# CNT Reinforced Nanocomposite Fiber Fabrication for Undergraduate Students

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**Abatract**

The research, development and teaching of nanofiber fabrication and characterization have recently gained much attention due to their unique properties and potential applications in various fields, such as medical, engineering, basic science and defense. Electrospinning is a unique method to produce nano/micro size (10 nm – 10 μm) polymeric wires/fibers that consist of higher surface area, porosity and flexibility when compared to conventional techniques. The objective of this study was to reinforce electrospun nanofibers using multi-wall carbon nanotubes (MWCNTs) and demonstrate the nanomanufacturing to undergraduate students in the College of Engineering at Wichita State University (WSU). In the present study, we added MWCNTs in the range of 0.5%, 1% and 2% in a polymeric solution (polyvinylpyrrolidone dissolved in ethanol) and determined the concentration effects on size and morphology of electrospun nanocomposite fibers.

**Keywords**: CNTs, electrospun nanofibers and demonstration for undergraduate students.

## 1. INTRODUCTION

### 1.1 Nanotechnology Education

Nanotechnology is the fabrication, manipulation and characterization of materials at nanoscale (usually between 1 and 100 nm), which will significantly affect economical, educational and social developments in all areas, such as engineering, science, defense, biomedical, biology, etc. It is one of the leading technologies for educational revolution in the new millennium. Nanotechnology education is being offered by many universities around the world for the integration of all engineering and science courses for our future generation [17-20].

Several nanotechnology programs and centers in the U.S., Japan, Europe, Australia and many other countries have been undertaken both by the government and private sectors to intensify the teaching, research and development in nanotechnology. It is reported that development in nanotechnology will change the traditional practices of design, analysis, simulation and manufacturing for new engineering products. This is a challenge for the academic community to educate engineering and science students with all the necessary information and leadership in this emerging field [17].

The corresponding author has developed two new courses, namely Nanomaterials Fabrication and Characterization (MS and PhD level) and Introduction to Nanotechnology (undergraduate level) in the College of Engineering at Wichita State University (WSU). In the first nanotechnology course, nanoparticles, nanofibers, nanofilms, nanotubes, nanocomposites fabrication techniques, and their applications were taught in detail. There were 35 students in the spring 2007 class, most of whom were engineering students. Homework sets involved the evaluation of recently published nanotechnology papers. In the term project, the students prepared reports on applications of nanomaterials and devices, such as nanocomposite manufacturing for aircraft industry, nanoelectromechanical systems, microelectromechanical systems, and nanotechnology applications for fuel and solar cells.

In the second nanotechnology course, which is offered in Fall 2007, the similar topics were covered at introductory level. There were homeworks, term projects, exams and laboratory sessions. A nanotechnology laboratory has also been set up in the Department of Mechanical Engineering at WSU, and dedicated to perform a number of nanotechnology experiments for students. This laboratory has several pieces of new equipment, such as atomic force microscope (AFM), corrosion testing units, electrospinning unit, UV lithography, dry and wet etching, plasma cleaner, AC/DC power units, UV-Vis spectroscopy, optical microscopes, zeta potential / nanosizer, fume hood, spin coating, capacitance bridge, contact angle and surface tension measurement devices, and electrostatic self-assembly (ESA) nanofilm coating unit. We plan to have undergraduate students work on the electrospinning method in the nanotechnology laboratory. Students will produce nanofibers using the described electrospinning method and then characterize properties, such as fiber size, porosity, mechanical strength and sound absorption utilizing the laboratory equipment available in the laboratory. This will provide them a first hand-on-experience on how nanomaterials can be produced and later how their properties can be characterized.

### 1.2. Electrospinning

Electrospinning is relatively easy and direct method of fabricating a non-woven mat of polymeric fibers compared to the conventional methods, such as melt spinning, wet spinning and extrusion molding. It offers a distinct advantage of forming fibers in the micro to nanometer range and a high surface area to volume ratio compared to the conventional fibers [1,2]. Electrospinning utilizes high electric field (or force) on the surface of a polymeric solution to overcome the surface tension, and produce a very slim charged jet. When charge is applied, mutual charge repulsion induces longitudinal stresses. As the intensity of the electrostatic field is increased beyond certain limits, the hemispherical surface of the solution at the tip of the capillary elongates to form a unique structure, called Taylor cone [1-4]. The jet first extends in a straight path for some distance, called jet length, and then instability occurs by bending the jet into a looping path that gives rise to a series of spiral motions [5,6]. In order to minimize these instabilities the jet undergoes large amount of plastic stretching that consequently reduces the fiber diameter towards to nanoscale. Finally, the solvent evaporates and nanofibers are collected on a collector screen placed at some distance from the capillary as in shown in Figure 1 [14].

