Factorial Study of Nanofibrous Cellulose Acetate Web Production using the Electrospinning Technique

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Introduction

CA membranes and nanofibrous materials are of particular importance in filtration [1,2]. As part of a wider study dealing with the assembly and characterisation of nanofibrous cellulose acetate (CA) materials for liquid filtration, a systematic parameter study was completed for producing nano-scale CA fibres that were substantially free of bead defects. The effect of different solvent systems and process parameters during electrospinning CA were evaluated and interactions were explored. Of particular interest was the influence of different factors on mean fibre diameter and fibre diameter distribution produced using a binary solvent system.

Experimental

Materials

Cellulose acetate (acetyl content: 39 .8 % – molecular weight 30,000), Acetone and N,N-Dimethylacetamide (DMAC/ Aldrich 99%) were

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the polymer in a binary solvent system of acetone:DMAC (2:1) at room temperature.

Apparatus and procedures

Solutions were contained in a 5-ml glass syringe fitted with 20 gauge stainless steel nozzle (Aldrich) and connected to a dual head syringe pump operating at a feed rate of 0.04

to 0.02 ml/min depending on requirements. Electrospinning was performed inside a fume cupboard at a voltage of 20 to 25 KV and a tip-to-collector distance of 150mm. A flat aluminium collector was used throughout.

The resulting fibre morphologies in the webs were probed by scanning electron microscopy (Camscan series 4 environmental) and fibre dimensions and freedom from formation defects e.g. beads, were determined.

Mechanical evaluations were performed by tensile testing heat treated and untreated CA nanofibre mats (Instron) at a cross-head speed of 10 mm/min.

IR spectra (4000 to 600 cm^{-1}) were recorded using a Perkin-Elmer Spectrum BX spectrophotometer with diamond ATR attachment with a resolution of 4 cm^{-1}, interval scanning of 2 cm^{-1} and 64 scans per sample.

Results and Discussion

Factorial experiments were conducted to determine the interaction between process parameters in respect of mean fibre diameter, fibre diameter distribution and freedom from bead defects. It was found that the effect of polymer concentration on mean fibre diameter was dependent on the voltage and flow rate (Fig.1). Furthermore, there was a significant interaction between the concentration, voltage and flow rate.

Fig. 1 Interaction between levels of polymer concentration and voltage at fixed flow rate (0.006 ml/min).

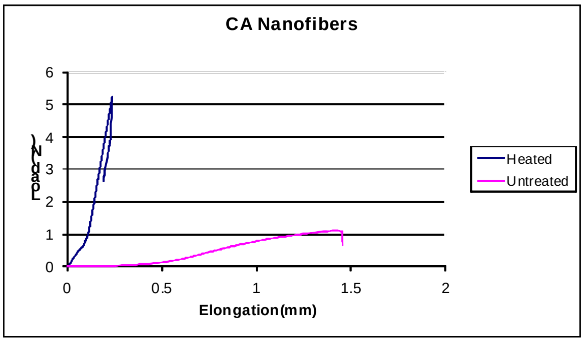


Fig. 2 Load-elongation curves for the untreated and heat treated CA nanofibre webs.

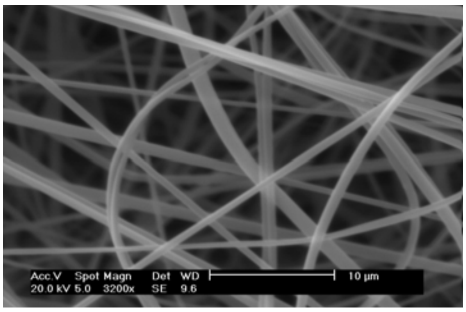
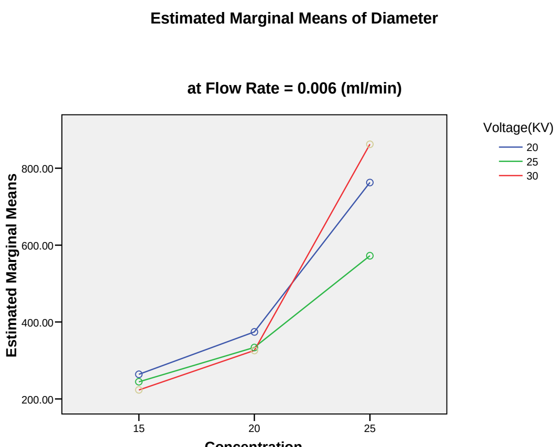


Fig. 3 SEM micrograph of CA web produced from Acetone:DMAC (2:1) solvent system

Unlike the heat treated CA materials, the untreated CA nanofibres exhibited a low tensile strength and high elongation at break (Fig.2). Both tensile modulus and



breaking strength were significantly higher following heat treatment due to chemical changes in the polymer confirmed by FT-IR. Increases in the web tensile strength were attributed in part to fusion at crossover points between nanofibres as evidenced by SEM analysis.

Conclusions

The results indicated a statistically significant interaction between polymer concentration, voltage and flow rate, such that the magnitude of changes in mean fibre diameter were dependent on the level of other process-related factors. Preferred electrospinning conditions were identified for CA that minimised the presence of structural defects in the web and promoted uniform fibre diameters. A method of CA nanofibre post-treatment was confirmed that significantly increased the tensile strength and modulus of CA nanofibrous webs.

References

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2. Zheng-ming Huang,S.Ramakrishna, A review on polymer nanofibers by electrospinning and their application in nanocomposites, Compose.Sci.Technol, 2003 ,Vol 63.2223-2253.