O 40. ANTIBACTERIAL PROPERTIES OF NYLON 6,6 NANOFIBERS CONTAINING SILVER NANOPARTICLES

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ABSTRACT: The aim of this study was to develop antimicrobial nanofibers to be a candidate for potential applications. Nylon 6,6 nanofibers were fabricated via electrospinning technique, then the silver nanoparticles (AgNPs) immobilization was performed in order to gain an antimicrobial activity. In this method, generated electrospun Nylon 6,6 nanofibers were immersed into $AgNO_3$ solution for 2 hours and removed from the solution, treated with water, and then reduced using $NaBH_4$ for 2 hours. The formation of AgNPs was occurred through the $NaBH_4$ reducing agent. The obtained Nylon 6,6 nanofibers containing AgNPs were washed with water and dried. Fabricated nanofibers were characterized using Scanning Electron Microscope (SEM) and Fourier-transform infrared (FT-IR) spectrophometer. Antibacterial activities of AgNPs immobilized nanofibers were evaluated using Gram-positive bacteria (*Staphylococcus aureus*) and Gram-negative bacteria (*Escherichia coli)*. The inhibition zones were measured and recorded. According to the analysis, nanofibers showed better antibacterial activity against to Gram-positive bacteria (*Staphylococcus aureus*) as compared to Gram-negative bacteria (*Escherichia coli).*

Keywords: Antibacterial, nanofiber, silver nanoparticle, nylon 6,6.

1. INTRODUCTION

In recent years, there has been an increasing interest on antibacterial products in a wide range of application areas such as water purification, protection, wound dressing, and textile industry etc (Kang et al. 2016). The products having antibacterial activity can be prepared in the form of beads, gels, films and fibers. Nanofibers are defined as fibers with diameters less than 1 micrometer. Nanofiber technology (fiber diameter less than 1 micrometer) is under development for the preparation of novel materials in nano-scale with multifunctional properties. Electrospinning is an elegant method for producing nanofibers with high porosity and high spesicific surface area (Huang et al. 2003). Electrospun polymer nanofibers containing silver nanoparticles has received much attention due to their antimicrobial properties. According to our knowledge, silver and silver compounds show excellent antimicrobial efficiency against organisms such as bacteria, fungi and viruse (Pant et al. 2012). AgNPs can damage the structure of the bacteria. Nanoparticles can also penetrate the cell membrane, where it interacts with phosphorous-containing DNA and attack thiol groups of respiratory chain enzymes, inhibiting cell division and respiration, finally leading to cell death (Jabur et al. 2017).

Main objective of this study was to prepare antibacterial (Nylon 6,6) nanofibers containing silver nanoparticles. Mechanical stirring and ultrasonic dispersion were used to prepare homogenous polymer-clay/nanoclay solutions. Then, the electrospinning technique was performed to achieve Nylon 6,6 nanofibers. Surface of the obtained nanofibers was loaded AgNPs using $AgNO_3$ salts. SEM, EDX and FT-IR spectra were utilized to focus on the morphology, surface elemental fiber mats, fibers and pore size diameters. Antibacterial activities of materials were evaluated by agar diffusion method.

2. MATERIAL AND METHOD

Nylon 6,6 and formic acid with purity of 99 vol% were obtained from Sigma Aldrich Company. Clay (Tixogel VP) and nanoclay (Cloisite 20A) were purchased from BYK company. Silver nitrate ($AgNO_3$, Sigma Aldrich) was used as a metal precursor.

