Polyamide 6 Nanofibrous Nonwovens via Electrospinning

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ABSTRACT: Electrospinning of Polyamide 6 (PA 6) in 2,2,2-trifluoroethanol (TFE) was investigated for the fabrication of nanofibrous nonwoven membranes useful for separation systems. The effects of solution characteristics such as concentration and conductivity as well as the effects of processing conditions such as relative humidity and applied potential on the resultant nonwoven fibers were studied. By changing the relative humidity of the electrospinning chamber and the conductivity of the solvent, it is possible to modulate the fiber's size and consequently the porosity of the mats. The morphology of the electrospin. The mechanical properties of the nanofibers were also studied. The results showed that PA 6 nanofibers having a

diameter ranging from 100 to 600 nm, has been successfully prepared. The electrospun PA 6 nanofiber mats show good mechanical properties, such as a high-tensile strength (12 ± 0.2 MPa) and elongation ($300\% \pm 50\%$). The strength of the web was high enough to use as filter without the need of any supporting matrix and could be applicable in the field of self-supporting membranes. The X-ray and DSC analyses of the PA 6 electrospun fibers show the presence of the γ -form of PA 6 crystallite that is usually obtained in the condition where a high stress of the fibers is applied. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 117: 1754–1765, 2010

Key words: polyamides; orientation; membranes; electrospinning; nanofibers

INTRODUCTION

Electrospinning is a simple and cost-effective method of producing polymeric fibers with diameters ranging from nanometers to a few micrometers and it has attracted much attention in the past decade.¹

According to Reneker and coworker,² the principle of electrospinning involves formation of a charged jet of polymeric solution by the application of an electric field. As the jet travels in the air, the solvent evaporates and a charged fiber left behind which can be collected on a grounded plate (collector). Electrospinning yields mostly nonwoven fiber mats having large surface to volume ratios and various fiber morphologies and geometries.

Potential applications of electrospun fibers have also been identified in the various fields such as, high-performance air filters³ protective textiles,⁴ sensors,^{5,6} advanced composites,^{7,8} photovoltaic cells,⁹ wound dressing,¹⁰ as scaffolds in tissue engineering,^{11–13} and as membranes in separation processes.^{14,15}

Theoretical predictions indicate that significant increase of the filter efficiency for penetrating particle size (between 0.1 and 0.5 μ m) can be achieved by using the nanofibrous filtering media.^{16,17} It is possible to produce nanofibrous mats with peculiar characteristics, such as high porosity, small pore sizes with an interconnected structure, and a large surface area per unit volume^{18,19} by using electrospinning technique. Nanofiber web can be used to remove the nanoparticles along with other poly dispersed aerosol particles where high-performance air purification is needed such as in research labs, electronic component manufacturers, food, pharmaceutical, biotechnology companies, etc.

Development of nanofiber mats into more useful applications requires a throughout knowledge of the parameters that influence the process of electrospinning. For widespread commercial viability, many important questions need to be resolved including as follows: what parameters control the fabrication of fibers and can fibers of uniform diameter be consistently obtained? Numerous attempts have been made to decipher trends in electrospun jet operation as functions of fluid and operating parameters under experimental control. The main parameters which affect and/or control the process of electrospinning and subsequent fiber morphology can be as follows: solution concentration, polymer molecular weight, viscosity, conductivity, surface tension, applied voltage, distance of source electrode from the target substrate, electric field, and solution flow rate.20

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Figure 1 Schematic drawing of electrospinning system: (1) electrospinning sealed chamber; (2) temperature and humidity controller; (3) diffuser of air with checked humidity; (4) flux-meter; and (5) valves to control dry and humid air flux.

Moreover, most investigations were carried out in an unconfined system without controlling either the vapor concentration of solvent or the role of the environmental conditions.

No systematic study was conducted on the influence of both the evaporation and solidification of the jet and the role of the shape of the electrospinning chamber.

The objective of this work was to find out feasibility to prepare submicron polymer fibers by the electrospinning of polyamide 6 (PA 6) in 2,2,2-trifluoroethanol (TFE) by using a home-made electrospinning device. The effect of polymer concentration, electric potential, conductivity by the addition of lithium chloride and relative humidity were investigated to the diameter of fibers and the morphology.

