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Preparation and Characterization of Polymer Nano Fibres Produced from Electrospinning

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Abstract The ultrafine nylon fibers were prepared by electro spinning of nylon-6 solution in formic acid in the concentrations ranging from 12–28 wt%. The morphology of the electro spun nylon-6 fibers were investigated by scanning electron microscope (SEM) and the viscosities are measured with the help of redwood viscometer. The effects of electro spun process parameters such as the spinnable concentration of nylon 6 in formic acid, viscosity of polymer solutions, and influence of polymer solution concentration on fiber morphology were also studied. The results show that the spinnable concentration of nylon 6/formic acid solution is in the range of 16–28 wt%. For the lower concentration of nylon-6 solution of 16wt%, no continuous fibers but beads and some fibres segments could be obtained and higher solution concentration of 24wt% favours the formation of uniform fibers without beads.

Keywords: *electro spinning, material, nano fibres, optical, polymer*

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1. Introduction

With the emergence of nanotechnology, researchers become more interested in studying the unique properties of nano scale materials. Various polymers have been successfully electro spun into ultrafine fibers in recent years mostly in solvent solution and some in melt form. When the diameters of polymer fiber materials are shrunk from micrometers to sub microns or nanometres (Ex: $10x10^{-3}$ – $100x10^{-3}$ µm), there appear several amazing characteristics such as very large surface area to volume ratio (this ratio for a nano fiber can be as large as 10 times of that of a microfiber), flexibility in surface functionalities, and superior mechanical performance (Ex; stiffness and tensile strength) compared with any other known form of the materials [1]. A number of processing techniques such as drawing, template synthesis, phase separation, self-assembly, electro pinning etc, have be used to prepare polymer nano fibres in recent years [2]. Electro spinning has been recognized as an efficient technique for the fabrication of polymer nano fibers. Electro spinning technique was first introduced by Formals in 1934 [3]. It involves the use of high voltage to charge the polymer solution placed within a syringe. The polymer resulting composite becomes opaque or no transparent due to light scattering. This limitation, solution can form a droplet stabilized by its surface tension at the end of the needle tip of the syringe.

However, when the applied voltage exceeds a critical value at which the electrostatic force overcomes the surface tension, a stable jet of liquid could be ejected from the droplet. Due to bending instability, the jet is subsequently stretched by many times to form much smaller polymer fibers [4]. Many parameters can influence the transformation of polymer solutions into nano fibers through electro spinning. These parameters include (a) the solution properties such as viscosity, elasticity, conductivity, and surface tension, (b) governing variables such as hydrostatic pressure in the capillary tube, electric potential at the capillary tip, and the gap (distance between the tip and the collecting screen), and (c) ambient parameters such as solution temperature, humidity, and air velocity in the electro spinning chamber [5,6]. Zheng-Ming reviewed that, the most important quantities related with electro spinning are (a) the diameters of the fibers be consistent and controllable, (b) the fiber surface be defect-free or defect-controllable, and (c) continuous single nano fibers be collectable. However, researches so far have shown that there three targets are by no means easily achievable [7].

One of the most important applications of traditional (micro-size) fibers, especially engineering fibers such as carbon, glass, and Kevlar fibers, is to be used as reinforcements in composite developments [8]. With these reinforcements, the composite materials can provide superior structural properties such as high modulus and strength to weight ratios, which generally cannot be achieved by other engineered monolithic materials alone. Needless to say, nano fibers will also eventually find important applications in making nano composites. This is because nano fibers can have even better mechanical properties than micro fibers of the same materials, and hence the superior structural properties of nano composites