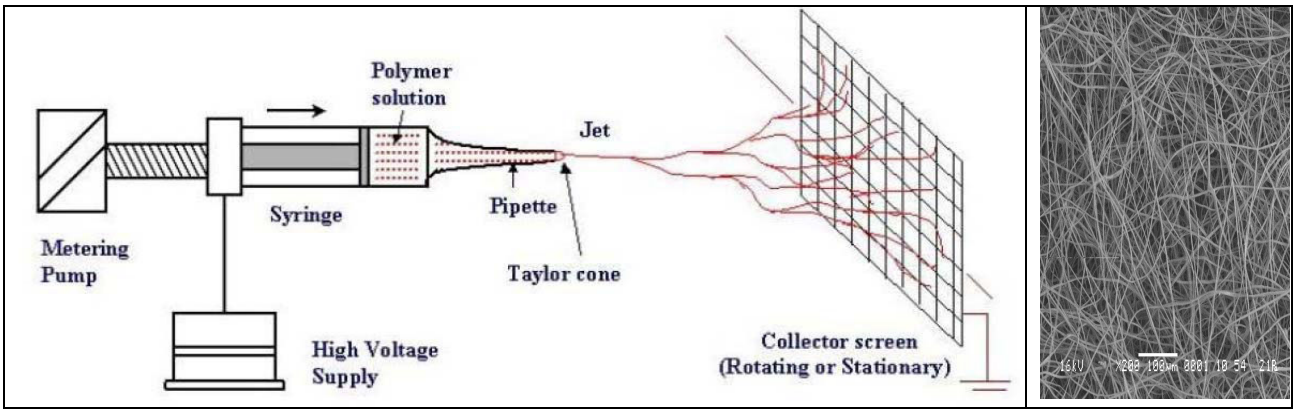


**Figure 1**: Schematic illustration of electrospinning process (left) and SEM image showing polymeric nanofibers.

Generally, the diameters of electrospun nanofibers are in the range of 50 nm to 500 nm, and they can be as small as 10 nm by changing the system and process parameters as is given below [1-4]. Understanding and optimizing of these parameters would finally result in a nanoscale fiber fabrication with a minimum bead formation.

***1. System Parameters***

- Polymer and solvent types and structures
- Viscosity, conductivity/chargeability and surface tension of polymers

***2. Process Parameters***

- Electric potential, flow rate and polymer concentration
- Distance between the capillary and collection screen
- Temperature, humidity and air velocity
- Nanoparticle inclusions
- Target rotation

The Electrospinning process is usually carried out at room temperature under normal conditions. However, if the temperature is a little above the room temperature, the evaporation rate will increase, which can help in reducing the fiber diameter. Increased humidity and airflow reduce fiber diameter to some extent. At high concentration or molecular weight, sufficient molecular chain entanglement in the polymer solution prevents the breakup of the electrically driven jet and allows the electrostatic field to elongate the jet. It has been experimentally determined that at higher viscosity, that spinning drop changed from hemispherical to conical shape, and the length of the jet improved. The spinning voltage needed to eject a jet from the tip of a capillary depends upon the solution viscosity. If the solution viscosity is too high, higher spinning voltage is needed to overcome the surface tension and viscoelastic forces. High voltage reduces the fiber diameter. Therefore, the solution viscosity should be compromised between very low and very high values depending on the needs. The tip to target distance has remarkable effect on electrospun fibers. As the distance increases, the jet will have longer time to stretch plastically, during which time more and more solvent evaporates, and hence the fiber diameter reduces. The morphology of the fibers depends upon the flow rate of the solution and some other parameters above. When the flow rate exceeds the threshold value, beads and pores on the fibers can be

observed. As the conductivity of the solution gets higher, the fiber diameter will get lower, too [1-8].

The electrospun nanofibers have a large surface area per unit mass, so these non-woven fibers can be used in various fields: filtration and separation of micron, submicron and nanosize organic and inorganic particles; HF antenna fabrication; light weight, colorful and invisible fabric productions; and biomedical applications, such as wound dressing in medical industry, tissue engineering scaffolds and artificial blood vessels. Other promising areas of electrospun nanofibers include advanced nanocomposites fabrications to improve crack resistance and aircraft interior noise reductions [4-8].

Even though a significant progress has been made for over a decade to increase the properties of nanofibers, the problem of mechanically strength nanofiber fabrication has not been solved yet. This paper presents the fabrication of mechanically strength nanocomposite fiber manufacturing using polyvinylpyrrolidone (PVP) [13], ethanol [12], and multi-wall carbon nanotubes (MWCNTs). In this demonstration, students learn, in detail, all the sample preparation and nanomanufacturing of nanocomposite fibers by electrospinning.

