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Effect of Needle Diameter on the Morphological Structure of Electrospun n-Bi_2O_3/Epoxy-PVA Nanofiber Mats

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***Abstract*—**The effect of needle diameter on the morphological structure of electrospun n-Bi_2O_3/epoxy-PVA nanofibers has been investigated using three different types of needle diameters. The results were observed and investigated using two techniques of scanning electron microscope (SEM). The first technique is backscattered SEM while the second is secondary electron SEM. The results demonstrate that there is a correlation between the needle diameter and the morphology of electrospun nanofibers. As the internal needle diameter decreases, the average nanofiber diameter decreases and the fibers get thinner and smoother without agglomeration or beads formation. Moreover, with small needle diameter the nanofibrous porosity get larger compared with large needle diameter.

***Keywords*—**Needle diameter, fiber diameter, agglomeration, porosity, SEM.

I. Introduction

RECENTLY, electrospinning; an advanced technique of fiber fabrication, has gathered significant interest among the researchers as it can fabricate different types of fibers ranging from microscale (10 μm) to nanoscale (<1000 nm) in diameter that has high surface area to volume ratio compared with other methods of fiber formation such as template synthesis, self-assembly, phase separation and drawing [1]. The fabricated fibers have been applied in many different fields such as; tissue engineering, drug delivery system, wound dressing, textile industry, cancer treatment as well as for fabrication new radiation shielding material [2], [3]. Electrospinning process utilizes a high voltage power supply imposed on a drop of polymer solution held by its surface tension at the end of needle tip. The electrospinning process initiation causes disturbance in the surface of polymer which results in formation a polymer droplet with spherical shape called as Taylor zone. Once the electrostatic repulsion prevails the surface tension, a jet of polymer solution ejected from the zone towards the target. As the jet travels through the air, the solvent evaporates leaving behind an ultrafine fiber collected on an electrically charged target [4]. Where the electrospinning is a straight forward process for fiber formation, it is still controlled by different parameters that can be adjusted to form different types of fiber morphology. These parameters are classified into three main categories which are solution parameters, instrumental parameters and ambient parameters [5]. The polymer concentration, conductivity, molecular weight and degree of polymer hydrolysis (DH) are the most reported solution parameters by researchers as it can affect the initiation and progress of electrospinning process [6] whereas, potential difference, flow rate of polymer solution, collection distance and needle diameter are the prominent apparatus parameters that must be carefully selected as it can influence on the morphology of electrospun nanofibers. Zhang et al. reported that the diameter of PVA nanofiber was increased from 553 nm to 1071 nm as the concentration of PVA solution increases from 15 wt. % to 25 wt. % [7]. Thus, their work concluded that the polymer concentration affected the electrospun nanofiber diameters. In their other work, they reported a similar result which proofing that increasing the sodium chloride (NaCl) concentration from 0.05% to 0.2% increase the average nanofiber diameter from 214 nm to 519 nm [7]. Meanwhile, another study done by Hekmati et al. reported that only small solution droplets were formed with no fibers formation occurred for the 5 wt. % of polyamide-6 (PA-6)/formic acid polymer solution. As the solution was increased to 15 wt.%, uniform, homogenous and thinner fibers were fabricated [8]. The molecular weight effect on the morphology of fibrous structure was studied by Tao et al. where they reveal that as the molecular weight of PVA increased, the fibrous structure changed from beads, beaded fibers, free beaded fibers to flat fibers [9]. The same results were observed by Mohammadian et al. where they found that with high molecular weight of poly L lactic acid (PLLA), the minimum concentration required to form uniform fibers without beads is 3.5%, while with low molecular weight, beaded fibers were observed with 9% polymer concentration [10]. Chowdhury et al. studied the effect of potential difference and solution flow rate on the morphology of electrospun nanofibers. They reported that the average nanofiber diameter decreases from 1353 nm to 1211 nm as the applied voltage increase from 12 kV to 18 kV, respectively. Regarding the effect of flow rate, they found that feeding the polymer solution with low rate (0.2 ml/hr.) fabricate uniform fiber with cylinder shape as well as thinner diameter compared with high feeding rate (0.3 ml/hr.) [11].

Few studies contributed to study the effect of needle diameter on the average electrospun nanofiber diameter. For instance, Macossay el al. reported that there is no correlation

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between the needle diameter and electrospun poly methyl methacrylate (PMMA) nanofibers [12]. Theoretically using small needle diameter results in formation of small drop of polymer solution with high surface tension compared with large needle diameter, so as a result high potential difference is required to overcome the surface tension and form a polymer jet. The higher potential difference increases the electrostatic force imposed on the polymer drop which is responsible about stretching the jet and makes it thinner, so the jet will get more thinner and small nanofiber diameter will br collected on the ground plate [13]. Therefore, this study investigates the effect of three different types of needle diameters on the morphological structure of electrospun n-Bi_2O_3/epoxy-PVA nanofibers using SEM.

