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Design of ultra-fine nonwovens via electrospinning of Nylon 6: Spinning parameters and filtration efficiency

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ABSTRACT

Electrospinning and its application in filtration area are worthwhile to look into as the large surface-tovolume ratio of nanoweb may affect the filtration efficiency by possibly giving more particle-capture sites. In this study, Nylon 6 is electrospun to produce ultra-fine nonwovens, and its characteristics as filter media are investigated. Electrospinning parameters including solution concentration, tip-to-collector distance, and the feed rate are changed in producing nonwovens in different fibre size distribution ranging from 50 to 150 nm in diameters. The solution concentration of 10 and 12 wt% produced the fibres with the average diameter of around 85 nm, where 15 wt% solution produced larger fibres with the average diameter of 121 nm. Finer fibres were able to process at longer tip-to-collector distance and at slower feed rate. The electrospun media that are processed at different spinning conditions are evaluated for its filtration efficiency and pressure drop. The electrospun nanofibre media shows the potential in application as HEPA and ULPA grade filter media, by comparing the filtering performance of nanoweb with the conventional melt-blown nonwoven media.

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1. Introduction

Electrospinning is widely accepted as a technique to fabricate submicron polymer fibres to engineer unique functional nanostructures. Electrospinning is fibre forming process, where a high voltage is used to create an electrically charged jet of polymer solution or melt from the needle. The polymer solidifies as it travels toward the collecting plate, often producing nanometer scale fibres. Nanofibre formation by electrospinning is affected by spinning parameters including solution properties and concentration, hydrostatic pressure in capillary tube, electric potential at the capillary tip, the tip-to-collector distance, and the chamber condition [1–3].

Electrospun nano-structured web has the potential to be applied in wide range of areas. The high surface area of the web can provide reactive sites for chemicals, making it a good candidate for chemical protective material. Nano to micro-pores of nanoweb provides good moisture and vapor transport properties, which can be applied in breathable sports wear fabrics. Small fibres with large volume of microscopic pores also provide many potential applications in insulating fabric, biomaterials, and high value-added textile fabrics [2–10]. Yet few articles have investigated the

feasibility of adopting the electrospinning technique as to producing filtration media [5].

Nylon nonwovens, for its toughness, resilience and easy processability, are extensively used in various applications including automobile parts, garment innerlinings, wipes, battery separators, synthetic suede and protective garments. If the nylon fibres are manufactured into nano to micro sized nonwovens, it would be able to further expand its application in specialty materials. Previous studies by Ryu et al. [7] and Fedorova et al. [11] reported the processes to structuring fine fibres using nylon.

Techniques most commonly used to produce ultra-fine fibre mats in general include melt-blown process, multi-component process, and electrospinning [4-8,11]. Those processes allow thermoplastic polymers to produce fibres in diameter of less than 500 nm. In the melt-blown process, polymer melts are pushed under relatively high pressure, through an array of nozzles, and fibres are formed under rapid cooling. Multi-component fibres consist of segments of different polymers, and are fabricated to extrude in desired configurations with multi-components. Melt-blown and multi-component processes have advantage over the electrospinning process in that they give relatively higher productivity. However, electrospinning is also considered as a promising process in terms of its flexibility in designing fibre structures by controlling the spinning parameters. In this study, nonwoven mat of submicron-sized nylon fibres was produced by electrospinning, and its applicability in filtration area will be explored. The electrospinning





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condition to form Nylon 6 nanofibres were experimentally determined by controlling parameters including solution concentration, tip-to-collector distance and feed rate. The filtration characteristics of the Nylon 6 nanoweb were evaluated in its pressure drop and aerosol collection efficiency, and the level of performance was discussed in reference to a couple of conventional melt-blown filter media.

2. Experimental

2.1. Material

High molecular weight Nylon 6 (63,000 g/mol) was purchased from BASF. Three polymer solution concentrations in 10 wt%, 12 wt% and 15 wt% were prepared by dissolving Nylon 6 in Formic acid (ACS reagent grade, Merck). The solution was stored at room temperature, and the experiments were carried out at room temperature as well.