EXPERIMENTAL

Materials

Polyamide 6 (molecular weight, $M_w = 30.000$; poly dispersity index, PDI = 1.75). The molecular weight and molecular weight distribution of each samples were verified using gel permeation chromatography. The solvent TFE was purchased from Sigma– Aldrich, which was used directly without further purification.

Preparation of polymer solutions

PA 6 in TFE with varying concentrations, Cp, ranging from 6.5 to 10 wt % were prepared at room temperature. Some solutions were prepared by using a solvent mixture of TFE, water, and LiCl with a weight ratio of 90 : 10 : 0; 90 : 9.5 : 0.5; and 90 : 9 : 1.

Electrospinning process

The polymer solution was placed in a 2-mL syringe having a needle with an internal diameter of 250 μ m, which was mounted on a syringe pump (Harvard Apparatus serial n° A-31896; USA). The syringe pump can provide a constant droplet of solution at the tip of the needle. An electric potential ranging from 5 to 30 kV was applied to the needle from a high-voltage power supply (Glassman High Voltage, 0–30 kV). An aluminum screen, placed at 15 cm from the tip of the needle was grounded and used as the counter electrode. The solution was subjected to electrospinning in a closed chamber and the relative humidity inside the chamber was controlled during electrospinning. Figure 1 shows the schematic and home-made set up for the electrospinning process. In Table I are reported the condition used for the all the electrospun solution.

Fiber characterization

SEM analysis

SEM characterization was performed by using a Jeol JSM-6380LV microscope, at an acceleration voltage of 15 kV at 15 mm working distance. Samples

TABLE I List of All Samples Prepared for This Research Work

Sample	Polymer concentration (wt %)	TFE : H ₂ O : LiCl (wt %)	Applied voltage (kV)	Relative humidity (%)
1	10	100:0:0	5	15
2	10	100:0:0	10	15
3	10	100:0:0	15	15
4	10	100:0:0	20	15
5	10	100:0:0	25	15
6	10	100:0:0	30	15
7	10	100:0:0	5	75
8	10	100:0:0	10	75
9	10	100:0:0	15	75
10	10	100:0:0	20	75
11	10	100:0:0	25	75
12	10	100:0:0	30	75
13	6.5	100:0:0	10	15
14	8	100:0:0	10	15
15	9	100:0:0	10	15
16	6.5	90:10:0	10	15
17	6.5	90:9.5:0.5	10	15
18	6.5	90:9:1	10	15



Figure 2 Electron micrographs and relative diameter distribution of PA 6 nanofibers electrospun from TFE solutions at Cp=10 wt %. The relative humidity on electrospinning chamber was 15%. (a) 5 kV; (b) 15 kV; (c) 20 kV; (d) 30 kV.

were mounted on an aluminum specimen stubs by means of double-sided adhesive tape and sputtercoated with a thin gold layer under rarefield Argon atmosphere, using a Polaron SC7620 Sputter Coater, with a current of 30 mA for 180 s. The diameters of the electrospun nanofibers were measured using ImageJ software directly from the SEM micrographs of fibers.



Figure 3 Electron micrographs and relative diameter distribution of PA 6 nanofibers electrospun from TFE solutions at Cp = 10 wt %. The relative humidity on electrospinning chamber was 75%. (a) 5 kV; (b) 15 kV; (c) 20 kV; and (d) 30 kV.

Thermal analysis

Differential scanning calorimetry (DSC) was performed by using a DSC 2920, TA Instruments, USA, calibrated by using an Indium standard. The calorimeter cell was flushed with N_2 at the rate of 80 mL/min. About 5 mg of the sample was accurately

600 400 200 0 0 5 10 15 20 25 30 35 Applied voltage (kV)

Figure 4 Influence of applied voltage and relative humidity on average diameter of fibers. Relative humidity: (1) 15% and (2) 75%.

weighed into Al crucible and which was used for the analysis. The temperature was programmed from -20 to 260° C, at a rate of 20° C/min. The PA 6 pellets, without any treatment, the cast films prepared by slowly evaporation of solvent from the corresponding PA 6/TFE solution and the electrospun fiber mats obtained from 8 wt % PA 6 solution were subjected to DSC analysis. All the samples were freeze-dried at room temperature before the DSC analysis to remove residual solvent and humidity.

