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Studies on Nylon6/CNT Nanofiber Membrane Applications for Heavy Metal Separation from Industry Waste Water

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ABSTRACT
Due to increasing population and industrial development, share of overall water usage in the world is rising day by day. Some of the heavy metal releasing industries are Printed circuit board (PCB) manufacturers, Leather processing industries, Pesticide manufacturing industries, Agrochemicals, Paint industries etc. heavy metals are toxic to human health and may cause diarrhea, liver damage, insomnia etc. The aim of this paper is to study the nanofiber membrane mats properties for heavy metal separation from water applications .This research work focuses on preparation of nylon 6 nanocomposite nanofibre mats with 2 and 20% Multiwalled Carbon Nanotube (MWCNT).The Electro spun Nylon 6/CNT nanofiber mats are characterized for thermal, mechanical, XRD and morphological properties.

Key Words: *Nylon6, CNT, Heavy metals, Electrospinning.*

1. INTRODUCTION

Water covers 71% of earth surface which is essential for living organism survival. [1] Nowadays, heavy metals originating from anthropogenic activities are frequently detected in sediments and water which cause contamination [2-5]. From electro plating, textile industry and washing effluent heavy metals can be released to water [6]. Nylon 6 has good fiber forming capacity, hygroscopic in nature and CNT has excellent aspect ratio, mechanical stability, thermal resistance, so combination of this is made use for metal separation [7].

Electrospinning is conducted at room temperature to obtain nanofiber. Major components of electrospinning are high voltage supply, capillary tube and collector. Polymer solution present in spinneret/syringe comes out through needle and forms droplet due to solution surface tension. Due to applied voltage charge repulsion within solution takes place. So surface tension of solution is opposed by electrostatic force thereby jet forms. As jet travels, solvent evaporates and fiber collected by collector. This approach is used to obtain a number of synthetic and natural fibers in nano scale [8-9].

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When micrometer diameter of polymer fibers are reduced to nanometer, properties like small pores in fiber, high surface area than the regular fiber are obtained. These excellent properties find various applications like tissue engineering, scaffolds, nanocatalysis, protective clothing, optical electronics, filtration of air and water, biotechnology, defense etc.[9-15].

2. EXPERIMENTAL

2.1 Materials

Nylon 6 pellets of fiber forming grade was obtained from SRF Ltd Chennai. Formic acid was obtained from Chemspure Chennai. Multiwalled CNT was obtained from Redex tech Pvt Ltd Noida. All these materials were used as purchased.

2.2 Method

Inbuilt ESPION electrospinning equipment was purchased from Physics Instrument Co, Chennai for nanofiber preparation. For preparation of nanofiber mat three different types of solutions were prepared.

In 2g of Nylon 6, 0%, 2% and 20% of CNT were dispersed using magnetic Stirrer in 10ml formic acid respectively and later electrospun. 2ml syringe was used, flow rate was maintained as 0.2ml/hr, and 20KV voltage was applied for electrospinning.

2.3. Characterization

Nylon 6, Nylon 6 with 2% CNT, Nylon 6 with 20% CNT mats were characterized for the following properties.

2.3.1 Optical studies

3.1.1. Scanning electron microscope (SEM)

Surface microstructure was studied using S-3400 **SEM** instrument for Nanofiber mats.

2.3.1.2. X ray diffraction (XRD)

WAXRD patterns of nanofiber mats were recorded using Rigaku instrument , Model Miniflex 600, with area detector operating system 30KV and 30mA of Cu Kalpha source Wavelength=1.54A^{o}.

2.3.2. Mechanical properties

Using Dak system Inc minitech Universal testing Machine, series 9000 having capacity of 250kg, Tensile test was carried out. Specimen size of length 30 mm, width 12mm, gauge length 20mm, cross head speed 12.7mm/min and Mat thickness 0.011mm were the conditions for testing [16].