2.2. Electrospinning

For electrospinning of Nylon 6, the polymer solution was taken in a 10 ml syringe to which a capillary tip of 0.4 mm inner diameter was attached. The positive electrode of the high voltage power supply is connected to the capillary tip. The grounded electrode was connected to a metallic collector wrapped with aluminum foil as shown in Fig. 1. Electric voltage was optimized at 15 kV. The polymer solutions in different concentrations were electrospun at three different feed rated 10, 15, and 50 μ l/min, and two different tip-to-collector distance of 15 and 20 cm. Table 1 shows the details of spinning parameters that have been adopted in this experiment.

2.3. Electrospun web characterization

2.3.1. Fibre diameter

The morphology of the electrospun Nylon 6 mat was observed by Scanning Electron Microscopy (SEM) using a JEOL JSM-6400 FE with Energy Dispersive X-Ray Spectroscopy (EDS) at 5 kV of accelerating voltage. The electrospun samples were coated (100 Å thickness) with Au/Pd using a K-550 X sputter coater before being mounted on SEM chamber. The fibres on the web were randomly selected to measure the individual fibre diameters by identifying two points at opposite ends of a fibre diameter.



Fig. 1. Schematic diagram of the electrospinning set-up.

Table 1

Experimental conditions to produce Nylon 6 nanofibres by electrospinning.

Parameters	Values
Polymer	High molecular weight Nylon 6
Solvent	Formic acid
Concentration (wt%)	10, 12, 15
Collector	Aluminum foil
Needle Length (in.)	4
Fibre diameter range (nm)	50-150
Electric voltage (kV)	15
Capillary diameter (gauge)	24
Tip to collector distance (cm)	15, 20
Feed rate (µl/min)	10, 15, 50
Relative humidity (%)	40
Temperature (°C)	25

2.3.2. Filtration characteristics

The main mechanisms in mechanical filtering actions are interception, inertial impaction and diffusion (Fig. 2). Interception is the filtering mechanism where a moving particle is blocked when it encounters a passageway or hole smaller than itself. Particles of relatively larger than pore sizes will have greater chance of being caught by the interception. Some particles do not follow the air stream but their inertia drives them to come into contact filter fibre and being captured. Small particles are more dependent of diffusion capture mechanism, where particles are captured through the random Brownian motion. The factors that determine the particle-capture mechanism and thereby influencing the filtration efficiency include particle size, weight and shape, air current velocity, filtering fibre size, and environmental conditions [12–14].

The pressure drop across the tested electrospun Nylon 6 mat was measured with a micro-manometer (FCO510, Furness Controls Ltd.) for various (1.0–4.0 m/min) face velocities. Face velocity is defined as the flow rate through a filter per unit filtration area. Electrospun mats were cut into circular sheets of 52 mm in diameter, and installed in a filter holder. The flow through the electrospun mats was derived by a vacuum pump, and controlled by an electronic mass flow controller.

Aerosol collection efficiency of the tested mats was calculated by measuring the aerosol concentration before and after passing through the mats. NaCl particles were used as a test aerosol. Polydisperse NaCl aerosol was generated by a collision atomizer (Model 3079, TSI Inc.) and dried by a diffusion dryer packed with silica gel. The dried particles entered an electrostatic classifier (Model 3080, TSI Inc.) extracting a known size fraction of sub-micrometer particles from the incoming polydisperse aerosol by using an electrical mobility detection technique. The electrostatic classifier produces monodisperse aerosol of known size. In this study, aerosols of 50, 70, 100, 150, 200, 300, and 400 nm in diameter were used. The classified particles were electrically neutralized by a Kr-85 aerosol neutralizer (Model 3077, TSI



A: Interception B: Inertial impaction C: Diffusion C: Aerosol Particles

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