage (J-V) characteristics of the *f*-MWCNTs-CNC polymer nanocomposites of different wt% of *f*-MWCNTs in NF were studied at room temperature where current across the nanocomposites is measured by a high impedance electrometer (Model: 614, Keithley Instrument Inc., USA) and DC voltage is supplied step by step by a stabilizer DC power supply (Model: 6545A, Agilent, Japan).

4. Results and Discussion

4.1. Surface Morphology

FESEM microscopy of *f*-CNTs is shown in Figure 1(a) where the length and diameter respectively is 10 µm and 10-30 nm. TEM microscopy of *f*-CNTs is also shown in Figure 1(b) which indicates the well functionalization of MWCNTs.

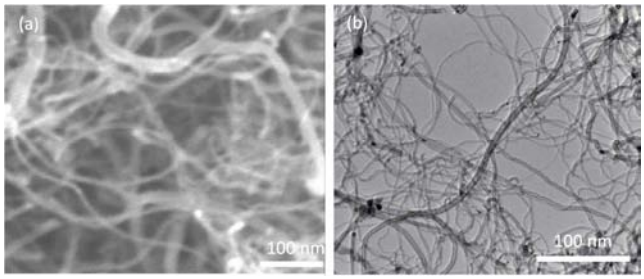

Figure 1. *(a) FESEM and (b) TEM im age of f-CNTs.*

In the aim of research BFs are prepared micro range to nanometer. In the extortion molted fiber tends to rang as 1.5 µm to 2 µm during the 3 hours period which is shown in Figure 2(a). The molted fiber become nanocellulose by double hydrolysis process and the area of CNC is 70-80 nm which is illustrated in Figure 2(b).

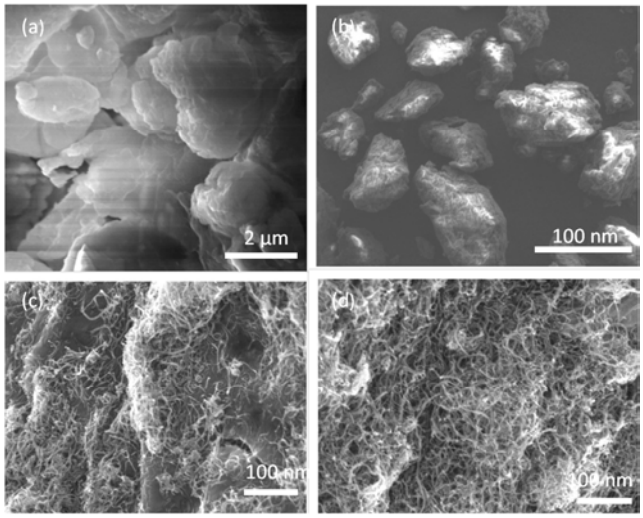

Figure 2. *FESEM images of (a) BFs, (b) CNC, (c) BFs/f-CNT and (d) CNC/f-CNT nanocomposite.*

In Figure 2(c), BFs/*f*-CNT composite are shown where *f*-CNT incorporation is ignorable because of BFs have no functional group to produce strong bond with *f*-CNT. On the other hand CNC becomes hydrophilic nature and cellulosic hydroxyl groups in the smooth surfaces of the CNC which is make a strong bond with *f*-CNT because of *f*-CNT have hydrophilic functional groups [19] and the incorporation of *f*-CNT is

homogeneous in the nanocomposite of CNC/*f*-CNT which is shown in Figure 2(d). This types of CNC/*f*-CNT nanocomposite is fully shield with CNT which produce high-density eclectic conductive network, also thermal and mechanical properties improve than BFs/*f*-CNT composite [30].

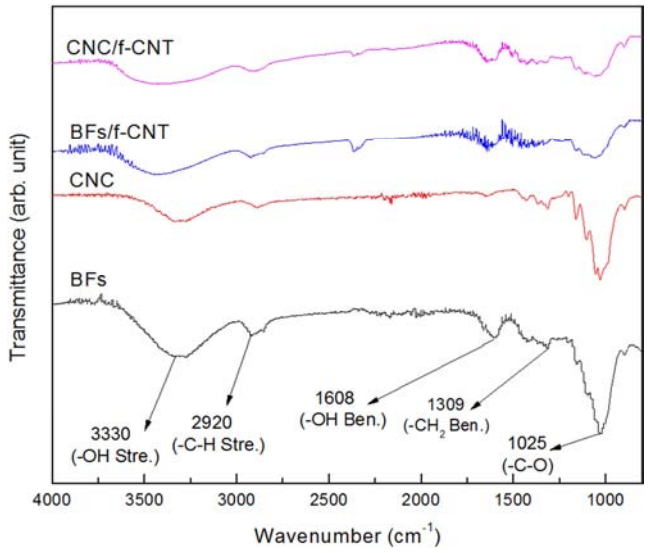

Figure 3. *FTIR spectra of the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite.*

