

PA6/SILVER BLENDS: INVESTIGATION OF MECHANICAL AND ELECTROMAGNETIC SHIELDING BEHAVIOUR OF ELECTROSPUN NANOFIBERS

PA6/GÜMÜŞ KARIŞIMLARI: ELEKTROEĞİRME İLE ÜRETİLMİŞ NANOLİFLERİN MEKANİK VE ELEKTROMANYETİK KALKANLAMA ETKİNLİKLERİNİN ARAŞTIRILMASI

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ABSTRACT

PA6 nanofibrous membranes containing different amount of silver nanoparticles AgNP (0wt.%, 1wt.% and 2wt.%) were fabricated by a basic electrospinning set up that includes “top to bottom” feeding system. Morphology of the membranes was observed with SEM analysis. Beadless and almost uniform nanofibers were yielded with the average fiber size ranged from 174 nm to 238 nm. Mechanical tests were also carried out in order to explore the tensile and elongation properties of the electrospun membranes, and it was noticed that the increased amount of AgNP in the blends decreased the mechanical performance of the membranes. In addition, fiber size distribution, feeding direction and agglomeration of AgNP were highly influent factors on both morphology and mechanical characteristics of the membranes. Electromagnetic shielding effectiveness (EMSE) of the electrospun nanofibrous membranes was determined according to the ASTM D4935-10 protocol by using coaxial transmission line measurement technique in the frequency range of 15–3000 MHz. It was observed that the membrane containing 2wt.% AgNP performed better EMSE than the others.

Keywords: Electrospinning, EMSE, nanofiber, nano silver, mechanical strength

ÖZET

Farklı oranlarda gümüş nano parçacık (AgNP ağırlıkça %0, %1 ve %2) içeren PA6 nano lif membranlar “üstten alta” beslemeli elektroçğirme ile üretilmiştir. Membranların morfolojileri SEM analiziyle incelenmiştir. Neredeyse eş dağılıma sahip ve ortalama boyutları 174 nm ile 238 nm arası değişen nano lifler elde edilmiştir. Elektroçğirmeyle elde edilen membranların çekme ve uzama özelliklerinin tayini için mekanik testler ayrıca gerçekleştirilmiştir. Yapılan deneylerde karışımlardaki AgNP miktarı arttıkça membranların mekanik performanslarının azaldığı tespit edilmiştir. Bunun yanı sıra, AgNP lif boyut dağılımının, besleme yönünün ve çökelpmenin membranların morfolojik ve mekanik karakteristikleri üzerinde büyük etkileri olduğu tespit edilmiştir. Membranların elektromanyetik kalkanlama etkinlikleri (EMKE) ASTM D4935-10 standardı baz alınarak koaksiyel transmisyon ölçüm tekniğine göre 15-3000 MHz aralığında belirlenmiştir. Yapılan deneylerde ağırlıkça %2 AgNP içeren membranın en iyi EMKE gösterdiği sonucuna varılmıştır.

Anahtar Kelimeler: Elektroçğirme, EMKE, nanolif, nanogümüş, mekanik dayanım

INTRODUCTION

Nanofibers and nanofibrous composites have recently gained impetus because of their distinguished properties such as large surface area to volume ratio, highly porous structure with very small pore size and expanded pore interconnectivity, variety of options in surface functionalities and superior mechanical performance compared with the other material forms. Hence, nanofibers ensure various specific usages in many developing application areas including tissue engineering, wound dressings, drug delivery, skin therapy, catalyst and enzyme carriers, filtration, protective equipments, nano-sensors and nano-electronics, composite reinforcement, energy harvest and storage, optics, electromagnetic interference shielding and so on [1–3].