## 2. EXPERIMENTAL

### 2.1 Materials

Unless otherwise specified, all chemicals used in the present studies were purchased from Sigma-Aldrich and are all reagent grades. PVP with a molecular weight of 130,000 and ethanol were directly used without further purifications or modifications [12,13]. MWCNTs (Fisher Scientific) has a diameter of 140 (+/- 30) nm and a length of 7 (+/- 2) microns.

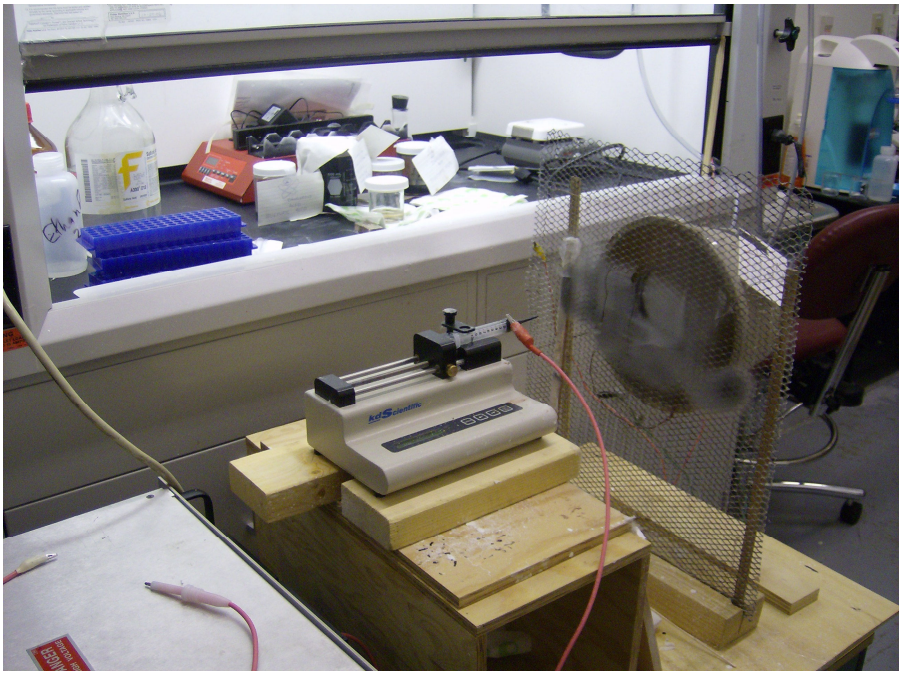

**Figure 2**: The photograph showing the electrospinning unit utilized in the present study.

## 2.2 Methods

Ethanol was used as a solvent for the dispersion of the MWCNTs due to the better solubility and dispersion ability. PVP was added to the dispersed solutions containing 0.5%, 1% and 2% of MWCNTs while stirring at 40 $^0$C, and then the dispersion was again continued to stir for additional 2 hrs. The prepared dispersion/solution was transferred to a syringe that was connected to a capillary needle with an inside diameter of 0.5 mm. A platinum electrode inside the syringe was attached to a high voltage DC supply for the nanocomposite fiber fabrication. Electrospun fibers were then collected on a grounded screen, and dried in an oven at 60 $^0$C for 8 hrs to remove all the residual solvent. Scanning electron microscope (SEM) was used to find out the fiber diameter and morphology. Figure 2 shows the photograph of the electrospinning unit utilized in the present study.

## 3. RESULTS AND DISCUSSION

In these experiments, only the MWCNT concentrations were changed from 0% through 2% while other processing parameters, such as PVP:Ethanol concentration (15:85), mass flow rate (3 ml/hr), distance (15 cm) and applied voltage (18 kV) were kept constant in order to determine the effects of MWCNTs on the fiber size and morphology. Figure 3 shows the SEM images of the nanocomposite fiber diameters and morphologies as a function of MWCNT additions. It is determined that the loading of MWCNTs generally resulted in increasing of nanocomposite fiber diameters. For instance, the average fiber diameter of 0% MWCNTs is about 700 nm, while that of 1% CNTs is about 1.6 μm. This may be because of the viscosity increase in the solution by increasing the load. A relationship between viscosity (η) and fiber diameter d) is given as: $d = 19.49\ \eta^{0.43}$ in which the fibers diameters increase with increasing solution viscosity [16]. As the concentrations of MWCNTs are varied, the fiber morphologies are varied too. It was also observed that reducing the viscosity of the solution led to break up of an electrically driven jet into individual droplets to promote bead formations in the nanofiber structure. Table 1 shows the summary of MWCNT reinforced nanocomposite fiber fabrication conditions and their average diameters.

