Bismuth Trioxide Doped Polyamide 6.6 Nanocomposites for Electrically Insulating Materials

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Research Article

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Abstract

Polymer-based insulators have attracted much attention amongst researchers due to their various advantages such as light weightness, lower cost and ease of production. In this study, it is aimed to manufacture lightweight and thin insulator by electrospun composite fibers and to observe the effect of bismuth oxide (Bi$_2$O$_3$) on insulating property of polymer-based composite structure. For that purpose, polyamide 6.6 polymer (PA6.6) with high dielectric constant was doped with bismuth trioxide micro particles and it was used for coating polyester spunbonds by electrospinning technique. Morphological properties, thermal behaviours and electrical resistance of coated spunbonds were investigated. Scanning electron microscopy (SEM) and electron dispersive X-ray spectroscopy (EDX) showed that Bi$_2$O$_3$ was successfully adapted into fiber structure and nano-scale PA6.6/Bi$_2$O$_3$ composite fibers were obtained. Thermal behaviours of coated samples were developed by increasing Bi$_2$O$_3$ loading according to differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) results. Addition of Bi$_2$O$_3$ caused remarkable increase on electrical resistance of PA6.6 electrospun surfaces. Bi$_2$O$_3$/PA6.6 nanocomposite fibers are promising and good candidates for thin electrically insulating polymer-based structures for micro- or nanodimensional devices.

1. Introduction

Usage of polymers and polymer based materials has been tremendously increased in specific fields for last many decades. Especially for electrical applications, polymer insulators have been increasingly used in both distribution and transmission of various voltage ranges by considering their medium or high dielectric constants. Polymers having high and low dielectric constants have satisfied demands for manufacturing of capacitors and developed dielectric materials, respectively. Besides their wide ranges of dielectric constants, polymeric materials offer many attractive advantages such as; high elastic surface, low surface energy, good thermal stability, good hydrophobic characteristics and compatibility with organic and inorganic fillers for electrical applications [1].

Bismuth oxide (Bi$_2$O$_3$) is a semi-conductive metal oxide [2]. It is generally used in various fields such as gas sensors, microwave entegrated circuits, optical devices and photovoltaic applications. Due to the high conductivity of oxygen ions, it has been taken in consideration for solid electrolyte applications [3]. In manufacturing of functional materials with Bi$_2$O$_3$, electro-deposition, synthesis with microwave method and chemical vapor deposition method are generally used and various composite structures or semi-conductive nanofibrillates can be successfully produced in form of nanoparticles, nanofilms or nanofibers [4–6]. Some studies are available in production of Bi$_2$O$_3$-based nanocomposites by electrospinning technique. Demir et. al. (2019) claimed that CNF- Bi$_2$O$_3$ nanocomposite material behaved as anode material exhibiting excellent rate capability. Mills et al. (2013) studied on developing detection sensitivity of polymeric ions by doping them with metallic nanoparticles. Li et. al. (2016) resulted that electrospun Bi$_2$O$_3$-decorated carbon nanofibers were next generation super capacitors [7–9].
Electrospinning is an alternative method to produce functional composite structures. In this technique, solvent is an actor from which is utilized to electrospin polymer or its blends under electrostatic field and deposition of randomly aligned micro or nanofibers is observed on collector [10]. The mostly known advantage of this manufacturing method is the ability of processing with different types of polymers [11–23]. Many researchers focused on electrospun nanostructure manufacturing due to unique and/or superior characteristics of nanostructures by means of electronic, magnetic, optical, mechanical, physical and chemical properties [24–30]. For changing electrical behaviours of materials, many methods have been studied such as; blending polymers with a conductive polymer or material, addition of co-solvent into polymer solution and using different solvents for dissolving polymer [31–35]. Electrical characteristics of electrospun structures have been studied by few researchers but most of these studies focus on improving electrical conductivity of end product [36–40]. On the other hand, a few current studies are available for producing electrically insulating material with different types of polymers and fillers. Song et al. (2018) studied on manufacturing of multilayer nanofibrillated cellulose hybrid films filled with various amounts of graphene and boron nitride and they reported that composite structure exhibited comprehensive performance with electrically insulating properties [41]. Guo et al (2019) produced electrically insulating nanofibrillated cellulose films with Al₂O₃ and graphene fillers and they claimed that these films were super insulators in next generation electrical packing field [42]. Zhang et al. (2020) fabricated electrically non-conductive polymer composites by compression molding and they measured electrical resistance of sample as 6.61 × 10¹³ Ω cm [43].