2.1. Preparation of nanofibers

Before fabricating of nanofibers, polymer was dissolved in formic acid. Polymer-clay solution was mixed by magnetic stirrer and ultasonicator in order to achieve homogeneous solutions. Nanofibers were produced by spinning the polymeric solution with and without nanoclay. Nylon 6,6-formic acid solution was placed in a 10-ml syringe with 19-gage needle tip and electrospun onto a aluminum foil to produce Nylon nanofibers. Similarly, Nylon 6,6-clay/nanoclay solutions were electrospun with different parameters. The distance between collectors and spinneret were maintained at 15 cm. Clay and nanoclay were incorporated into the Nylon 6,6 solution in order to improve the mechanical properties of fibers.
Generated electrospun Nylon 6,6 nanofibers were immersed into $AgNO_3$ solution for 2 hours and removed from the solution, treated with water, and then reduced using $NaBH_4$ for 2 hours. The formation of AgNPs was occurred through the $NaBH_4$ reducing agent. The obtained Nylon 6,6 nanofibers containing AgNPs were washed with water and dried (Figure 1b).

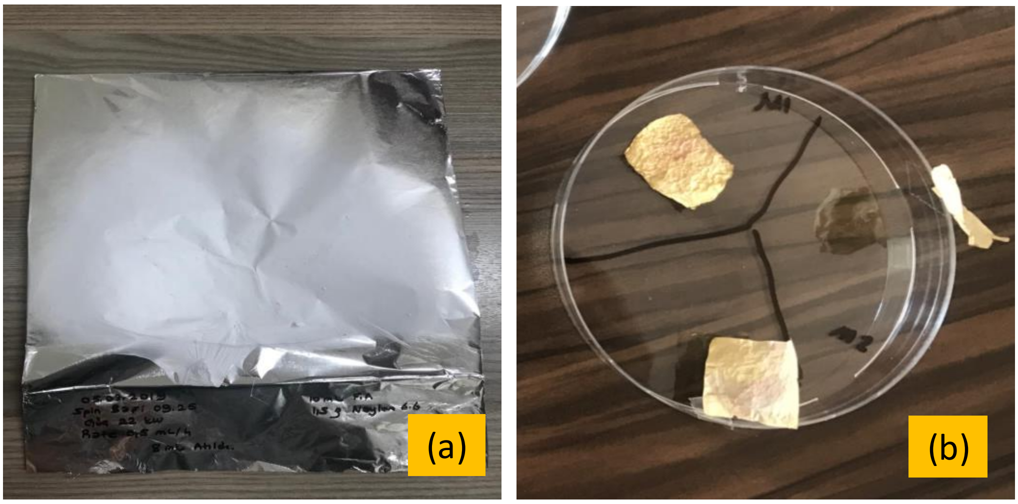


Figure 1. Electrospun Nylon 6,6 nanofibers and modified nanofibers with AgNPs

2.2. Characterization of nanofibers
Fiber morphology was analyzed using Scanning Electron Microscopy (ZEISS evo LS10). FT-IR spectrum was recorded at range 400 to 4000 cm^{-1} at 4 cm^{-1} resolution using a Bruker Vertex FT-IR spectrometer.

2.3. Antibacterial Activity
Antibacterial activities of Nylon 6,6 nanofibers containing AgNPs were tested against the Gram-positive bacterium, *Staphylococcus aureus* and the Gram-negative bacterium, *Escherichia coli* using agar diffusion method. Fibers cut into equal sizes were placed on nutrient agar plates previously seeded with 0.1 mL of the tested bacteria (~10^8 cfu/mL). After 24 hours of incubation at 37°C the diameter of inhibitory zones surrounding the samples were measured. Fibers lacking AgNPs were used as control.

3. RESEARCH FINDINGS
3.1. SEM analysis
Fig. 2 shows scanning electron micrographs of the fabricated Nylon 6,6-AgNPs fibers. The average diameters were 200-400 nm. As seen from the figure, uniform bead-less fibers were succesfully obtained. The formation of AgNPs was further confirmed by SEM-EDX analysis (Figure 3).