can be anticipated [9,10,11,12]. Moreover, nano fiber reinforced composites may possess some additional merits which cannot be shared by traditional (microfiber) composites. For instance, if there is a difference in refractive indices between fiber and matrix, the however, can be circumvented when the fiber diameters become significantly smaller than the wavelength of visible light. Polyamides (nylon) are often used to produce fibers with the diameter in the order of 30 μm by conventional melt spinning. However, the fibers with much smaller diameters are preferred for many industrial applications [13]. For example, the nylon fiber-reinforced composites will have good transparent property if the fiber diameter is lower than that of light wavelength. So the novel spinning methods with the advantages in producing smaller fiber should be developed to fulfil the industrial requirements, and electro spinning is proved to be a powerful method. So far, no report has been found in the open literature regarding measurement of the mechanical properties of single polymer nano fibers and also polymer nano fibers made from electro spinning have been much less used as composite reinforcements [14]. Only limited researchers have tried to make nano composites reinforced with electro spun polymer nano fibers. Up to date, the polymer composites reinforced with electro spun nano fibers have been developed mainly for providing some outstanding physical (example: optical and electrical) and chemical properties while keeping their appropriate mechanical performance [15]. Due to the small size of the electro spun polymer fibers, the membranes collected -from electro spun fibers possess a large surface area per unit mass and a very small pore size These characteristics make the electro spun fibers have many potential applications such as optical materials, sensor materials, nano composites materials, tissue scaffolds, wound dressings, drug delivery systems, in filtration and as protective clothing [16,17,18]. Solution viscosity plays an important role in determining the fiber size and morphology during spinning of polymeric fibers. The viscosities of polymer solution of different concentration are measured with the help of Redwood viscometer by keeping the temperature of solution at room temperature of 26℃ and a test temperature of about 50℃ [19]. The morphology of the electro spun nylon-6 fibers was observed with a scanning electron microscope. The diameters of the electro spun nylon-6 fibers were measured directly from the printed SEM micrographs of fibers [20].

The main goal of the study to prepared the ultrafine nylon fibers by electro spinning of nylon-6 solution in formic acid. The effects of electro spun process parameters such as the spinnable concentration of nylon 6 in formic acid, viscosity of polymer solutions, and influence of polymer solution concentration on fibres morphology were also studied.

2. Material and Methods

2.1. Materials

The polymer used for producing the nano fiber by electro spinning is Nylon-6. This polymer have low melting point, excellent mechanical property, wearing resistance, oil resistivity, heat resistance and can be used for the production of mono- and multi filaments, fibers, films and nets [21]. The nylon 6 used in this study, with an average molecular weight of about 23,000 g/mol and a particle size of 3mm was supplied by Sigma–Aldrich Co, Germany. Formic acid (HCOOH) was purchased from Merck specialties private limited, Mumbai. The chemicals used in this study were analytical grade and were used without further purification.

2.2. Preparation of Polymer Solution

Various polymer solutions with concentrations ranging from 16 to 28 wt% were prepared at room temperature by dissolving the nylon-6 in formic acid, and stirred by magnetic force to speed the dissolution. To remove insoluble fraction or impurities, the solutions were filtered with a nonwoven fabric. The prepared solution was then subjected to the electrospinning experiments [22].

2.3. Electrospinning Process

A high-voltage electric field for electro spinning process was produced by a changeable high voltage power supply (DC-high-voltage generator supplied by Best Mech Engineering, India) as shown in Figure 1. The polymer solution was placed into a 10ml glass syringe fitted with a needle and with an inner diameter of 0.6 mm. A clamp connected to a high-voltage power supply, which could supply a positive voltage from 0 to 50 kV. A piece of aluminium foil was placed at a distance of 12 cm from the needle tip.

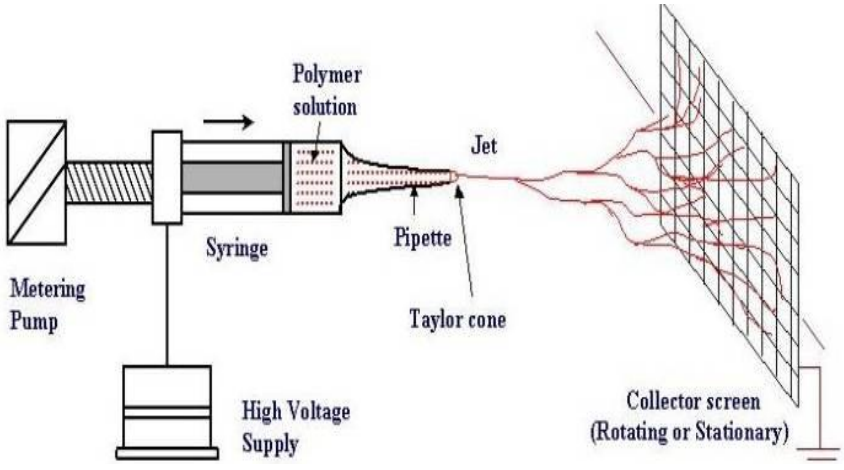

Figure 1. Line diagram of electro spinning assembly

The polymer solutions were electro spun with a fixed mass flow rate of 0.5 mL/h. This flow control unit was supplied by Cole parmer, Vernon hills, Illinois. The polymer solution formed a droplet at the tip of the syringe due to its weight and surface tension. The anode of the high-voltage power supply was connected to a needle, which was immersed in the polymer solution. By applied voltage between the anode and cathode, the droplet was instantly disintegrated into fibers that deposited on the aluminium foil at last. All electro spun samples were dried in a vacuum oven at room temperature for 48 h to remove the residual solvent [23].