Electrospinning is a very simple and versatile process that enables electrostatic forces to fabricate polymer fibers in submicron range [4]. Preparation of polymer fluid (solution or melt), charging of the fluid, formation of the cone-jet, fining of the instable polymer jet in an electric field and deposition of the fibers on a collecting platform are the characteristic processing steps of electrospinning. Solution properties (viscosity, conductivity, molecular weight, surface tension, polymer concentration, and solvent types and blends), process parameters (intensity of applied electric field, collection distance and feeding rate) and ambient conditions (humidity and temperature of the environment) are influent factors that affect fiber diameter and morphology during electrospinning [5-8].

Potential hazardous effects of electromagnetic interference (EMI) on living creatures as well as on the performance of electronic equipments are common concerns among scientists [9]. The fundamental cause of EMI formation is the undesired electromagnetic emission being radiated or conducted [10]. The term “Electromagnetic Shielding Effectiveness (EMSE)” is described as the inducing of EMI by using conductive and magnetic barriers [11]. This effectiveness strongly depends on electromagnetic frequency, thickness of the shield and the distance between the shield and the source. One can compute EMSE in decibels (dB) using Eq. (1):

$$SE = 10 \log \frac{P_0}{P_t} = 20 \log \frac{E_0}{E_t} = 20 \log \frac{H_0}{H_t} \quad (1)$$

where P_0 , E_0 and H_0 are the power, the electric and the magnetic field intensities pertaining to the shield, respectively and P_t , E_t and H_t are the factors transmitted through the shield [12].

Though metals are the most effective electromagnetic shielding materials, polymer composites are more likely to be put into use due to their lower cost and weight, corrosion resistance, better thermal stabilities, ease of manufacturing and shaping [13]. Therefore, applying the conductive components such as metallic particles into/onto the textile-based structures (fibers, fabrics, films *etc.*) is the main point in inducing the EMI effect. Several studies have recently been reported in the literature in terms of developing conductive nanostructures by using electrospinning technique. Erdem et. al. electrospun Polyamide 6 nanofibers and sputter coated them with gold and palladium to create

functional nanofibrous membranes that possess EMI shielding ability [14]. Kim et. al. used both electrospinning and metal deposition methods to prepare Fe_2O_3 added nanofibrous composites made of Ag-decorated poly(vinyl alcohol) for EMI shielding applications [15]. Chiscan et. al. explored the microwave frequency absorption of PVC/ Fe_2O_3 , PVC/ Fe_3O_4 , and PVC/ $CoFe_2O_4/CoO$ nanofibers produced by electrospinning [16]. Fu et. al. utilized two different methods as electrospinning and sol-gel to fabricate highly conductive silvered electrospun silica nanofibers via Poly(dopamine) functionalization [17]. Kim et. al. utilized metal deposition (Cu, Ni, Ag) and electrospinning techniques to obtain nanofiber mats with EMI shielding properties [18].

Unlike the above studies which are complicated and include more than one processing step, in our experimental study, silver nanoparticles (AgNP) were added into the polymer solution directly and the electrospinning process was conducted in newly manufactured equipment which was designed and constructed by our crew. In this test rig, metal particles in the solution are not allowed to precipitate or agglomerate for a while. High electrical conductivity, stretchability, corrosion resistance and thermal stability under wide temperature range make silver one of the most appropriate candidate for metal filler in composite structures [19-23]. Applications on electrical devices using silver nanowires such as electrodes, low temperature sintered conductive adhesives, transparent conductive film, superconductive thick film circuit, microwave absorbing materials and electromagnetic wave absorbing materials have also been carried out [24-30]. Nylon 6 (PA6) is considered as an important class of thermoplastics that has high modulus, good mechanical strength, dimensional stability under even elevated temperatures and chemical resistance against many moderately polar and non-polar organic species [31-33].

EMSE is defined as a specific value for EMI protection. On the other hand, mechanical characteristics are very crucial for the materials regarding manufacturability, lifetime and resistance against external effects. The purpose of this study is to produce AgNP incorporated PA6 nanofibrous membranes by using electrospinning technique and to determine the mechanical characteristics and EMI shielding performances of these membranes.