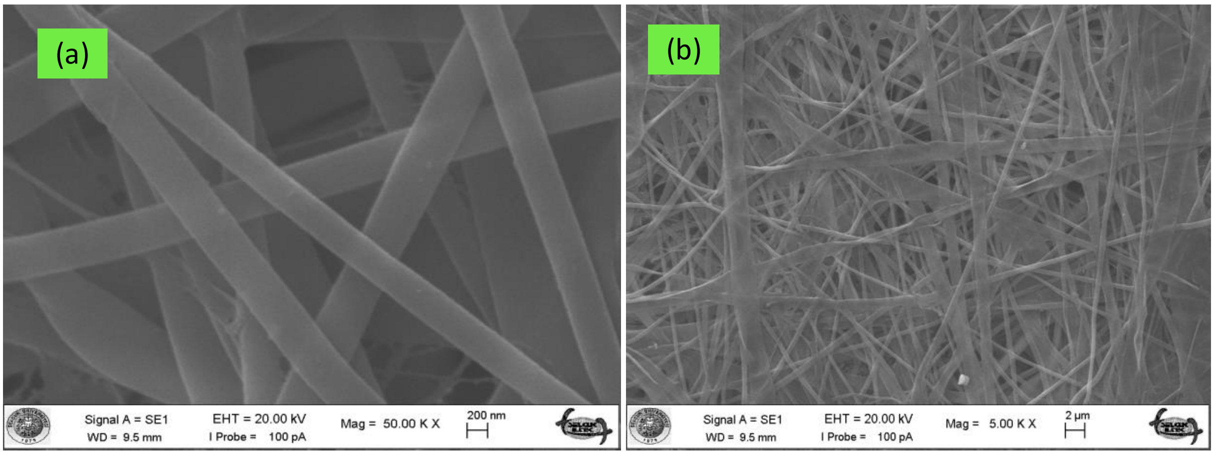
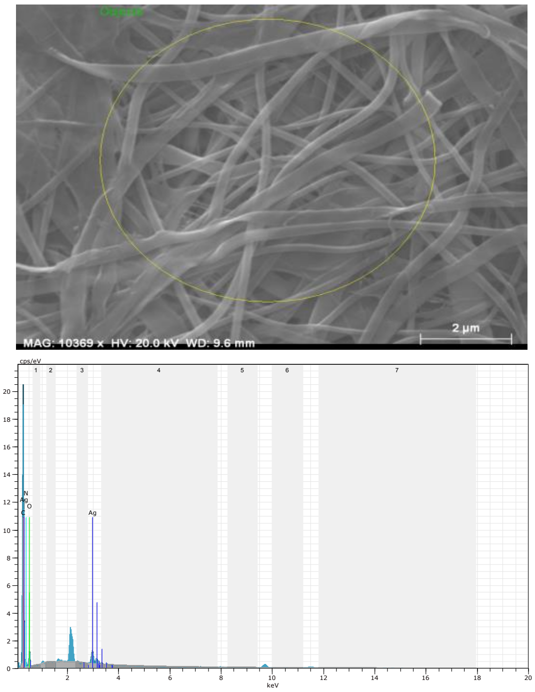


Figure 2. SEM micrographs of electrospun Nylon 6,6 nanofibers containing AgNPs

Energy dispersive X-ray (EDX) spectrum of AgNPs loaded Nylon 6,6 displayed in Fig. 3 shows strong carbon and oxygen peaks, which could be attributed to the surrounding Nylon 6,6 polymer. Three strong peaks of silver in the spectrum appeared around 3 keV indicate the existence of silver that was diffused into Nylon 6,6 nanofiber. The results obtained from EDX spectrum together with those of the SEM images are strong evidence of the deposition of AgNPs on the surface of nanofibers.