4.2. Structural Analysis

Mostly natural fiber containing cellulose and the structural composition is studied by the use of FTIR spectrum [31, 32]. The FTIR spectrum presented the typical vibration bands of BFs, CNC, BFs/*f*-CNT and CNC/*f*-CNT. In Figure 3 has an absorption band of –OH stretching bond at around 3330 cm^{-1} for the sample of BFs and CNC which signifies the presence of numerous hydroxyl groups in it [33]. In the case of BFs a large peak at 2920 cm^{-1} indicates C –H stretching vibration for from $–CH_2$ group which conform the presence of cellulose and hemicellulose and after alkali treatment in CNC sample a small peak at 2910 cm^{-1} for cellulose because alkali treatment removes lignin and hemicellulose [34]. The specific peaks at 1608 cm^{-1} for –OH stretching bond, on other hand 1309 cm^{-1} and 1025 cm^{-1} corresponding to associate for $–CH_2$ bending and C–O–C glycosidic bonding network of cellulose and hemicellulose. After incorporation of CNTs in the BFs and CNC the bands are almost unchanged but –OH stretching bond shifted marginally to higher wavenumber. C –H stretching and $–CH_2$ bending peaks are towards lower wavenumber due to non-convalent interactions between the CNC and CNTs.

4.3. Thermal Properties

Thermal enactments of the BFs, CNC and nanocomposite are shown on Figure 4. Generally natural fiber thermal degradation involves depolymerization and decomposition of glycosyl units. The measured value of the weight loss for the samples is illustrated in Table 1. In our study T_{onset} is indicates initial where degradation starts, in temperature 40-240°C the weight loss is 2% for BFs due to evaporation of moisture however BFs moisture effect up to 6.03% [35]. 17%, 18% and 6% weight loss respectively for CNC, BFs/*f*-CNT and CNC/*f*-CNT samples because oxygen molecules may be staying in the nanocomposite. $T_{50\%}$ specifies the degradation temperature for 50% weight loss. The maximum weight loss detected at the range 240–395°C due to the degradation of cellulose and hemicellulose. TGA curves for BFs, CNC, BFs/*f*-CNT and CNC/*f*-CNT sample show residue of 12%, 18%, 23%, and 52%, respectively, after 650°C which needs higher temperature for degradation. In the case of CNC/*f*-CNT thermal stability is more enhanced which indicates well desperation of the *f*-CNTs in CNC/*f*-CNT nanocomposite.

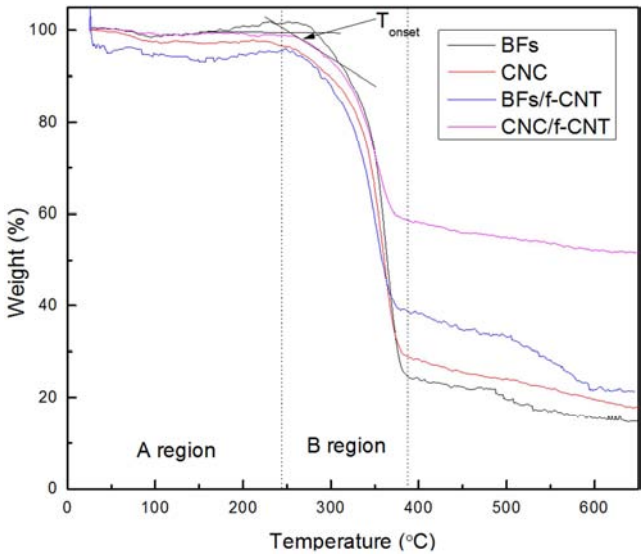

Figure 4. *a TGA curves for the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite.*