Experimental Work

2.1. Materials

PA6 (Tecomid[®], d : 1.49 g/cm³) was provided by Eurotec (Turkey). Silver nanoparticles (20nm, spherical, metal basis, d : 10.5 g/cm³) was obtained from US Research Nanomaterials, Inc. and formic acid (FA) was purchased from Sigma Aldrich (Germany).

2.2. Preparation and characterization of the polymer solution

Three different solutions (Pure PA6; PA6 with 1 wt.% AgNP; PA6 with 2 wt.% AgNP) were prepared for the experiments. Firstly, PA6 (20 wt.%) was dissolved in FA by utilizing laboratory type magnetic stirrer (Stuart, SB 162) at room temperature for 6 h. Then, AgNP was added to the solutions at the ratios mentioned above. As the nanoparticles added

into the polymer solution, they exhibited a tendency to precipitate after a while due to the clustering activity of the particles. In order to disperse these particles, after stirring, sonication (220V/50-60 Hz) was performed for 1 h in an ultrasound generator (Alexandra Ultrasonic). Viscosities of the solutions were determined with Brookfield Digital Viscosimeter by using S21 type spindle with the rotational speed of 20 rpm. Electrical conductivity of the solutions was also measured with a laboratory type conductivity meter (WTW, Cond3110) under ambient atmosphere.

2.3. Electrospinning process

Electrospinning was carried out in the self-designed laboratory spinning unit (feeding direction is from top to bottom, Figure 1). Matsusada Handy Type (Japan) was used as high voltage power supply unit, and microfluidics syringe pump (NE 1002X) was used for solution feeding. Each solution was placed in a 10 ml syringe and sent to the drum collector (covered with aluminum foil) through a 20-gauge nozzle. The power supply (AC) was set up for a positive voltage of 32 kV. The flow rate of the solution was also adjusted by setting up the syringe pump at 0.40 ml/h. The rotational speed of the drum collector was 100 rpm and its distance was set to be 13 cm away from the nozzle. During the experiments, relative humidity and temperature values ranged from 40% to 45% RH and 28°C to 30°C, respectively.

2.4. Mechanical Testing of Nanofibrous Membranes

Firstly, membrane thicknesses were measured according to TS 128 EN ISO 5084 by using R&B Cloth Thickness Tester (James H. Heal & Co. Ltd.). Then, in order to determine the mechanical properties (tensile strength and elongation) of the Pure PA6, PA6 with 1wt.% and PA6 with 2 wt.% nanofibrous membranes, tensile and recovery tests were performed by utilizing an Instron Machine (Instron4411) at ambient environment (22°C± 3°C and 50% ± 5%RH). The samples were cut into approximately 50 mm × 10 mm (length × width) in both machine and width directions in order to load properly into the uniaxial testing machine. During the experiment, 50N load cell under a cross-head speed of 10 mm/min was applied to the samples. Five

repetitions were taken for each sample in order to calculate the average tensile strength and elongation at break values.