**Table 1**: Conditions for nanocomposite fiber fabrication at various MWCNT concentrations and average fiber diameters.

| **Conditions** | **Average Fibers Diameter** |
|---|---|
| PVP/Ethanol (15:85) + 0.0 wt % MWCNTs | 700 nm |
| PVP/Ethanol (15:85) + 0.5 wt % MWCNTs | 1.4 μm |
| PVP/Ethanol (15:85) + 1.0 wt % MWCNTs | 1.6 μm |
| PVP/Ethanol (15:85) + 2.0 wt % MWCNTs | 1.5 μm |

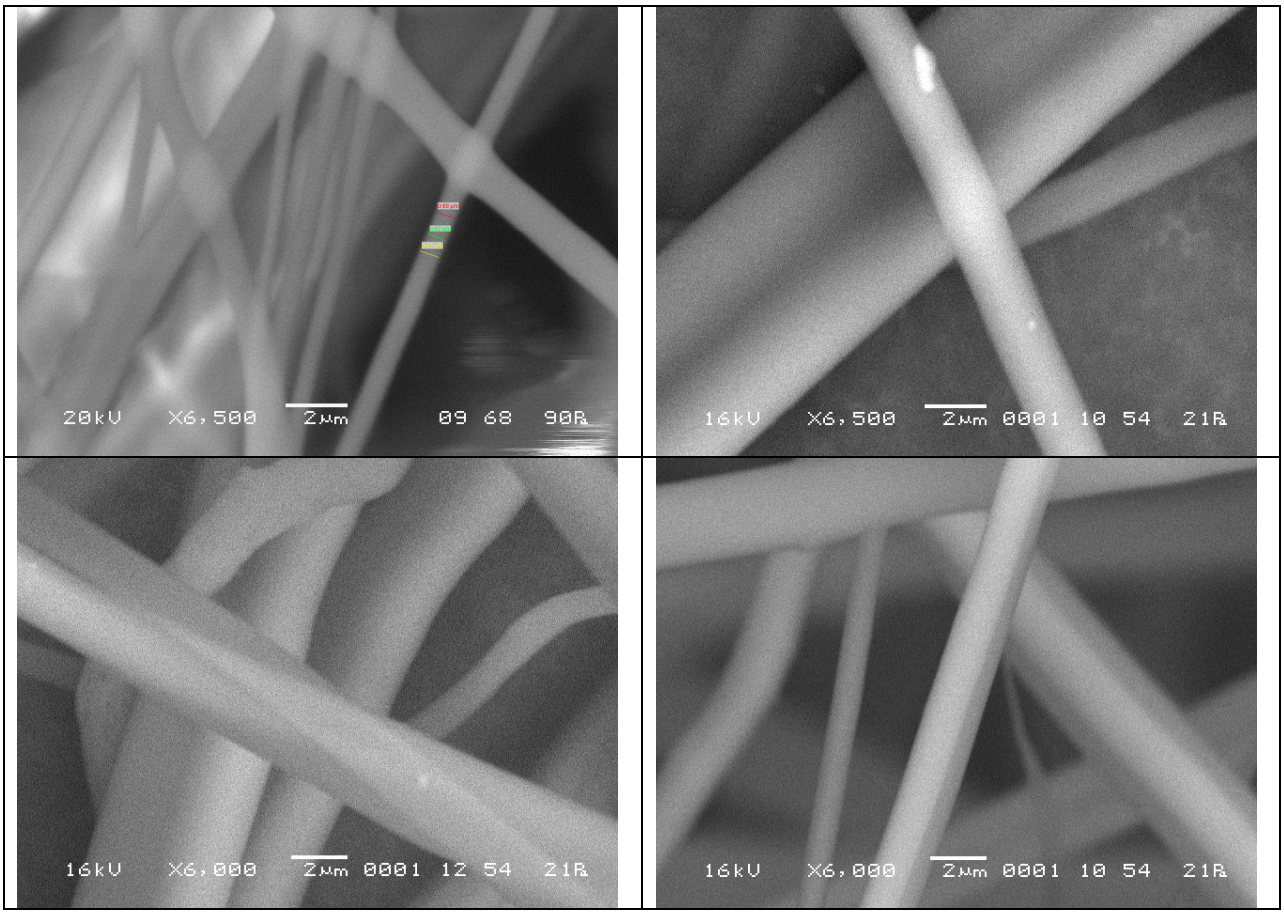


**Figure 3**: SEM images showing the MWCNT reinforced nanocomposite fibers and morphologies as a function of concentrations.

## 4. CONCLUSION

In the present study, we have demonstrated the synthesis of MWCNT reinforced nanocomposite fibers using electrospinning method. CNTs in the range of 0% to 2% were dispersed with ethanol, and then PVP was added to the dispersion. Electrospinning experiments were conducted on these samples at constant pump speed, DC voltage, solvent concentration and ground mesh screen distance. The SEM analysis proved that the average diameters of nanocomposite fibers were increased from 700 nm to 1.6 μm by increasing the CNT concentrations. This demonstration improves the knowledge of the students on how to design, analyze and manufacture nanomaterials and devices using electrospinning method.

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