Spectrum: Objects

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
Carbon	K-series	49.36	49.36	55.90	15.5
Oxygen	K-series	29.04	29.04	24.69	10.2
Silver	L-series	1.85	1.85	0.23	0.1
Nitrogen	K-series	19.75	19.75	19.18	7.6
	Total:	100.00	100.00	100.00	

Figure 3. SEM-EDX analysis of nanofibers

3.2. FT-IR analysis

Figure 4 shows the FT-IR spectra of obtained Nylon 6,6 and nanofibrous mats coated with AgNPs. Nylon 6,6 has some characteristic peaks at 3300 cm^{-1}: (O-H deformation); 2940 ve 2860 cm^{-1}: (C-H stretching and O-H groups); 1640 cm^{-1}: (C=O deformation); 1420 cm^{-1}: (C-O-H deformation); 1256 cm^{-1}: (C-O stretching). No additional peak formation was observed in the FI-IR spectrum of Nylon 6,6-

AgNPs fibers, suggesting that no chemical bond formation occurred between the polymer and AgNPs. The intensities of peaks silver loaded nanofibers decreased.

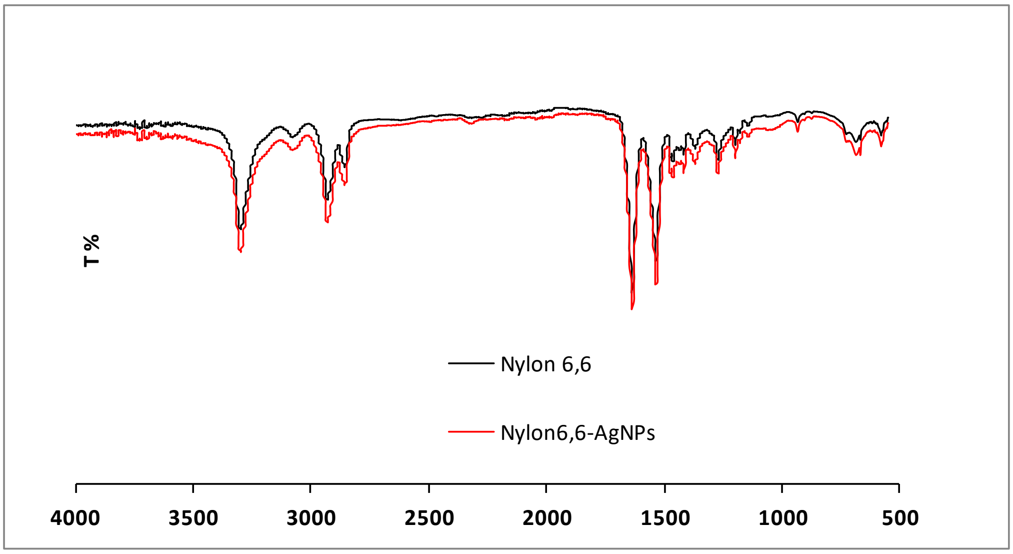


Figure 4. FT-IR spectrum of electrospun nanofibers

3.3. Assay of Antibacterial Activity

The antibacterial activity of AgNPs loaded nanofibers evaluated by diffusion method against two types of bacteria; Gram positive and Gram-negative (*S. aureus and E. coli* respectively) are shown in Figure 5 and 6. The control samples exhibited no inhibitory effect on the test bacteria. Nylon 6,6 nanofibers containing AgNPs showed low levels of inhibition against *E. coli.* Antibacterial activity of the fibers were greater on *S. aureus* compared to *E. coli*. Addition of clay/nanoclay to the fibers containing AgNPs resulted in a significant reduction/loss of antibacterial activiy.