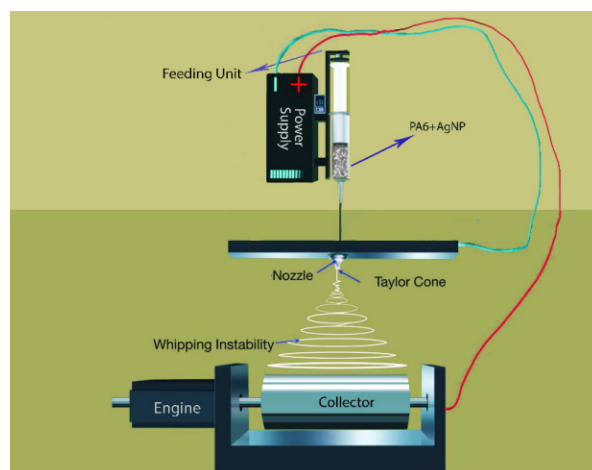


Figure 1. Schematic illustration of electrospinning set up

2.5. Measurement of Electromagnetic Shielding Effectiveness (EMSE)

All nanofibrous membranes were conditioned at 20°C± 2°C temperature and 65% ± 2% relative humidity. Measurements were repeated three times on different areas of the membranes and the average values of the measurements were calculated. A coaxial transmission line method specified in ASTM D4935-10 was used to test the EMSE of the knitted fabrics. The specimen was prepared with a standard test size of various thicknesses. The outer ring of the specimen was 133 mm in diameter. Two specimens were used for the test, one for reference and another for load testing. Various researchers have described the detailed set-up and testing procedure using a plane-wave electromagnetic field in the frequency range of 15MHz–3GHz. A network analyzer (Rohde Schwarz, ZVL) to generate and receive the EM signals and a shielding effectiveness test fixture (Electro-Metrics, Inc., EM-2107A) were used to measure the EMSE, which was measured in decibels (dB) [34,35].

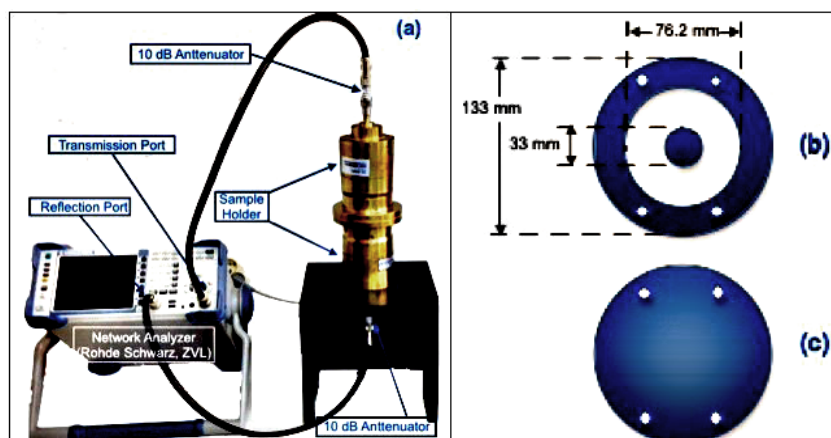


Figure 2. a) Set up of the Electromagnetic Shielding Effectiveness testing apparatus; (b) and (c) Specimen for reference and load respectively.

This standard determined the shielding effectiveness of the fabric (in our case nanofibrous membrane) using the insertion-loss method. The technique involved irradiating a flat, thin sample of the base material with an EM wave over the frequency range of interest, utilizing a coaxial transmission line with an interrupted inner conductor and a flanged outer conductor. A reference measurement for the empty cell was required for the shielding-effectiveness assessment (Figure 2(b)). The reference sample was placed between the flanges in the middle of the cell, covering only the flanges and the inner conductors. A load measurement was performed on a solid disk shape, which had an equal diameter with the flange (Figure 2(c)). The reference and the load measurement were performed on the same material. The shielding effectiveness values were evaluated from Eq. (2), which is the ratio of the incident field to that which passes through the material [34].

$$\text{EMSE (dB)} = 10 \log \frac{P_1}{P_2} \quad (2)$$

where P_1 (watts) is received power with the fabric and P_2 (watts) is received power without the fabric. The input power used was 0 dB, corresponding to 1 W. The dynamic range (difference between the maximum and minimum signals measurable by the system) of the system was 80 dB [36]. The results of shielding effectiveness (SE) were evaluated according to ASTM D4935 standards. SE of the textile materials was measured in dB and percent values corresponding to these dB values were pinned down referring to FTTS-FA-003 [37].

3. RESULTS AND DISCUSSION

3.1. Morphology of Nanofibers

Solution property is highly influent about the adjustment of electrospun fiber diameter and morphology. Table 1 illustrates the viscosity and conductivity measurement

results for individual electrospinning solutions of Pure PA6, PA6 with 1wt.% of AgNP and PA6 with 2 wt.% of AgNP. When the amount of AgNP was increased more than 2wt.% in the blend, some limitations about forming stable jet during electrospinning was observed. This was most probably due to increased agglomeration among AgNP at higher concentration in the solution. Increment of AgNP in the solutions caused an increase in viscosity due to increase in solution concentration with the incorporation of AgNP. Conductivity also reached to 5.12 $\mu\text{S/cm}$ for the PA6 with 2wt.% AgNP due to the increased amount of conductive metal particles in the solution.