3. Results and Discussion

3.1. Spinnable Concentration of Nylon 6 in Formic Acid

In this study, continuous nylon-6 fibers were successfully electro spun from the nylon-6/formic acid solutions in the concentration range of 16–28 wt%. For the lower concentration of nylon-6 solution of 16 wt%, no continuous fibers but beads and some fiber segments could be obtained. For the higher concentration of nylon-6 solution of 28 wt%, the electro spinning process became difficult. On the one hand, the nylon-6 sample did not dissolve easily in the formic acid to form such concentrated solution. On the other hand, higher viscous fluid balls could be gradually gathered outside the tip of the needle after the solvent volatilization. As a result, no matter how high an electric voltage had been applied, no fibers could be obtained on the collector ultimately. Hence, the spinnable concentration of nylon- 6/formic acid solution in electro spinning is 16– 28 wt%. Therefore, it was concluded that extensive chain entanglements were necessary to produce continuous fibers by electro spinning. The solution concentration of 24 wt % was chosen to fabricate nonwoven randomly arranged nylon 6 fibers with nano meter-scale diameters.

3.2. Viscosity of Polymer Solutions

Table 1. Viscosity of polymer solutions

S.No (centistrokes)	Nylon 6 wt%	Redwood seconds	Kinematic viscosity	Absolute viscosity (centipoises)
1	4	123	30.6	22.2
2	8	252	64.8	47.2
3	12	425	110.1	80.1
4	16	562	145.8	106.1
5	20	712	184.9	134.5
6	24	1225	318.4	231.5
7	28	2255	586.2	462.4

Hsiao et al. had reported that the solution concentration or the corresponding viscosity was one of the most effective variables to control the electro spun fiber morphology. We also investigated the effect of the nylon-6/formic acid solution properties on the electro spinning process. Table 1 gives the resultant redwood seconds measured from redwood viscometer by keeping the temperature of solution at room temperature of 26 ℃ and a test temperature of about 50 ℃. Figure 2 shows the relationship between the spinnable concentrations (from 16 to 28 wt %) and the corresponding viscosities of the nylon- 6/ formic acid solutions.

The viscosities of nylon-6 solutions increased sharply from 106 cP to 231 cP with the increase of the nylon-6 solution concentration from 16 to 24 wt%. The electro spun depositions were observed to distinguish the spinabilities of the following three classes: (1) the coexistence of beads and fibers, (2) homogeneous fibers (very few or no beads), and (3) the failure of the spinning jet.

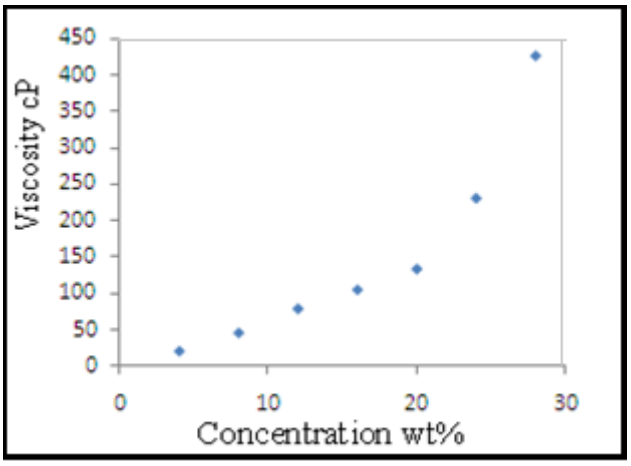

Figure 2. Solution viscosity as a function of nylon 6/ formic acid solution concentration

In Figure 2, three kinds of areas are indicated according to the resulting morphologies of the electro spun depositions: area A (5–100 cP), fibers and coexisting beads; area B (100–400 cP), deposition of a homogeneous fiber network; and area C (>400 cP), failure of the spinning jet because of a high viscosity.