In the present study, it is investigated whether electrospinning technique can be used for production of a thin composite insulator. For that purpose, PA 6.6. polymer having high dielectric constants was doped with Bi₂O₃ micro particules and nanocomposite fibers were deposited on PES spunbond. The objective of current study was to produce light weight polymer-based insulator using electrospinning technique with preparation of well dispersed Bi₂O₃ in polymer fiber mats and examine the effect of Bi₂O₃ doping on electrical conductivity, morphological properties and thermal characteristics of PA 6.6. electrospun surfaces.

2. Experimental Details

2.1. Materials

Bismuth (III) oxide (Bi₂O₃) (purity > 99 %) with particle size of 1.246 μm was purchased from Across Organics. Polyamide 6.6 (PA6.6, Mₚ=262.35 g/mol, 3.4 @1MHz) polymer and PES spunbond (50 g/m²) was supplied from Sigma Aldrich and Mogul Nonwovens, respectively. Formic acid (FA) and dichloromethane (DCM) were obtained from Merck Chemicals. All reagents were used as received without further purification.

2.2. Method
2.2.1. Solution preparation: PA6.6 pellets were dissolved in FA/DCM (3:1) binary solution [11,44]. By keeping total concentration at 10 % in w, Bi$_2$O$_3$ micro-particules were added at 0-10 wt% to PA 6.6. polymer solutions, homogenized under ultrasonification for 6 h at 35 °C and then stirred vigorously for 8 h at room temperature.

2.2.2. Electrospinning conditions: Polymer solutions were transferred to a 10 ml syringe with 11.99 mm inside diameter. The applied voltage and flow rate was ~ 37-38 kV and 1 ml/h, respectively. Distance between needle tip and target was 8 cm. The resulting randomly-oriented Bi$_2$O$_3$/PA 6.6. fibers were deposited on flat collector covered with PES spunbond for 45 minutes.

2.2.3. Characterization: The average fiber diameter of as-electrospun Bi$_2$O$_3$/PA 6.6 fibers and morphology of Bi$_2$O$_3$ powder and Bi$_2$O$_3$/PA 6.6 composite fibers were determined by Zeiss EVO/LS10 scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDX). On behalf of possible bond occurrence, the samples were examined by Agilent Technologies Cary 630 fourier transform infrared spectroscopy (FT-IR). Thermal decomposition behaviours of electrospun fibers were characterized by increasing heat flow from 30 °C up to 300 °C at heating rate of 20 °C/min via Shimadzu DSC-60 differential scanning calorimetry. Mass loss of samples were determined within a temperature range of 30 °C up to 800 °C at a heating rate of 10 °C/min via Shimadzu TGA-50 thermogravimetric analyse device. Thickness of Bi$_2$O$_3$/PA 6.6 composite fibers coated PES spunbonds were measured by LYK 5318 digital fabric thickness measurement device and average thickness values of samples were calculated after three readings for each sample. Finally, electrical resistance of coated samples were measured at room temperature by using temperature-controlled four point probe via Entek Electronics FPP electrical resistance and conductivity measurement device. Electrical conductivity values were obtained after 50 readings for each sample by entering sample thickness.

3. Results And Discussion

3.1. Scanning Electron Microscopy (SEM) & Electron Dispersive X-Ray Spectroscopy (EDX)

Table 1 shows presence of bismuth element in PA6.6-based composite fibers doped with different amount of Bi$_2$O$_3$. Results showed that amount of Bi$_2$O$_3$ in electrospun PA 6.6 fibers increased as increasing addition of powder in polymer solutions. Besides, Bi$_2$O$_3$ powder was adapted into nanofiber structures by considering SEM analysis of Bi$_2$O$_3$/PA 6.6 nanocomposite fibers.