2.3.3. Thermal studies

Using SII Nanotech TG/DTA6200 Instrument Thermo gravimetric analysis and Differential thermal analysis (DTA) were conducted. 5mg of sample was heated up to 800^{o}C from room temperature. Heating rate was 20^{o}C in nitrogen atmosphere.

3. RESULTS AND DISCUSSION

3.1. Scanninig Electron Microscopy

Prepared nanocomposite mats were characterized for morphology i.e. Dispersion and orientation of CNT in Nylon6 using SEM. Fiber diameter was determined using image analysis software. Figure 1a shows that electrospun nanofibers were randomly oriented and average diameter of neat Nylon 6 single fiber was 228.5nm.

From Figure 1.b Nylon 6 single nanofiber diameter was found to be 232.9nm. CNT are nearly oriented in fiber direction this is in good match with published literature [17].

From Figure 1c. it is observed that CNT appeared as bright tiny dots for Nylon 6 with 20% CNT and this is found absent in neat Nylon 6 or in nylon 6 containing 2% CNT.

3.2. X-Ray Difraction

From Figure 2a It is observed that sharp peak ensures that material is crystalline in nature. Since it is crystalline, it is fiber forming grade polymer.

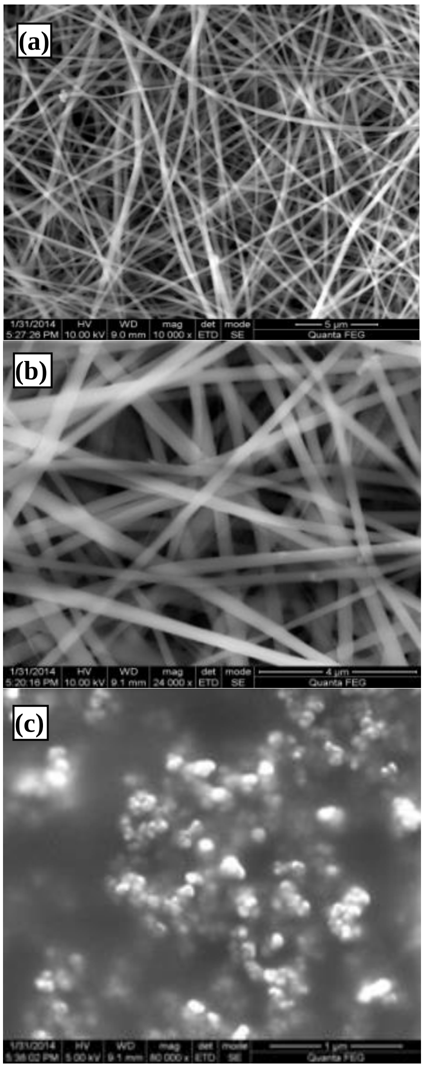

Figure 1. SEM image of Nylon 6 nanofiber (a), SEM images of Nylon 6/2% CNT nanofiber (b), SEM images of Nylon 6/20% nanofiber (c). And 2θ value of 21.4^{o} proves material is Nylon 6.

This result is in good correlation with [18].From XRD data, it is found that 2θ values are super imposable for Nylon6 nanofiber and Nylon6/2% CNT and 20% CNT showing good dispersion of CNTs in the nylon matrix.

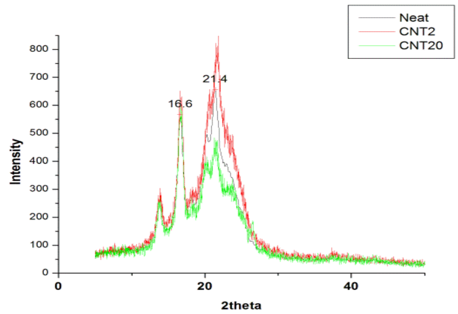

Figure 2. XRD pattern for Nylon 6 nanofiber with and without CNT.