It is commonly accepted that the solution viscosity is affected by polymer molecular weight and polymer/solvent blend ratio [38]. In our case, metal particles also contributed to the increase of solution viscosity. On the other hand, electrical conductivity of the solution is obviously critical for the generation of non-beaded nanofibers. Increase in the electrical conductivity of the solutions (charge volume density) can result in electrospun cylindrical fibers and finer fiber diameters since the polymer solution is subjected to more stretching under the high electrical field [39,40]. In Figure 3, SEM micrographs of electrospun nanofibrous membranes are presented. It was observed that beadless, mostly uniform and randomly distributed fibers were yielded. Also, very thin and highly porous webs among the fibers were formed which may be due to the uncontrolled drops of the solution from the nozzle (at top) to the rotating collector (on the ground) during electrospinning. Average fiber diameters were measured as 214 ± 159 nm for Pure PA6, 174 ± 70 nm for PA6 with 1 wt.% and 238 ± 162 nm for PA6 with 2 wt.%. In addition, thicknesses of electrospun nanofibrous membranes ranged from 0.138 mm to 0.156 mm (Table 2). Furthermore, it was found that AgNP particles with small size along with agglomerated AgNP were dispersed whole over the membrane by either embedded on the fiber surface or located among the fibers (Figure 3).

Table 1. Solution properties and electrospun nanofibrous membranes characteristics

Polymer	Viscosity (cP)	Conductivity ($\mu\text{S/cm}$)	Nanofiber Diameter (nm)
Pure PA6	988	4.49	214 ± 159
PA6 with 1 wt.% AgNP	1035	4.51	174 ± 70
PA6 with 2 wt.% AgNP	1643	5.12	238 ± 162

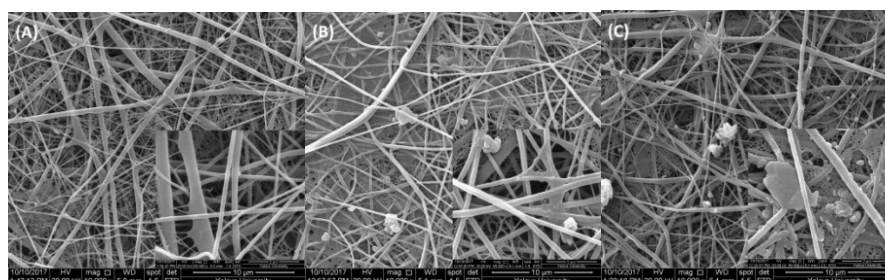


Figure 3. SEM pictures of electrospun nanofibrous membranes at 10000x and 50000x magnifications; (A) Pure PA6, (B) PA6 with 1wt.%, (C) PA6 with 2wt.%

It was noticed that much bigger and very thin sizes of fibers were fabricated together through the same system without changing processing parameters. This situation enlarged the fiber size distribution enormously for almost each nanofibrous membrane. Some justifications may be stated for this circumstance. Since our experiments were conducted in the vertically designed (from top to bottom) electrospinning set up, feeding rate of the solution to the electric field might be influenced from the feeding direction (opposite or in the same direction of gravity) and irregular polymer jet acceleration towards the collector.

Yarin et. al. reported that jet surface area increases significantly when the jet undergoes high stretching and elongation as a result of the electrical forces. Such increment in the jet surface area immediately accelerates solvent evaporation. Solvent rate decreases fast in the bending loops (approximately 90% solvent evaporates), only a few centimeters below the starting point of the bending (whipping) instability. Once the polymer concentration achieves to around 90%, the jet sustains elongating, but at a much lower rate. The decline in the elongation rate is because of the increment in viscosity and elastic modulus of the solution at greater polymer concentrations [41]. During our experiment, due to the irregular polymer jet acceleration –most probably because of the agglomerated particles in the solution–, the cross-sectional radius of the jet might be altered much while very thick and thin fibers were produced at the same time. However, the obtained fibers were still bead free.