X-ray data

The electrospun films were subjected to Wide-Angle X-ray Diffraction (WAXD) at 20°C using a Siemens D-500 diffractometer equipped with a Siemens FK 60-10 2000 W tube (Cu K α radiation, $\lambda = 0.154$ nm).

Tensile properties

Tensile properties of electrospun films were measured according to Huang's method.²¹ Rectangular shaped samples with a dimension of 8.5 \times 50 mm were prepared and subjected to the analysis. Mechanical properties of the fibers were measured using an Instron dynamometer mod. 5500, with a load cell of ± 5 kN at 25°C. A crosshead speed of 13 mm/min was used for all the specimens corresponding to a strain rate of 1.3 s^{-1} . Elastic modulus, stress at yield strength, deformation at fracture, and stress at fracture were calculated from the average of at least 10 measurements from stress-strain curves.

RESULTS AND DISCUSSION

Influence of relative humidity on fiber morphology

Aliphatic polyamide solutions in TFE are very susceptible to the process of electrospinning. The role of applied potential, solution concentration, and distance between electrodes were studied and reported in the literature.²¹

Preliminary results indicated that the electrospun fibers, obtained from PA 6 solutions in TFE, show some significant differences in the morphology even when the electrospinning is performed at constant solution concentration, flow rate, applied voltage, temperature, and tip-to-collector distance. Morphology of electrospun fibers was found to change day by day. The impossibility to control the morphology of the nanofibers is obviously a limit in the potential applications of this process.

Apart from above mentioned variable parameters, the relative humidity is a parameter that was not controlled and it might have a considerable influence on the morphology of electrospun fibers.

To control the relative humidity of the electrospinning atmosphere, a new set up containing a closed chamber as shown in Figure 1 was realized. Relative humidity of about 2 to 85% can be achieved by the application of dry and wet air into the closed chamber. By using this new set up, it was found to get reproducible results. PA 6/TFE solutions with varying concentrations ranging from 6 to 14 wt % were successfully electrospun at different relative humidity conditions.

Figures 2 and 3 show the scanning electron micrographs and fiber diameter distributions of nanofibers electrospun from 10 wt % solution of PA 6 in TFE at 15–75% of relative humidity and at varying applied potential ranging from 5 to 30 kV. In all cases the needle tip-to-collector distance was kept at 15 cm. The diameter size distribution of every sample of electrospun fibers was statistically calculated from, at least, 150 fibers present in the SEM images.

Figure 4 shows the influence of applied potential and relative humidity on the average diameter of electrospun fibers. It is obvious from the graph that the applied potential has very negligible influence on the fiber diameter, whereas the diameter was found to increase considerably by the decrease in the relative humidity. The average diameter of fibers was found to increase from 300 to 600 nm, when the relative humidity decreased from 75% to 15%. Moreover as shown in Figure 3, at high degrees of relative humidity, some beads were observed on fibers and it was found that the presence of beads were decreased by the decrease of relative humidity even at constant applied potential.

The bead formation can be correlated to several other parameters such as the instability of the jet of polymer solution, the solution viscosity, the net charge density carried by the electrospinning jet, and the surface tension of the solution.²² This behavior can also be related to the influence of the relative humidity on the TFE evaporating rate. The





Figure 5 Electron micrographs and relative diameter distribution of PA 6 nanofibers electrospun from TFE solutions at (a) 6.5%; (b) 8%; (c) 9%; and (d) 10%, at 15% relative humidity and 10 kV applied voltage.

evaporation rate and the solidification of the fluid jet strongly affect on the fiber diameter and morphology. If solvent evaporation decreases, due to higher aqueous vapor concentration in the electrospinning chamber, fiber diameter becomes smaller because the fluid jet can continue to elongate before the solidification. Consequently, the surface area increases and the charge density on the surface of the jet

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Figure 6 Polymer solution concentration effect on average diameter of fibers.

decreases. In this condition, a capillary instability were able to develop and thin fiber with many beads began to form.