3.3. Tensile test

From Table 1. It is clear that with the incorporation of 2% CNT to nylon 6 there is a significant increase in tensile strength value by 30% and elongation at break by 50%. On the other hand 20 % CNT addition to Nylon 6 do not have much effect on tensile properties. This may be due to the poor dispersion of CNTs in nylon 6 matrix.

Table1: Tensile values for Nylon 6, Nylon6/2%CNT and Nylon 6/20% CNT nanofiber mats

Nanofiber mat	Initial decomposition temperature	50% decomposition temperature
Nylon 6	300	414.9
Nylon 6 with 2% CNT	354.4	454.5
Nylon 6 with 20% CNT	316.6	450.7

3.4 Thermo gravimetric analysis (TGA)

Figure 3 and Table 2 show the results obtained in TGA. From Table 2 it is found that initial decomposition temperature and decomposition temperature at 50% of Nylon 6/ 2% CNT nanofiber mat is better when compared to Nylon 6 nanofiber mat and Nylon 6/ 20% CNT nanofiber mat.

3.5 Differential thermal analysis

DTA analysis of the nylon samples does not show much change in the melting point values which was aound 220 °C)

3.6. Nylon 6 nanofiber mats for separation studies.

Cupric chloride solutions of concentrations from 50ppm to 1000ppm were prepared and the absorbance values are measured using UV-Vis spectrophotometer. The nylon6 with 2% CNT mats were kept in Cupric chloride solutions for half hr and the solutions were tested for UV – Vis absorbance. These absorbance values are plotted in

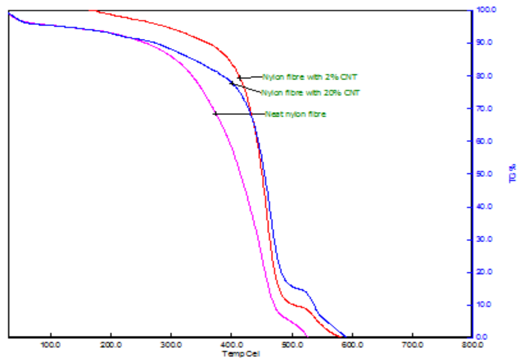

Figure 3. TGA curves for Nylon 6, Nylon6 /2% CNT nanofiber mat and Nylon 6 /20 % CNT nanofiber mat.

Table 2: TGA values for Nylon 6, Nylon6/2%CNT and Nylon 6/20% CNT nanofiber mat

Property	Nylon 6 nanofiber	Nylon 6 with 2% CNT	Nylon 6 with 20 % CNT
Tensile strength (Mpa)	20.63	26.65	24.22
Elongation at break(%)	41.49	62.26	41.29

Figure 4. There is a decrease in the absobance values with nylon /CNT fiber mat suggesting that it absorbs the cupric chloride solution. Further studies are in progress to find out the efficiency of separation process

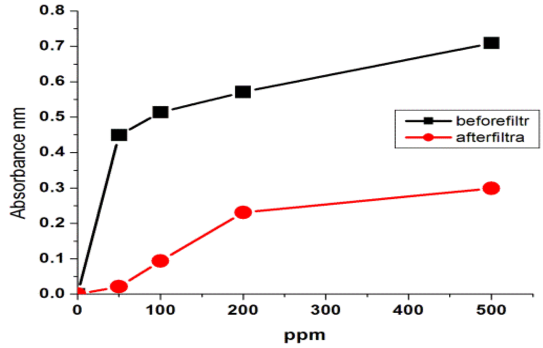

Figure 4. UV-Vis absorption studies

4. CONCLUSION

Various Nylon 6 nanofiber mats with and without CNT(2% and 20%) were prepared using Electrospinning process. Then these mats were characterized for various properties like SEM, XRD, Tensile, TGA, DTA. Nylon 6 / 2% CNT nanofiber mat showed outstanding properties. Further nylon6 / 2% CNT was used for copper l separation. Preliminary UV-Vis spectroscopic studies show that nylon with 2% CNT absorbs copper salt. Further studies are in progress.

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