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Carbon nanotubes in electrospun polyethylene oxide nanofibres: *A potential route to conducting nanofibres*

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Abstract. Polyethylene oxide solution containing multi-walled carbon nanotubes have been electrospun onto a rotating collector to produce highly aligned arrays of electrospun nanofibers ranging in diameters from (200 – 360) nanometres. The addition of a surfactant (Triton X-100) is highly effective in dispersing carbon nanotube within an aqueous solution of polyethylene oxide and the resulting mixture can be electrospun without excessive clumping to produce nanofibers containing high loadings of nanotubes; in this case up to 5% wt thereby providing an effective route to electrically conductive nanofibres.

1. Introduction

Electrospinning is a manufacturing technique that utilises an electric field to prepare micro- to nano-scale polymer fibres from a solution or melt [1]. The process works by application of a high voltage to a polymer solution contained within a spinneret. The droplet at the spinneret tip is extruded as a jet and through the process of jet whipping (referred to as bending instabilities), the jet is stretched into a micro-to-nano scale, solvent evaporates off and a solid polymer fibre is deposited onto a grounded electrode. Electrospun fibres offer some useful applications such as in electronics, biomedical applications, textiles and composite materials [1]. The fibres themselves boast impressive properties, which can be further improved with the addition of nanoparticles, for example carbon nanotubes (CNT's), which have an abundance of properties ranging from thermal, optical, electrical and mechanical [2]. CNT's typically have diameters on the order of several nanometres, but they can be micrometres in length. The tubes exhibit improved electrical and mechanical properties parallel to its axis making alignment desirable [3], which makes the electrospinning process attractive for as a method of alignment due to shear forces and narrowing of the jet which can aid in orientating and confining the tubes parallel to the fibre axis [4]. In addition to alignment, nanoscale particle agglomeration from strong intermolecular forces must be overcome to aid in a uniform dispersion. This can be facilitated through use of surfactants, for example in one such study Rastogi *et al.* showed Triton X-100 providing a good nanotube dispersion [5].

Previous studies incorporating CNT's into electrospun fibres have shown improvements to the electrical conductivity of the fibres. Examples include the addition of nanotubes to polystyrene fibres



[6], polyvinylidene difluoride [7] and polypropylene [8], all cases showing improvements to fibre conductivity. The key to high electrical conductivity is the network connectivity of the CNT's, with higher loadings naturally leading to an enhanced number of contacts between tubes. There are three possible models of nanotube incorporation outlined in Figure 1. In Type A carbon nanotubes are stabilised by a rigid polymer matrix which solidifies as the fibres are produced; in Type B the orientation is stabilised by cross-linking, in Type C the nanotubes are linked together as a polymer chain and in this scenario, the system resembles a main-chain liquid crystal polymer [9] contained within a polymer matrix. In this work cross-linking was not invoked so the system resembles more closely (a).

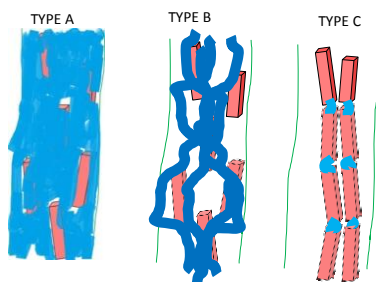


Figure 1: Routes to retaining alignment of the nanotubes in a polymer composite fibre

In this study we propose a methodology for preparing nanofibers containing high concentrations of carbon nanotube loadings using the polymer matrix to retain the alignment of the tubes. For this study we selected polyethylene oxide as the encapsulating polymer as it can be processed in aqueous solution, i.e. without the use of volatile organic compounds and that it readily produces nano scale fibres.

2. Materials and Methods

2.1. Materials

Multi-walled carbon nanotubes (MWCNT) purchased from Sigma-Aldrich with an outer diameter of 6–9nm and a length of $\sim 5\mu\text{m}$ were used. Polyethylene oxide (PEO), M_v 900,000 gmol^{-1} (Sigma-Aldrich) was selected as the encapsulating material. Solutions were prepared using deionised water as the solvent with the addition of the surfactant, Triton X-100 (TX-100) (Sigma-Aldrich) in selected solutions to aid nanotube dispersion.

2.2. Methods

2.2.1. Solution Preparation

Aqueous (deionised water) solutions/suspensions were prepared by weight with loadings of MWCNTS corresponding to 0, 0.05%, 1%, 3% and 5% wt in the final fibre mat. The mixtures were stirred using a vortex mixer and placed in a sonicator for 4-5 hours to encourage dispersion. 1% wt TX-100 was added to selected solutions prior to stirring and sonication, corresponding to $\sim 16\%$ wt loading of surfactant in the final fibres. Suspensions prepared with surfactant appeared to have a better dispersion of nanotubes based upon a visual inspection. After sonication PEO was added to the suspension at 4.8% wt concentration and allowed to dissolve with further sonication and mixing on the vortex mixer.