Influence of solution concentration on fiber morphology

Polymer concentration has an important role in determining the spinnability of the solutions. Figure 5 shows the SEM images and the size distribution of PA 6 fibers electrospun by keeping constant relative humidity of 15% and at constant applied voltage of 10 kV by varying the solution concentrations. Homogeneous fibers were found to be produced in between a critical range of polymer concentrations from 6 to 10.5 wt %. Droplets were found when the solution concentration was lower than 6 wt %, whereas concentrations higher than 10 wt % microfibers and/or microribbons were observed. With in the above mentioned critical range, the average diameters of the fibers were found to increase with the increase in solution concentration. This effect can be obvious from Figure 6. At 6.5 wt %, the electrospun fibers were found to have an average diameter of 175 nm, whereas the average fiber diameter was found to increase to 616 nm, when the solution concentration was increased to 10 wt %. Moreover, it has been found that the fiber diameter distribution is narrower at lower solution concentrations.

At low concentrations of solutions, less than 6 wt %, the formation of polymer droplets were observed and this can be correlated to the insufficient chain entanglements.²³ On the other hand, with solutions at high concentrations, more than 10.5%, a change in the fiber morphology was observed. Microfibers together with ribbons were observed at higher concentrations. This behavior could be correlated to the increase in the viscosity with concentration and the difficulty to control the solution flow rate through

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the capillary. The viscosity of PA 6/TFE solution with concentration of 10 wt % was measured to be less than 0.3 Pa s, which is very low, and so in this case, viscosity may not be a parameter that affect the fiber morphology. For the PA 6/TFE solution, we observed a phase separation at concentration around 14 wt %, and since the solvent used was very volatile, a quick phase separation can also occur at the jet of polymer solution in the electrospinning process. This can create an instability responsible for the change in morphologies at higher concentrations.

Influence of conductivity (addition of LiCl) on fiber morphology

The solution conductivity is one of the main parameters in the electrospinning process. The polymer solution is being stretched due to the repulsion of the electrostatic charges present on its surface, and more charges can be carried at higher solution conductivity. The fiber jet of high-conductive solutions will be subjected to a higher tensile force in the presence of high electric fields. According to Baumgarten,²⁴ the radius of the fiber jet is inversely related to the cube root of the solution conductivity.

To increase the conductivity of PA 6/TFE system, some solutions were prepared by adding water or aqueous LiCl to the TFE. The concentration of the PA 6 was maintained constant, 6.5 wt %.

The conductivities of solvent and solutions are reported in Table II. The addition of LiCl, increases more than a thousand times the conductivity of the solution. Figure 7 shows the SEM images and the diameters distribution of nanofibers, electrospun from 6.5 wt % solution of PA 6/TFE containing varying concentrations of LiCl in water at 15% relative humidity, 10 kV, and needle tip-to-collector distance of 15 cm. It has been observed that the consignificant ductivity has influence on the morphology of the electrospun fibers.

From Figure 7 it is clear that the nanofibers resulted from polymer solutions in pure TFE or TFE-water = 90 : 10 water show many beads [Fig. 7(a,b)], whereas nanofibres obtained from polymer

TABLE II Conductivities of Solvent and Solutions with Different Amount of Lithium Chloride

Polymer	TFE : H_2O : LiCl	Conductivity	
concentration (wt %)	(wt %)	(µS/cm)	
0	100:0:0	5.7	
6.5	100:0:0	5.3	
6.5	90:10:0	6.8	
6.5	90:9.5:0.5	5621	
6.5	90:9:1	8219	



Figure 7 Scanning electron micrographs and relative diameter distribution of PA 6 nanofibers electrospun in air at 15% relative humidity, 10 kV applied voltage, and 15 cm needle tip-to-collector distance from solutions prepared using different mixtures of TFE, water, and LiCl. (a) 100 : 0 : 0; (b) 90 : 10 : 0; (c) 90-9.5-0.5; (d) 90-9-1. Micrographs (c') and (d') show samples (c) and (d) at higher magnification.

solutions containing TFE/water and LiCl at different concentration were without defects and with an average diameter of less than 100 nm [Fig. 7(c,d)]. By the increase in conductivity, bead-free and uniform thinner fibers were produced and this may be correlated to the fact that high-conductive solutions



Figure 8 DSC thermographs showing the effect of some parameters on the melting behavior of PA 6/TFE electrospun nanofibers mats: (a) polymer concentration; (b) applied voltage; (c) relative humidity; and (d) PA 6 pellets (line 1) compared with cast film (line 2) and electrospun nanofibers (line 3).

may undergo more stretching at higher applied potential.