3.3. Influence of Polymer Solution Concentration on Fiber Morphology

The SEM images and size distributions of the electrospun nylon-6 fibers with polymer solution concentrations of 16wt%, 20wt% and 24wt%, by keeping the voltage of 27 kV, needle tip-to-collector distance of 12 cm is shown in Figure 3. From the SEM images (Figure 3(a)) a lot of beads could be seen on the electrospun fibers prepared from the 16 wt% nylon-6 solution. As the concentration of the nylon-6 solution increased, the beads gradually disappeared and the shape of the beads gradually changed from spherical to spindle-like and only very little beads could be found on the fibers electrospun from the 20 wt% nylon- 6/formic acid solution (Figure 3(b)). Continuous and smooth fibers could be formed at 24 wt% as shown in (Figure 3(c)) when the other processing parameters like voltage, needle tip-to-collector distance as constant. The formation of beaded fibers had been observed widely. It was found that the formation of beaded fibers is related to the instability of the jet of polymer solution, the solution viscosity, net charge density(voltage) carried by the electrospinning jet and surface tension of the solution. Higher viscosity favoured the formation of fibers without beads. It can be seen that the nylon-6 fiber diameters became larger with the increase of the solution concentration. The 16 wt% nylon-6 solution resulted in the smallest fiber diameter, being

around 60 nm. The fibers electrospun from 20 wt% and 24 wt% solutions had an average diameter of about 90 nm and 110 nm, respectively. In addition, the fibers electrospun from 16 wt% nylon-6 solution had narrower distributions compared with those of the other concentration solutions. In short, the morphology and the average diameter of the electrospun nylon-6 fibers can be effectively controlled by polymer concentration.

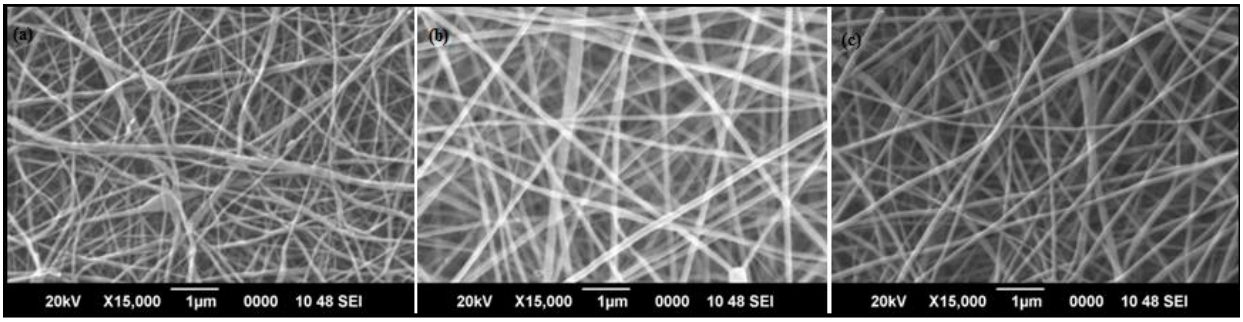

Figure 3. SEM images of electrospun nylon fibre (a) 16wt% concentration, (b) 20 wt% concentration and (c) 24wt% concentration.

In the spinnable concentration range, the higher the solution concentration, better the fibre morphology and the bigger the fiber diameter. Although, the size of the electrospun nylon- 6 fibers can be controlled by increasing the concentration of polymer solution, the size distribution of the fibers is really broad. There are at least three reasons for the broad size distribution: (1) during the electrospinning, the polymer droplet disintegration process induced by a high-voltage power supply may not be uniform, which results in the broad fiber size distribution; (2) the broad fiber size distribution may be associated with the formation of a large number of satellite droplets during the breakup of the solution jet; (3) many parameters can influence the formation of fibers such as solution concentration, surface tension, solvent used, voltage, temperature, and relative humidity. The soft fluctuations of these parameters during the experiments (probably inevitable) may broaden the fiber size distribution.

4. Conclusions

The nylon-6 nano fibers were successfully prepared by electros pinning. The spinnable concentration of the nylon 6/formic acid solution is in the range of 16–28 wt%. A lot of beads could be seen on the electro spun fibers prepared from the 16 wt% nylon-6 solution. Higher solution concentration favours the formation of uniform ultrafine fibers without beads. The viscosities of nylon-6 solutions increased sharply from 106 cP to 231 cP with the increase of the nylon-6 solution concentration from 16 to 28 wt%. As the concentration of the nylon-6 solution increased, the beads gradually disappeared and the shape of the beads gradually changed from spherical to spindle like shape. Continuous and smooth fibers could be formed at 24 wt% when the other processing parameters were constant.

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