Table 1. EDX analysis of PA 6.6/Bi$_2$O$_3$ composite nanofibers
| Nanofibers     | PA6.6-0 | PA6.6-5 | PA6.6-10 |
|---------------|---------|---------|----------|
| Element       | w %. (average) |         |          |
| Carbon (C)    | 73.38   | 62.36   | 54.15    |
| Nitrogen (N)  | 14.29   | 17.69   | 15.79    |
| Oxygene (O)   | 12.32   | 14.16   | 14.61    |
| Bismuth (Bi)  | -       | 5.79    | 15.44    |

SEM micrographs of Bi$_2$O$_3$ powder showed that Bi$_2$O$_3$ was layerless material having irregular spherical shapes with uncertain surface uniformity. Particle size of Bi$_2$O$_3$ powder changed between 650.4 nm and 1.724 μm and average particle size was found as 1.246 nm. According to SEM images of PA6.6-based samples, nanocomposite fibers were manufactured with/without microparticular filler. Average fiber diameter of neat PA6.6 nanofibers (PA6.6-0) was 68.25 ± 20 nm. Bi$_2$O$_3$/PA 6.6 nanocomposite fibers with average diameter of 81.5 ± 10 nm and 84.10 ± 12 nm were obtained for PA6.6-5 and PA6.6-10, respectively. There was no linear relation between Bi$_2$O$_3$ content and average fiber diameter for Bi$_2$O$_3$/PA 6.6 nanocomposite fibers. Due to continuous and partially beaded nanofiber manufacturing, it was concluded that FA/DCM binary solvent mixture did not satisfactorily contribute to homogeneity of PA6.6 polymer solution containing Bi$_2$O$_3$ but studying on suitable concentration range with these solvents supported nanofiber occurrence. [45, 46] Figure 1 shows the SEM images of Bi$_2$O$_3$ powder and Bi$_2$O$_3$/PA6.6. nanocomposite fibers.

### 3.2. Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR spectra of samples were examined by considering EDX test results to detect possible bond occurrence in nanocomposite fibers. In Figure 2, bands at 3296.655 cm$^{-1}$, 2919.519 cm$^{-1}$ and 1634.330 cm$^{-1}$ wavelengths represented stretching vibrations of N-H, CH$_2$ and C=O bonds, respectively. Besides, N-H bending vibration was shown at wavelength of 1634.330 cm$^{-1}$. Characteristic peaks of PA6.6 nanofibers were consistent with previous studies [47]. Peak intensity of Bi$_2$O$_3$/PA6.6 nanocomposite fibers increased without any shift in characteristic band ranges and these increases were results of increase in Bi$_2$O$_3$ concentration.

### 3.3. Differential Scanning Calorimetry (DSC) & Thermogravimetric Analysis (TGA)
Bi₂O₃/polymeric electrospun fibers had similar pattern with little shifts by increasing Bi₂O₃ loading. In Figure 3, DSC results showed that endothermic peaks were observed in PA6.6. Tg values of Bi₂O₃-free PA6.6 sample was consistent with mats electrospun with polymer concentration of 10% (w) in previous studies and it was reported as 56.44 °C [48-50]. Bi₂O₃ loaded nanofibers showed higher Tg values compared to their unloaded samples. DSC thermograms of Bi₂O₃-free electrospun fiber were relevant with literature but these peaks were shifted and observed at higher temperature values for electrospun fibers containing Bi₂O₃ micro particules. Electrospinning was performed at room temperature and further process was not applied to surfaces, such as drying in a oven for awhile. Therefore, no crystallization would occur during electrospinning. Besides, randomly aligned electrospun fibers were manufactured and perfect alignment of nanofibers were not observed which was a factor triggering crystallization [50]. Tm and Tg values of samples are given in Table 2.