II. Experimental Details

A. Materials and Sample Preparation

Polyvinyl alcohol, PVA (molecular weight: 80,000–98,000; hydrolysis rate: 99 %; density: 1.3 g/cm^3) was purchased from Sigma-Aldrich. FR 251 epoxy resin (Bisphenol-A diglycidyl ether polymer) and FR 251 hardener (Isophoronediamine) were purchased from Buehler company. Ethanol and N, N-dimethylformamide (DMF) were used as solvent. Bismuth(III) oxide (Bi_2O_3) (density: 8.9 g/cm^3, particle size: 10-100 nm) was purchased from Sigma-Aldrich and it was used in this study for X-ray attenuation for radiation shielding purpose.

PVA powder was dissolved in distilled water at 90 °C and stirred for 2 h to ensure that powders dispersed uniformly. Bi_2O_3 was dissolved in ethanol with hardener by stirring for 10 minutes. Epoxy resin was dissolved by ethanol. The ratio of epoxy resin to hardener was 4:1. The prepared PVA solution was mixed with hardener and Bi_2O_3 solution. After this step, the solution was sonicated with epoxy-ethanol solution for 30 minutes at 60 °C to prevent the nanoparticle agglomeration. The final step is mixing these two solutions together at 80 °C for 15 minutes to get homogenized solution.

B. Electrospinning Process

The prepared composite polymer solution was poured into plastic syringe with different internal diameter (ID) of needle which are 0.84 mm, 0.26 mm and 0.21 mm. The flow rate was 1 ml/h. and the distance between the needle tip and the collector was 27 cm. The solution was electrospinning at 12 kV and collected on ground plate for 5 hours.

C. SEM

Two types of SEM techniques were used. The first one is backscattered SEM, while the second is secondary electron SEM. Backscattered SEM was used to detect the Bi_2O_3 nanoparticles within the nanofibers for radiation shielding purpose and it was done just for the first sample with 0.84 mm ID. SEM investigates the nanofiber diameter using Quanta FEG 650. The samples were coated with gold in two 60 s consecutive cycles at 20 mA with Quorum Q 150 TS sputter to prevent the electrical charges.

III. Results and Discussion

SEM micrographs of Bi_2O_3/epoxy-PVA nanofibers obtained with three different types of needle diameters are presented in Figs. 1 (a)-(c). Fig. 1 (a) presents the backscattered SEM for nanofibers fabricated with 0.84 mm ID, while Figs. 1 (b) and (c) present the secondary electron SEM for nanofibers fabricated with needles with 0.26 mm and 0.21 mm ID, respectively.