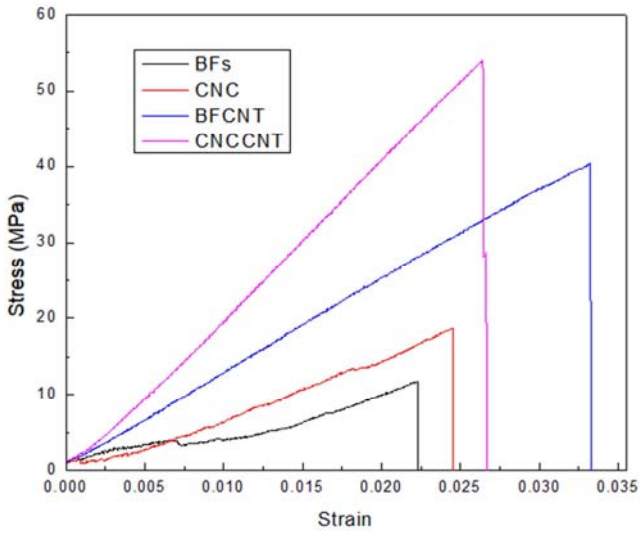

Figure 5. *Stress-strain curves for the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite.*

Table 1. *Weight loss calculation for the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite.*

Sample Name	Weight loss (%)		T_{onset} (°C)	$T_{50\%}$ (°C)
	A Region (40-240°C)	B Region (240-395°C)		
BFs	2	76	260	353
CNC	17	70	240	360
BFs/*f*-CNT	18	62	250	366
CNC/*f*-CNT	6	40	260	380

Table 2. *Tensile strength, elongation at break, and Young's modulus for the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite.*

Sample Identification	Ultimate Tensile Strength (MPa)	Elongation at Break%	Young's Modulus (MPa)
BFs	09±2.6	1.03	0.60
CNC	17±1.9	0.85	0.53
BFs/*f*-CNT	38±3.70	4.30	2.25
CNC/*f*-CNT	56±1.22	5.60	2.80