2.2.2. Electrospinning

The electrospinning apparatus was arranged in a horizontal configuration. Solutions were loaded into plastic syringes with 22 gauge metallic needle (0.41mm inner diameter) attached and placed into a syringe pump set to 4.07mLhr^{-1} . A Glassman 0 – 30kV high voltage power supply was used to supply the high voltage. Electrospinning was performed using a potential of 15kV at the needle with samples

collected onto a grounded rotating collector, with a surface tangential velocity 7ms^{-1} positioned 15cm from the needle tip. A layer of aluminium foil placed over the surface of the rotating drum was used as the collector.

2.2.3. Electron Microscopy

Scanning electron microscopy (SEM) was performed on sections of the fibre mats produced. The microscopy was performed using a Cambridge Instruments SEM360 in high vacuum mode with an accelerating voltage of 20kV. Fibres were coated with gold prior to examination. Fibres were selected at random from micrographs obtained and measurements made using the software *ImageJ*.

Transmission electron microscopy (TEM) of the fibres was performed using a Philips CM20 at an operating voltages of 200kV with samples prepared on a copper TEM grid, 300 mesh. Samples were prepared for TEM by electrospinning directly onto the grid which was attached to the foil on the rotating collector. TEM on a pure sample of nanotubes was performed on a JEOL 2010 at 200kV. The sample was ground up in isopropyl alcohol with a pestle and mortar then pipetted onto a Lacy carbon TEM grid.

3. Results and Discussion

Preliminary observations of the electrospinning process were conducted by eye. For solutions containing CNT's without the surfactant, dripping occurred from the needle tip throughout the spinning process, where as those solutions with surfactant only the higher CNT loadings, 3% and 5% wt exhibited any observable dripping. The level of dripping still appeared to be to a lesser extent than that of the solutions without surfactant. The dripping was attributed to agglomeration of the nanotubes at the needle tip producing a large scale material that cannot be incorporated into the nanofibre and thus drips off. The presence of the surfactant allowed for a better dispersion of the nanotubes, thereby allowing a more continuous electrospinning process and hence electrospinning of fibres containing higher CNT concentrations. Fibre mats collected for different nanotube loadings were found to vary in appearance with higher loadings having a darker appearance.

Examination of the fibre mats through the use of SEM showed well aligned parallel arrays of fibres for the majority of samples collected, for which two examples are provided in Figure 2. The fibres appear fairly smooth and continuous with the exception of a few beads within the sample. Average fibre diameters for samples varied, increasing with nanotube loadings from $\sim 200\text{nm}$ with no nanotubes (Figure 2a) up to 360nm for 5% CNT loading (Figure 2b).

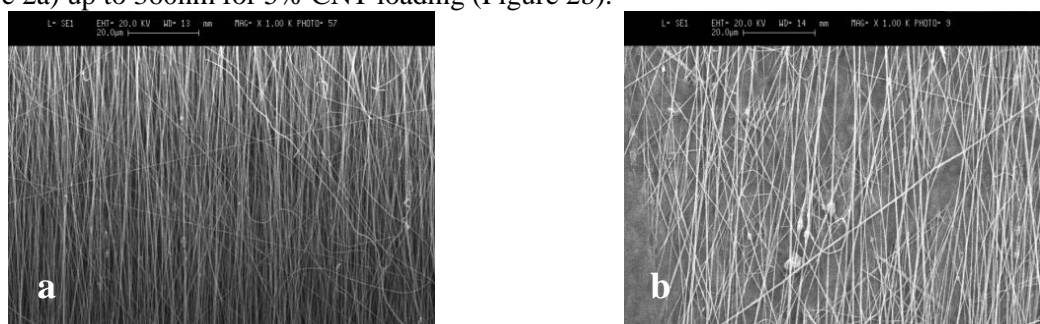


Figure 2: SEM micrographs of a) PEO without TX-100 and b) PEO +5% CNT with TX-100

Figure 3 shows the TEM micrographs for the nanotubes (figure 3a) along with the sample containing 5% wt loading of nanotubes (figure 3b). The mean CNT diameter was measured as 12 nm, slightly larger than the 6–9 nm quoted by Sigma with some diameters measuring near 20 nm. Figure 3b shows that when the nanotubes were straight there is alignment with the fibre, but where they bend there are defects, leading to kinks at the edge of the fibre, similar to observations made by Dror *et al* [4]. The bending can be reduced by the use of stiffer nanotubes such as those prepared through arc discharge, as opposed to the ones used here which appear to be prepared through chemical vapour deposition.



Figure 3: TEM micrographs of a) CNTs b) PEO electrospun fibres containing CNT's

The colouration of the fibre mat and the TEM micrographs show that high loadings of CNT's within nanofibers are possible. There is a visible number of tubes along the fibre axis (Figure 3b) which shows the potential for good connectivity of CNT's in the nanofibers leading to a higher level of conductivity. This was accomplished without need to modify the nanotubes prior to mixing to make them more dispersible in an aqueous media and high levels of loading were achieved, reducing dripping and promoting spinning by the addition of a surfactant.

4. Conclusions

We show that it is possible to electrospin polymer nanofibres containing CNTs from aqueous solution without additional nanotube treatment. The addition of a surfactant provides an effective route to even dispersions of CNT's reducing agglomeration and for allowing a more continuous electrospinning process. With the addition of the surfactant loadings of up to 5% CNT were obtained leading to a potential route for conducting nanofibers with high loadings of nanotubes.

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