### Table 2. Main thermal transition temperatures of samples

|       | PA6.6  |      |      |
|-------|--------|------|------|
|       | 0 %    | 5 %  | 10 % |
| Tm    | 271.44 | 273.38 | 273.79 |
| Tg    | 56.44  | 57.37 | 58.22 |
| ΔH    | 29.60  | 36.78 | 67.57 |

TGA thermograms and percentage weight loss of samples are given in Figure 4. TGA analysis was used to determine the thermal stability of neat PA6.6 and Bi₂O₃/PA6.6 nanocomposites with different percentages. For neat PA6.6 (PA6.6-0), small weight loss was observed above 200 °C due to evolution of traces of moisture or unreacted monomers and decomposition was available above 350 °C due to decomposition of base polymer to leave residual carbon from polymer back bone. [51]. For Bi₂O₃/PA6.6 nanocomposites, onset decomposition temperature increased with corresponding to neat PA6.6 by 5% and 10 % Bi₂O₃ loading. Weight loss of loaded samples were seen above 450 °C. This case explained that Bi₂O₃ loading developed thermal behaviours of samples.

## 3.4. Thickness of samples:

The thickness of PES spunbond was measured as 0.192 mm with digital fabric thickness device. The fabric thicknesses of PA6.6-0 and PA6.6-10 coated PES spunbond was measured as 0.258 mm and 0.300 mm, respectively. By means of fiber diameter, fineness of PA6.6-based nanofibers changed between 68.25
nm and 84.10 nm. There was consistent relationship between Bi$_2$O$_3$ loading and fabric thickness as well as fiber diameter. Bi$_2$O$_3$/polymer coating increased fabric thickness. Thickness values of coated PES spunbonds are illustrated in Figure 5.

### 3.5. Electrical analysis by dielectric spectroscopy:

Electrical insulating property of a solid material can be characterized by considering dielectric constant of corresponding material. Dielectric constants ($\varepsilon$) of PA 6.6 polymer was reported as 3.4-3.6 at 1 MHz and PA 6.6 was considered as an insulating polymer amongst other commercial polymers [52-54]. Electrical resistance of Bi$_2$O$_3$/PA6.6 nanocomposite fiber coated PES spunbonds are shown in Figure 6. 5% Bi$_2$O$_3$ loading increased electrical resistance of PA6.6-0 from 592.287 k$\Omega$.cm to 949.398 k$\Omega$.cm. Coated PES spunbonds showed more insulating property than uncoated PES spunbond. This can be correlated with tortuosity effect of nanofibers collected on PES spunbond and slow discharge of electrical energy in nanofiber layers [55]. On the other hand, there is reciprocal proportion between electrical resistance and conductivity. Electrical conductivity of uncoated PES spunbond was obtained as 5,864E-06 S/cm. Bi$_2$O$_3$ loading lead to more insulating material production. This case could be explained that decrease in electrical conductivity was related with polymer type and nanofiber production utilized both Bi$_2$O$_3$ propagation and decisive characteristics’ exhibition in composite structures [52-55].

### 4. Conclusion

In summary, we develop thin nanocomposite surfaces with electrically insulating property by electrospinning technique. In particular, Bi$_2$O$_3$ microparticules were adapted to PA 6.6 nanocomposite fibers. By means of fiber fineness, addition of Bi$_2$O$_3$ microparticules did not result to a considerable increase in fiber diameter of PA 6.6 composite fibers and this was result of easy electrospinnability of PA6.6 polymer. FT-IR spectra showed that structural formations were preserved and negligible shifts were observed due to concentration changes of Bi$_2$O$_3$ loadings in PA 6.6 nanocomposite fibers. According to DSC and TGA thermograms, thermal characteristics of samples developed and Bi$_2$O$_3$ loading decreased mass loss percentages for studied samples. Electrical conductivity of Bi$_2$O$_3$/PA6.6 coated PES spunbonds have been significantly declined by increasing Bi$_2$O$_3$ loading and increase in Bi$_2$O$_3$ loading lead to crucial increase in electrical resistivity. Results demonstrate that Bi$_2$O$_3$/PA6.6 nanocomposite structures are promising candidates for being used as insulating materials in miniaturized electronic devices with advantages of their light weightness and fineness. Further studies should be performed on different concentration values of various polymers.

### Declarations

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Conflict of Interest

The authors declare that we have no conflict of interest.

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