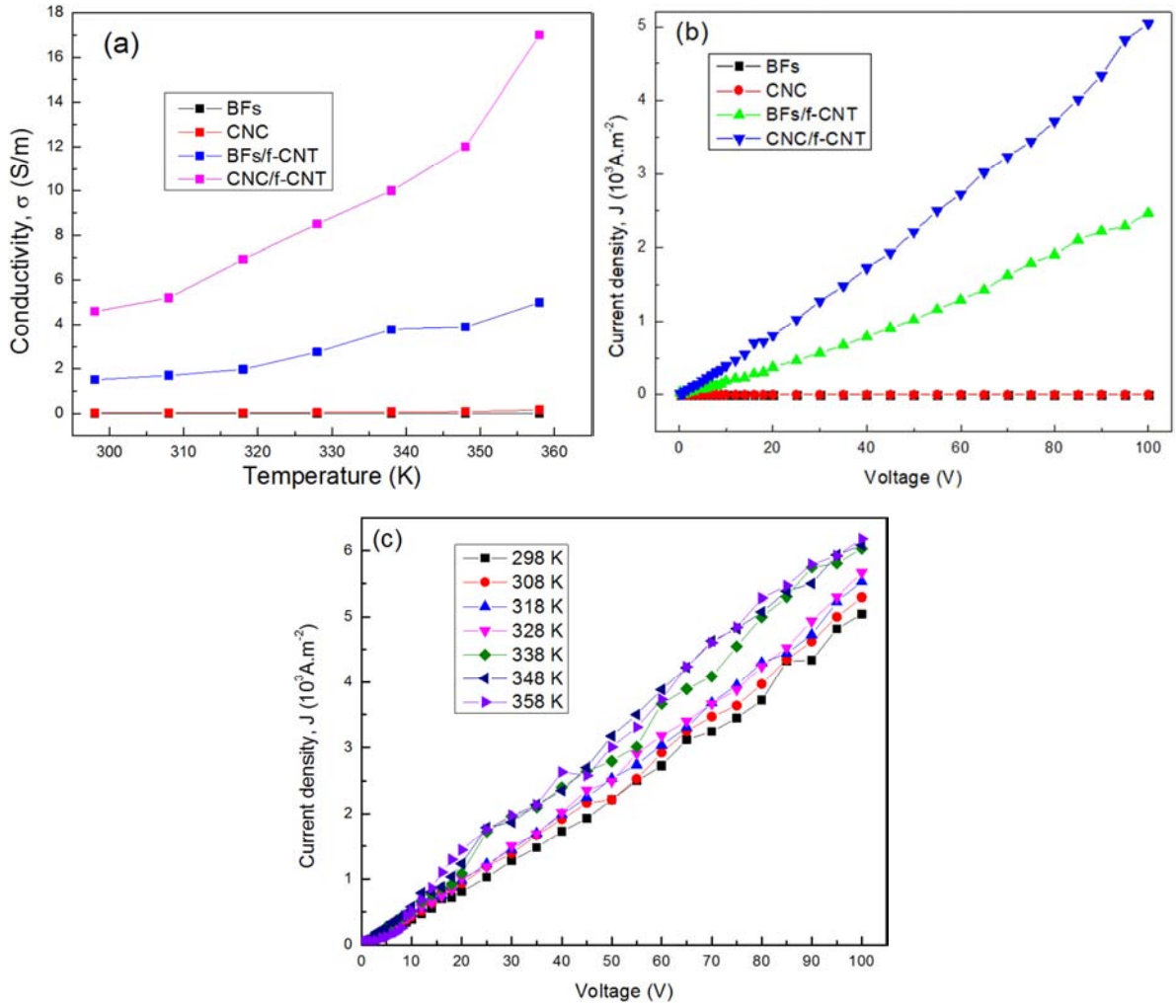

Figure 6. *(a) Conductivity with temperature for the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite (b) J-V characteristic curves for the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite at room temperature and (c) at different temperatures for the BFs, CNC, BFs/f-CNT and CNC/f-CNT nanocomposite.*

4.4. Mechanical Properties

The stress-strain carves shown in Figure 5 where tensile strength (*TS*) and elongation at break (*EB*%) of BFs, CNC, BFs/*f*-CNT and CNC/*f*-CNT are calculated and the results are illustrated in Table 2. TS and EB% are measured five times for each sample of all the samples. TS of BFs sample is very low because of inter molecular bonds are very low after making them powder besides CNC and the data enhancement are supporting the results of Bia et al. [36]. After incorporation of *f*-CNTs TS and EB% are significantly increase in natural fiber matrix also makes stiff and ductile. It is observed that, TS of the nanocomposites increase from 9 to 38 MPa respectively BFs to BFs/*f*-CNT which almost 4 times more and CNC TO CNC/f-CNT nanocoomposite the TS is

increased 17 to 56 MPa after well incorporation of *f*-CNTs.

4.5. Electrical Properties

Natural fibers have insulating properties, in our case BFs and CNC also are the same but CNC have a little bit higher electrical conductivity with increasing temperature. After incorporation of a little amount of CNTs incorporation with BFs and CNC, the nanocomposite are shown the significant influence of electrical conductivity in Figure 6 (a) is indicates the mark. The electrical conductivity, σ of the fibers is calculated using Eq. (1):

$$\sigma = \frac{l}{RA}. \quad (1)$$

Where A and l are active electrode area and length of the sample, respectively. In the case of nanocomposite electrical conductivity is increased with increasing temperature and the increases are 2 to 5 S/m for BFs/*f*-CNT and 4.5 to 17 S/m for CNC/*f*-CNT sample at 273 K to 358 K. The well dispersed-CNTs are incorporated into natural fiber; the nature of electrical conductivity becomes semiconductor behavior.

The current density-voltage (J-V) characteristic curves for the BFs, CNC, BFs/*f*-CNT, and CNC/*f*-CNT nanocomposite at different temperatures are presented in Figure 6(b and c). The J-V characteristics curves of the BFs and CNC nanofibers represent insulating behavior, but the BFs/*f*-CNT and CNC/*f*-CNT nanocomposite show a significant increase in J and the entire nanocomposite exhibit an almost linear relationship between the J and V. The J value is higher for the CNC/*f*-CNT nanocomposite as compared with that for the BFs/*f*-CNT, which confirms the low attachment of CNTs with the BFs shown in Figure 2(c) [37].

5. Conclusions

Ecofriendly, Light weight and cost effective nanocomposite are fabricated by enhancing CNTs with CNC which thermal, mechanical and electrical properties are promising. The FESEM image conformed to the formation nano size of CNC. FTIR clarified the non-covalent bond of cellulose and nanocomposite. The nanocomposites are satisfied in thermal stability, mechanical tensile strength increases 9 to 56 MPa. Biodegradable insulating natural fiber can be usable in electronic application after CNTs coding and the electrical conductivity is increased in more than 13 Sm^{-1}. Therefore Natural fiber is not only a household product but also it is usable advanced technology as well as nano filler composite.

6. Recommendations

1. Raw fiber modification to CNC which have more capability to incorporation with reinforcing materils like CNTs.
2. Further studies are needed to prepare nano sensor from this biodegradable ecofriendly composite.

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