Feeding direction may be another influent factor for the enlarged fiber distribution, because, in our previous experiments performed in another machine where the solution was fed from bottom to top, a fibrous structure with a more homogenous fiber size distribution was obtained even if the process conditions and solution concentrations were similar to the current study [14]. Therefore, feeding direction which was towards to gravity might cause the irregular feeding of the polymer solution into the electric field which induced production of very thin and thick fibers at the same time.

3.2. Mechanical properties of electrospun nanofibrous membranes

The tensile strength of a material expresses how much stress the material will tolerate before suffering permanent deformation/tearing. According to the tensile strength graph (Figure 4), the highest average values both in machine direction (11,13 MPa) and width direction (8,18 MPa) were recorded for Pure PA6 nanofibrous membrane. Tensile strength of the membranes declined gradually as the amount of AgNP increased in the membrane. This may be explained by the deformation of the membrane integrity to a certain degree, due to the locating of AgNP on the fibers surfaces and the pores among the fibers. On the other hand, the tensile strength of the nanofibrous membranes was found slightly greater in the machine direction (MD) comparing with the width direction (WD). However, difference between MD and WD was found greater for pure PA6 compare to other samples. The reason behind of this result may be the more parallel fibers laid along MD because of the rotating drum.

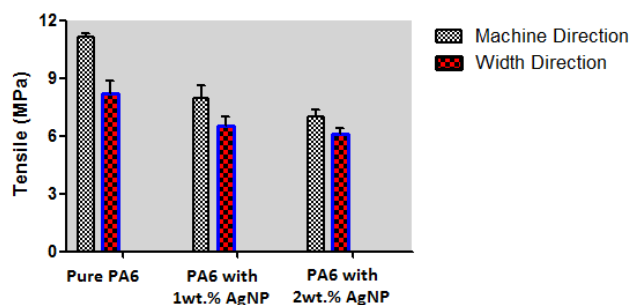


Figure 4. Tensile strength of electrospun nanofibrous membranes

In literature, there are several comments about the impacts of nanoparticles on the elongation properties of the electrospun membranes. For instance, An et. al. reported that the addition of nanoparticles to the polymers usually results in a reduction of the strain at break. On the other hand, Wang et. al. determined that the elongation at break increases by addition of nanoparticles [42,43]. In our case, elongation values decreased with introducing AgNP to the membranes. This may be attributed to the increase in stiffness of the membrane with the increase in AgNP concentration. Augmented concentration may lead to an increase in agglomeration, thus, higher stiffness and lower elongation [44]. Therefore, the highest average value was obtained for the Pure PA6 membrane in both machine (73%) and width (67%) directions.

