## USE OF ELECTROSPINNING TECHNIQUE IN PRODUCTION OF CHITOSAN/CARBON NANOTUBES

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Chitosan/carbon nanotube nanocomposite fabrics have been successfully prepared through electrospinning. The electrospun nonwoven fabric was characterized by scanning electronic microscopic (SEM) imaging. Under optimization conditions, homogenous chitosan/carbon nanotube nanofibers with a mean diameter of 455 nm and known physical characteristics were prepared.

Keywords: carbon nanotubes, chitosan, electrospinning, biocomposites, nanocomposites, industrial tools

#### **INTRODUCTION**

Over the recent decades, the fabrication of polymer nanofibers for many biomedical applications, such as tissue engineering, drug wound delivery. dressing. enzyme immobilization, etc., has been recorded.<sup>1</sup> Nanofiber fabrics have unique characteristics, such as very large surface area, ease of functionalisation for various purposes and superior mechanical properties. Electrospinning is an important technique that can be used for the production of polymer nanofibers with diameters from several micrometers down to tens of nanometers. In electrospinning, the charged jets of a polymer solution, collected on a target, are created by using an electrostatic force. Many parameters can influence the quality of fibers, including the solution properties (polymer concentration, solvent volatility and solution conductivity), the governing variables (flow rate, voltage and tip-to-collector distance), and the ambient parameters (humidity, solution temperature, air velocity in the electrospinning chamber).<sup>2</sup>

In recent years, scientists have manifested increased interest in electrospinning of natural materials, such as collagen,<sup>3,4</sup> fibrogen,<sup>5</sup> gelatin,<sup>6</sup> silk,<sup>7</sup> chitin<sup>8</sup> and chitosan,<sup>9,10</sup> due to their high biocompatible and biodegradable properties. Chitin is the second most abundant natural

polymer in the world and chitosan (poly-(1-4)-2amino-2-deoxy- $\beta$ -D-glucose) is the deacetylated product of chitin.<sup>11</sup>

Researchers are interested in this natural polymer because of its properties, including solid-state structure and chain conformations in dissolved state.<sup>12</sup>

This short communication discusses electrospinning of chitosan/carbon nanotube dispersion. The SEM images show homogenous chitosan/carbon nanotube nanofibers with a mean diameter of 455 nm.

#### EXPERIMENTAL

#### Materials

Chitosan polymer (deacetylation degree of 85% and molecular weight of  $5 \times 10^5$ ) was supplied by Sigma-Aldrich, and the multi-walled carbon nanotube, with an average diameter of 4 nm and purity of about 98%, was provided by Nutrino.

# Electrospinning of chitosan/carbon nanotube dispersion

The multi-walled carbon nanotube was sonicated for 10 min in solvent and then stirred for 24 h. About 3 mL of chitosan/carbon nanotube dispersion were placed into a 5 mL syringe with a stainless steel needle having an inert diameter of 0.6 mm, connected to a positive electrode. An aluminum foil, used as a collector screen, was connected to the ground. A high

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voltage power supply (Gamma High Voltage Researcher ES30P-5W) generated DC voltages in the range of 1-25 kV. The voltage and tip-to-collector distance were fixed at 18-24 kV and 4-10 cm, respectively. The electrospinning experiments were performed at room temperature.

## **RESULTS AND DISCUSSION**

Different solvents, including acetic acid 1-90%, formic acid and TFA/DCM, were tested for the electrospinning of chitosan/carbon nanotube. No jet was seen on applying a high voltage, even above 25 kV, by using acetic acid (1-30%) and formic acid as a solvent for the chitosan/carbon nanotube. When 30-90% acetic acid was used as a solvent, the beads were deposited on the collector. Therefore, under these conditions, no nanofiber of carbon nanotube/chitosan was obtained.

Figure 1 shows the scanning electronic micrographs of carbon chitosan/nanotube electrospun fibers, at different chitosan concentrations, in a TFA/DCM (70:30) solvent. As presented in Figure 1a, at low concentrations of chitosan, the beads deposited on the collector and the thin fibers coexisted. As the concentration

of chitosan increased (Figs. 1a-c), the beads decreased significantly. Figure 1c shows homogenous electrospun fibers with minimum beads, thin fibers and interconnected fibers. The increase of chitosan concentration leads to an increase of the interconnected fibers, as shown in average diameter of Figures 1d-e. The chitosan/carbon nanotube fibers increased when increasing the concentration of chitosan (Figs. 1 a-e). Hence, a chitosan/carbon nanotube solution of TFA/DCM (70:30) with 10 wt% of chitosan assured optimized conditions for electrospinning of this solution. When the voltage was low, the beads were deposited on the collector (Fig. 2a). As shown in Figure 2a-d, the number of beads decreased with increasing voltage from 18 to 24 kV. In our study, the average diameter of fibers prepared by 18 kV was measured as 307 nm. As the applied voltage increased, the average fiber diameters also increased. The average diameter of fibers for 20 kV (2b), 22 kV (2c), and 24 kV (2d), was 308 (194-792), 448 (267-656) and 455 (306-672), respectively.



Figure 1: Scanning electron micrographs of electrospun fibers at different chitosan concentrations (wt%): (a) 8, (b) 9, (c) 10, (d) 11, (e) 12, 24 kV, 5 cm, TFA/DCM: 70/30



Figure 2: Scanning electron micrographs of electrospun fibers at different voltages (kV): (a) 18, (b) 20, (c) 22, (d) 24, 5 cm, 10 wt%, TFA/DCM: 70/30



Figure 3: Scanning electron micrographs of electrospun fibers of chitosan/carbon nanotubes at different tip-to-collector distances (cm): (a) 4, (b) 5, (c) 6, (d) 7, (e) 8, (f) 10, 24 kV, 10 wt%, TFA/DCM: 70/30

The morphologies of chitosan/carbon nanotube electrospun fibers at different tip-to-collector distances are presented in Figure 3. When the tipto-collector distance was low, a little interconnected fiber (with high fiber diameter) was deposited on the collector (as shown in Fig. 3a). At a 5 cm tip-to-collector distance (Fig. 3b), more homogenous fibers with negligible beads were obtained. However, the beads increased with increasing the tip-to-collector distance (Figs. 3b to 3f).

Also, our study has demonstrated that the diameter of electrospun fibers decreased by increasing tip-to-collector distance (as shown in Figs. 3b, 3c, 3d; 455 (306-672), 134 (87-163), 107 (71-196)). The fibers prepared within a distance of 8 cm (Fig. 3e) and 10 cm (Fig. 3f) presented defects and non-homogenous diameter. However, a 5-cm tip-to-collector distance appears to be reliable for electrospinning.

## CONCLUSION

Several solvents, including acetic acid 1-90%, formic acid and TFA/DCM (70:30), were used for electrospinning of chitosan/carbon nanotube dispersion. It has been observed that the TFA/DCM (70:30) solvent is the only solvent with a proper reliablity for the electrospinnability of chitosan/carbon nanotube. This is a significant the improvement in electrospinning of chitosan/carbon nanotube dispersion. It was also observed that homogenous fibers with an average diameter of 455 nm (306-672) could be prepared with chitosan/carbon nanotube dispersion in TFA/DCM 70:30. In addition, the SEM images showed that the fiber diameter decreased by decreasing voltage and increasing the tip-tocollector distance.

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