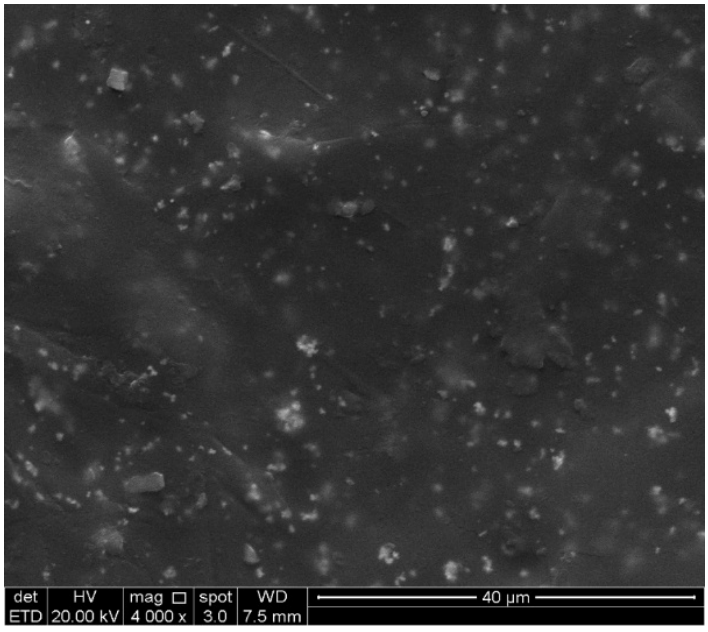

Fig. 1 (a) SEM of Bi_2O_3/epoxy-PVA nanofibers, using 0.84 mm ID of needle

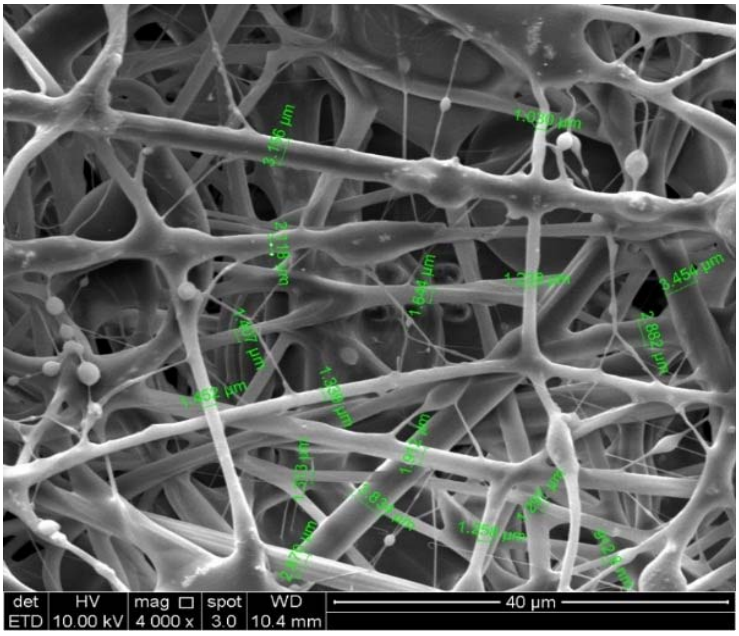

Fig. 1 (b) SEM of Bi_2O_3/epoxy-PVA nanofibers, using 0.26 mm ID of needle

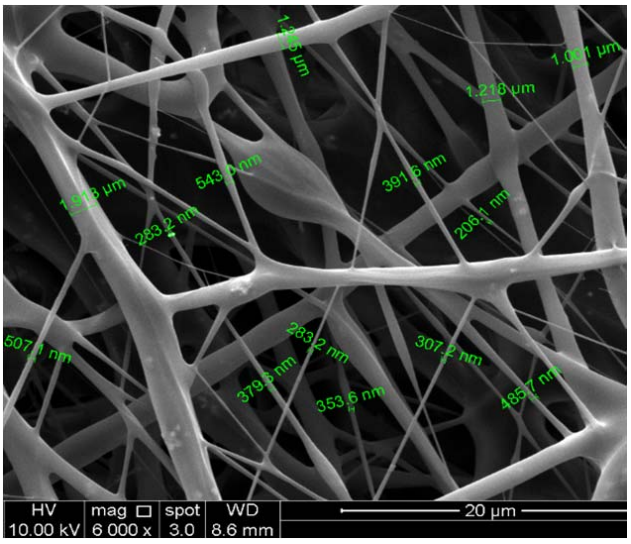

Fig. 1 (c) SEM of Bi_2O_3/epoxy-PVA nanofibers, using 0.21 mm ID of needle

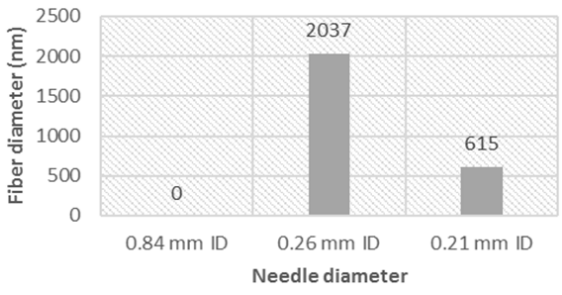

Fig. 2 Needle diameter and average nanofiber diameter obtained

As shown in Figs. 1 (a)-(c) there is a significant difference in the morphology of electrospun n-Bi_2O_3 /epoxy-PVA nanofibers between the different types of needle diameters. There was no electrospun nanofibers were fabricated with 0.84 mm ID as shown in Fig. 1 (a), while there is an electrospun nanofiber with 0.26 mm and 0.21 mm ID, but with different morphology. As shown in Fig. 1 (b), the fabricated fibers are spindle, beaded and branched in shape with a lot of agglomeration, while in Fig. 1 (c), the electrospun nanofibers are thinner, smoother and bead free without agglomeration as well as higher fiber porosity was observed with small needle diameter compared with electrospun Bi_2O_3/epoxy-PVA nanofibers with large needle diameter. In addition to, the average diameter for 10-15 individual fibers was measured.

With 0.26 mm ID, the average diameter was 2037 nm ± 402, while with 0.21 mm ID, the average was 615 nm ± 22 as shown in Fig. 2.

So, with small needle diameter, the fabricated nanofibers were thinner, smoother and bead free without agglomeration as well as high fiber porosity was observed compared with large needle diameter.

These results have explained as follow, that with small needle diameter, small drop of polymer solution will form at the end of needle tip with high surface tension compared with large needle diameter. Polymer with high surface tension require to increase the potential difference to overcome the high surface tension. Using high potential difference increase a force called electrostatic force which is responsible about stretching the polymer jet and divide it into many smaller jets, so thinner and smoother nanofibers will be collected. These results contradict with Macossay et al. (2007) [12] results, where they reported that there is no correlation between needle diameter and electrospun PMMA nanofibers.

IV. Conclusion

Small needle diameters with 0.21 mm ID fabricated thinner and smoother nanofibers without agglomeration or bead formation compared with large needle diameter 0.26 mm ID. The porosity of nanofibers gets increase with decrease the needle diameter from 0.26 to 0.21 mm ID, in addition to the smaller fiber diameter results from smaller needle diameter. These results contributed to electrostatic force which responsible about stretching and thinning the polymer jet.

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