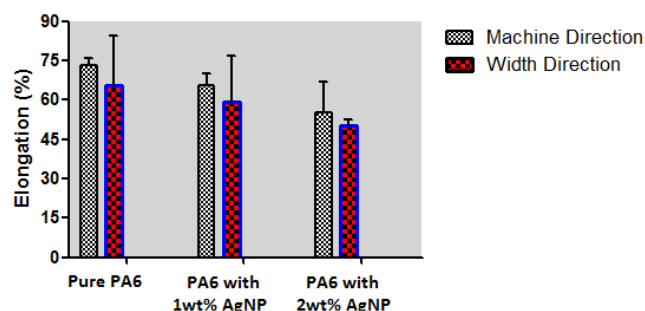


Figure 5. Elongation (%) of electrospun nanofibrous membranes

3.3. Electromagnetic shielding (EMSE) properties of electrospun nanofibrous membranes

In previous studies, several metal oxide nanofibers have been electrospun from sol-gel solutions and metallorganic precursors in order to obtain conductive structures [45,46]. Chen et. al. prepared conductive nanofibers by electrospinning of poly(methyl methacrylate) and silver trifluoroacetate in an organic cosolvent of methyl ethyl ketone and methanol. The final product gained from this study exhibited sheet resistances as low as 15 Ω /sq and total diffusive transmittances of 54% [47]. Demirsoy et. al prepared composite nanofibers of PAN with 1 wt.% and 3 wt.% AgNO₃ content by electrospinning. The conductivity of the nanofibrous membranes was measured as approximately 10-8S/cm [44]. Erdem et. al. sputter coated the electrospun PA6 nanofibers with gold and palladium to yield functional nanofibrous membranes that possess electromagnetic shielding ability. The coated membranes exhibited almost 14 dB EMSE [14].

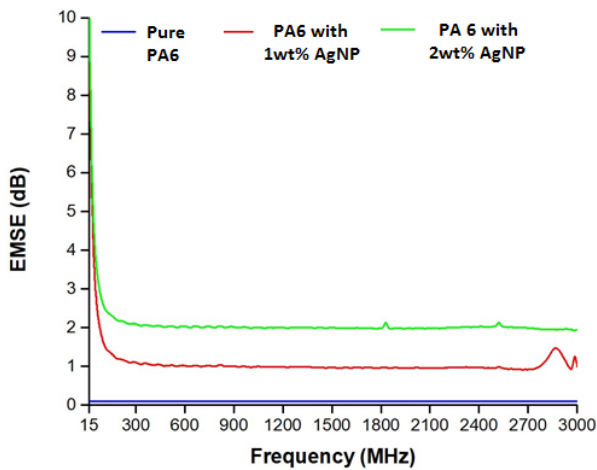


Figure 6. Electromagnetic shielding effectiveness (EMSE) of electrospun nanofibrous membranes

In our case, simple electrospinning process was performed to gain conductive membranes without the requirement of any further treatment. Figure 6 shows EMSE values of PA6/Ag nanofibrous membranes in 15-3000 MHz frequency ranges. The EMSE values of all PA6/Ag nanofibrous membranes showed a steady advance with the increment in frequency. Test results have clearly proved that EMSE is related to amount of Ag used in nanofibrous membrane because of increasing conductivity with AgNP fillers. The EMSE values of PA6/Ag nanofibrous membranes in the 15-3000 MHz frequency range were not high and below 3dB. Pure PA6 membrane did not perform any EMSE activity since it did not possess any conductive feature. AgNP have great potential to form highly conductive materials due to the increased surface area. Nanofibrous membranes are also very suitable to create a kind of networking environment for the interaction of conductive nanofillers to each other,

regarding establishing flexible and efficient conductive platforms. To increase the EMSE performance of such materials, selection of proper polymers and nanofillers, and optimization of processing parameters are very crucial.

4. CONCLUSIONS

In this study, EMSE and mechanical properties of PA6 nanofibrous membrane with different Ag nanoparticles concentrations produced by electrospinning were investigated. PA6/Ag nanofibrous membrane in the 15-3000 MHz frequency range depicted better performance than those of pure PA6. The results show that in order to achieve higher shielding performance, the percentage of the nanofiller (Ag) in the nanofibrous membrane must be increased to an extent not to encounter challenges in electrospinning process. The highest electromagnetic shielding found in this work was around 2 dB for PA6 nanofibrous membrane with 2 wt.% Ag. More precise control of electrospinning process as well as fortification of the solution by incorporating auxiliary chemicals may impede or minimize AgNP precipitation which also yields achievement of nanofibers with better morphology. This well dispersion may positively affect fiber size distribution in the matrix which is also related to improved mechanical strength. Feeding process from top to bottom ensures the existence of AgNPs in the fibers and should be well controlled to prevent dripping. Consequently, more precise control of these processes leads to either incorporation of more AgNPs in the solution (better EMSE values) or improved mechanical properties.

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