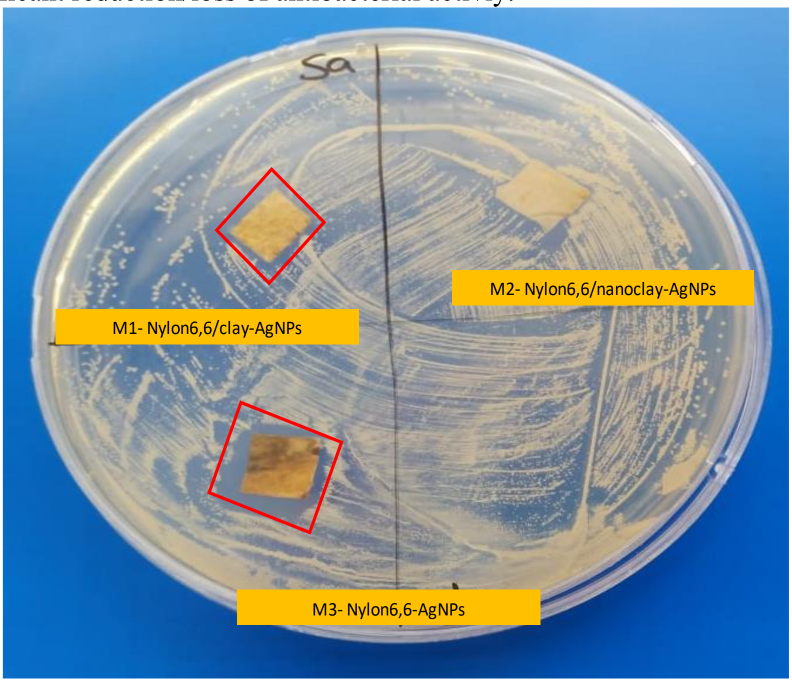


Figure 5. Inhibition zones for nanofibers against bacteria *Staphylococcus aureus*

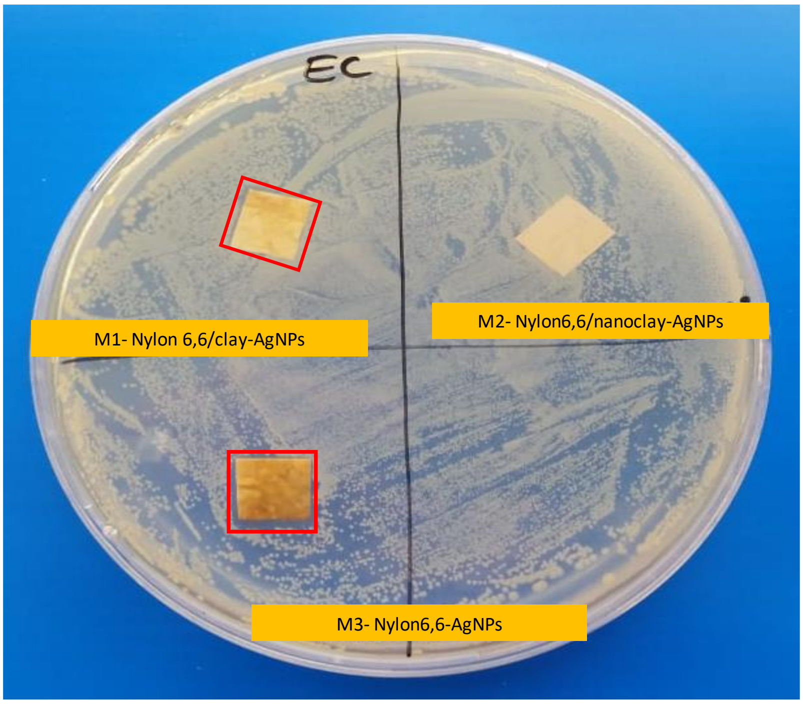


Figure 6. Inhibition zones for nanofibers against bacteria *Escherichia coli*

4. CONCLUSIONS

Antibacterial Nylon 6,6 nanofibers were succesfully fabricated in two steps. SEM and SEM-EDX analysis indicated that AgNPs were decorated on the surface of nanofibers. The antibacterial test shows that Nylon 6,6-AgNPs nanofibers exhibit a superior antimicrobial activity compared with nanofibers having clay or nanoclay. Thus, the Nylon 6,6-AgNPs nanofibers would have potential applications in areas such as water treatment techniques, health care and self-sterilizing textiles.

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