Thermal analysis and X-ray diffraction of PA 6 nanofibers

To know the structure and the degree of crystallinity of the electrospun fibers, the samples were subjected to DSC and X-ray analysis. Fibers were electrospun with 6.5, 8, and 10 wt % solutions of PA 6/TFE at applied voltage of 10 and 30 kV and at 15 and 70% of relative humidities. DSC thermogram and X-ray plots of electrospun fibers were compared with that of a cast film obtained from 8 wt % PA 6/TFE solutions.

Figure 8 shows the role of polymer concentration, humidity, and electric voltage on the melting behavior of the polymer.

The melting enthalpy and melting temperature obtained from DSC are shown in Table III.

TABLE III Thermal Characteristics of PA 6 Electrospun Fibers Obtained by Using Various Conditions Compared with a PA 6 Cast Film and PA 6 Pellets

Concentration of electrospun solution (wt %)	Relative humidity (%)	Applied voltage (kV)	T_m (°C)	ΔH_m (J/g)	Cristallinity (%)
6.5	15	10	227	61	32
8	15	10	228	78	41
10	15	10	227	77	41
8	15	30	226	74	39
8	70	10	227	66	35
Cast film PA 6 Pellets PA 6			224 228	120 97	63 51



Figure 9 X-ray diffraction pattern of PA 6: (a) cast film from TFE solutions, (b) electrospun nanofibers mats. Deconvolution into amorphous (line 2), crystalline gamma-phase (line 3), and alpha phase (line 1) is also shown.

It can be observed that the electrospun fibers have a melting temperature that do depend neither from concentration nor applied voltage nor relative humidity. It has been observed that electrospun films show higher melting temperature and lower melting enthalpy than that of corresponding cast films. For example, the melting enthalpy of the electrospun nanofibers was found to be about 70 J/g for all the samples which was lower than that of the corresponding cast films (120 J/g). This indicates that the electrospun nanofibers have lower percentage of crystallinity about 35% compared with that of the corresponding pellets 51% and cast sample 63%.

This can be correlated to the rapid solidification process of the stretched macromolecular chains under the high-elongational rate in the electrospinning process. The rapid solidification can limit the development of crystallinity because the macromolecular chains had no time to form crystalline array. For the cast films, where the evaporation of the solvent is slow, the macromolecules have enough time to adjust their chains in a crystalline structure.

It has been observed that there are some differences in the thermograms of the electrospun PA 6 nanofibers and the corresponding PA 6 cast samples. In particular, the PA 6 electrospun fibers show a large endotherm transition at about 85°C and a shoulder in the melting peak at about 219°C.

TABLE IV Degree of Crystallinity and Amount Percentage of α-form and γ–form Crystallite Result from WAXD Pattern Deconvolution

Sample	Crystallinity	α-form	γ-form
	(%)	crystallite (%)	crystallite (%)
Cast film	61.5	95.6	4.4
Electrospun mats	38	67.1	32.9

It is well-known that PA 6 is a polymorphic material, having more than one crystalline structure. The most common structure is the α -form, where a fully extended planar zigzag conformation is present.²⁵ The characteristic melting temperature of the α -form reported in literature is about 223°C that correspond to the melting peak temperature of the PA 6 cast film as shown in the Figure 8.

The other crystalline structure of PA 6 is the γ -form, where a pleated sheet conformation was determined.^{26,27} The melting temperature of the γ -form is about 214°C and correspond to the shoulder of the electrospun PA 6 nanofibers. The endothermic peak at low temperature was observed for PA 6 samples and could be correlated to a $\gamma \rightarrow \alpha$ transition as already reported in literature.²⁸

PÅ 6 electrospun fibers were further investigated by wide-angle X-ray diffraction (WAXD). Figure 9 shows the WAXD patterns of cast PA 6 film (curve A) and electrospun PA 6 fibers (curve B). Deconvolution of the curves into its amorphous and crystalline contributions is also shown. Both the α -form, peak at $2\theta = 20^{\circ}$ and $2\theta = 24^{\circ}$, and γ -form, peak at $2\theta = 22^{\circ}$, are apparent, as well as an amorphous peak. The cast film shows a higher degree of crystallinity (61.5%), and essentially α -form crystallite. The electrospun PA 6 fibers exhibited a lower degree of crystallinity (38%) and the presence of 12.5% of the γ -form of PA crystallite. In Table IV are reported the degree of crystallinity determined as follows: Xcⁱ = Acⁱ/ \sum_i (Acⁱ + Am), where Acⁱ is the integrated area



Figure 10 Typical stress–strain curves of PA 6 nonwoven membranes obtained using different polymer concentration.

TABLE V				
Mechanical Characteristics of PA 6 Electrospun Fibers Obtained at Different Concentration Compared with a PA 6				
Microfibrous Commercial Air Filter				

Sample	Elastic modulus (MPa)	Stress at yield strength (MPa)	Deformation at fracture (%)	Stress at fracture (MPa)
Commercial air filter	38.4	1.9	45	3.4
Electrospun mats from solution at 6.5 wt %	38.3	2.1	165	3.1 10.8
Electrospun mats from solution at 10 wt %	39.5	2.7	310	11.8

undernest the crystalline peak of the crystalline *i*-form and Am is the integrated area of the amorphous peak.

These results confirm what is reported in literature where X-ray studies have shown that nanofibers obtained from electrospinning of PA 6-HFIP solutions contains some amount of γ -form crystallite.^{29–31} It has also been reported³² that the γ -form is obtained in the melt spinning when high take-up speeds are used, this is correlated to the high stress of the fibers. Therefore, during the fiber formation in the electrospinning process, the kinetical locking of the formation of γ -form is possible by the application of high stress on the polymer chains, due to high-evaporation rate of the solvent.

Mechanical properties

Figure 10 shows the results of the mechanical tests carried out on PA 6 electropun nonwoven mats as well as comparison on a commercial air nonwoven-mat microfibrous filter media. The initial part of the curve shows similar behavior for all the samples. The high resistance to deformation may be due to cohesive forces in the fiber assembly as a result of the large number of fiber-to-fiber contacts and that may be a consequence of the nanoscale fiber diameter. Increasing the strain, a pseudo-yield point is observed at the beginning, followed by a gradual reduction in the modulus caused by the initiation of fiber slippage. The modulus of the electrospun PA 6 nonwoven mats are determined by the mechanical tests and are shown in Table V. These values are found to be lower than that of PA 6 fibers because of fiber slippage caused by frictional forces between fibers during testing. These values are comparable or higher in respect to the values observed for the commercial air filter made of PA 6 microfibers.

CONCLUSIONS

In this work, PA 6 nanofibers-based filtering media, made up of fibers having diameter ranging from 100 to 600 nm, has been successfully prepared by using electrospinning technique. The fiber mats were

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found to be applicable in the field of self-supporting membranes with good mechanical properties. The effects of solution characteristics such as concentration and conductivity as well as the effects of processing conditions such as relative humidity and applied potential on the resultant nonwoven fibers of PA 6/TFE were studied. PA 6 fibers were investigated qualitatively by means of scanning electron microscope (SEM), thermal analysis (DSC), and mechanical characterization.

The electrospun PA 6 nanofiber web has shown good mechanical properties, such as high-tensile strength (12 ± 0.2 MPa) and elongation ($300\% \pm 50\%$) which are important features for a membrane for filtration to avoid damage during handling. The strength of the PA 6 nanofiber web was high enough to use as filter without the need of any supporting matrix.

By controlling the relative humidity inside the electrospinning chamber and by changing the conductivity of the solvent, it is possible to modulate the fiber size and consequently the porosity of the mats. Electrospun PA 6 membranes are found to have properties such as light weight and small pore size that make them appropriate for a wide range of applications in the area of filtration. PA 6 nanofiber membrane offers unique properties like high specific surface area and good interconnectivity between pores and it is possible to have enough surface modification to improve their spectrum and capture efficiency of filtration.

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