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Preparation and Studying of Nanofiber Membranes for Heavy Metals Absorption and Antibacterial Application

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Abstract: This search involved preparation of polymer nanofibers blend (polyamide 6/chitosan) for heavy metal absorption and (polyamide6/chitosan) mixed with nanosilver for antibacterial activity by electrospinning technique. Two concentration of solution blend of (PA6/Chitosan+formic acid) as (90/10) and (95/5) were prepared and pumped through electrospinning system. Also, these ratios of mixtures with nanosilver were mixed for preparing the antibacterial textile by electrospinning technique. Some test on these sample were performed such as: FTIR, AFM, Thickness, heavy metal absorption and antibacterial activity. The results showed the ability of (PA6/Chitosan) nanofibers membranes on absorb Pb^{+} ions also it gain antibacterial activity after mixed with nanosilver particles against to two types of bacteria, gram (-) and gram (+). The average fibers diameter obtained from Atomic Force Microscopy (AFM) test ranging from 93-107 nm.

Key words: Heavy metals, antibacterial activity, polyamide 6, electrospinning, chitosan nanofibers, FTIR

INTRODUCTION

Nanofibers membranes is appropriate for using in several filtration applications because it's ability on filtering the fine particles from air, due to their high surface area, high porous structure. Polymer nanofibers prepared by electrospinning technique through accelerating a charged polymer jet in an electric field. The output nanofibers have diameters from 10 μm (Lu and Ding, 2008). PA6 is synthetic polymer has high-quality mechanical properties, biodegradable and biocompatible with good resistant to the humidity.

The antibacterial effect can be exhibited through the filtration, adding of silver nanoparticle or silver ion to nanofibers, leads to control the microbe's growth, virus and bacteria inhibition, through absorbing of cell elements and destroy the interior configuration. Hung and Leung (2011) prepared composite nano fibers from integrating nano silver in nylon solution through electrospinning method for using in filtration. Different parameters of the electrospinning fibers is studied and establish that adding nano silver is suitable for antibacterial property in nylon composite fibers as well as, nano fiber morphology and nanofiber diameters are affected when solution concentration and the amount of nanosilver changes (Hung and Leung, 2011).

Abdelgwad *et al*. (2014) produced PVA/CS-Ag-NPs nanofibers blends in addition to PVA/CS nanofibers blendsby using electrospinning technique. They found that when PVA blended with CS, PVA isincompletely miscible with chitosan, also, Ag-NPs individual the solution o f polymer blend of PVA and CS enhanced the antibacterial capability of the electrospinning nano fibers and the electrospinning process also, that will supporteda good wound dressing material Abdelgawad *et al*. (2014).

Shahvaziyan *et al*. (2013) manufactured air mask from chitosan by using electrospinning method. By varying the conditions, the SEM consequences is the diameters of nanofiber are 100 and 500 nm at (%1.7, 1 mL/min 15 kV and 13 cm) conditions in addition to EDAX test publicized that chitosan nanofiber mask is further metal polluters are absorbed compared with the normal air mask (Shahvaziyan *et al*., 2013).

Ghani *et al*. (2014) prepared polyamide 6/chitosan nanofibers filtermedia and investigated direct and acidic dyes with regard to solution andmembrane parameters during filtration systemcharacterizedby Scanning Electron Microscopy (SEM), Water Contact Angle (WCA) and Fourier Transform Infrared Spectroscopy (FTIR). The optimum values for initial dye concentration, solutionp H, chitosan ratioand electrospinning time were predicted to be 5, 50 mg/L, 4 h, 30% and 5, 100 mg/L, 4 h, 10%, respectively, for achieving 96 and 95% removal of solopheny 1 red 3BL and polar yellow GN. They established that PA6/CS nanofibrous membrane has a huge appropriatepotential in dye removal from aqueous solutions (Ghani *et al*., 2014).

Sarma (2014) produced composite fibers by means of core-shell structure through using (co-axial) electrospinning technique. The nano silver (Ag) impregnate in PVA polymer shell forming while PA6

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polymer is the core. He clarified that nano silver stabilize near the composite fiber surface. These exceptional structures from (PVA-nano silver/PA6) nanofibers be able to locate applications in membranes and textile technology mainly in air filters besides antibacterial property. FE-SEM, EDS and DTA measured the existence of Ag nano on the outside surface of shell fiber (Abdelgawad *et al*., 2014).

The aim of this search is producing air filter membrane has the ability to absorb heavy metals (Pb^{+}) as well as antibacterial activity to use in hospital care, smoke lounge and general surgery applications.

MATERIALS AND METHODS

Experimental part: Polyamide 6 $(C_6H_{11}NO)n$ powder with molecular weight (80-100) g/mol, Chitosan $(C_6H_{11}NO_4)n$ as shells with ≥75% degree of deacetylation from India was provided for blending with polyamide 6. Silver nanopowder (Ag 99.99%, metal basis, 50-80 nm) obtained from Sigma-Aldrichchemistry, USA is used as antibacterial material. Formic Acid about 85% (CH_2O_2) with (101 Tb) and (1.57 cP) viscosity it used to dissolve the polymer materials. Two concentrations are prepared from (Polyamide6/chitosan) dissolving in formic acid at ratio (95/5) and (90/10) wt. % in addition to (Polyamide6+chitosan+Nano silver) concentration is prepared by dissolving in formic acid at ratio {(95/10)+2% Ag}. All concentrations put on magnetic starrier for 24 h, for getting homogenous solutions before electrospinning process.

Electrospinning process: In this study nano-azma direct system is used it consists of: direct high voltage power supply (DC-HV with 0 kV-50 kV). A syringe pump with flaw rate 0.1-10 mL/h stainless steel metallic collector as a rotate cylinder with (8 cm as a diameter and 13 cm as a length). A syringe which has the polymer solution is located in a syringe pump to create an invariable flow of fluid during the needle. A negative electrode of the HV was connected to the earthly metallic collector while the needle was connected to a positive electrode of HV.

The conditions that used in electrospinng process for all samples are 45 kV as voltage, 1 mL/h as flow rate, 15 cm as the distance between the needle and collector and 8 h is the time of electrospinning process.

RESULTS AND DISCUSSION

FTIR result: The functional groups present in the Polyamide6 (PA6) powder (raw material), (PA6/CS) nanofibers and (PA6/CS)-Ag-NPs nanofibers were studied by using the Fourier Transform Infrared spectroscopy (FTIR).

The FTIR spectra of PA6 powder shown in Fig. 1. The FTIR spectra clearly show fingerprint peaks of PA6, the wave numbers at 3000 cm^{-1} indicates the stretching vibration for N-H bond which are more evident in the PA6 spectrum, absorption peak at 2905.66 cm^{-1} is due to the C-H vibration of $-CH_3$. The absorption band at 1521 cm^{-1} represents the stretching vibration of the carbonyl group C = O. Other absorption characteristic for

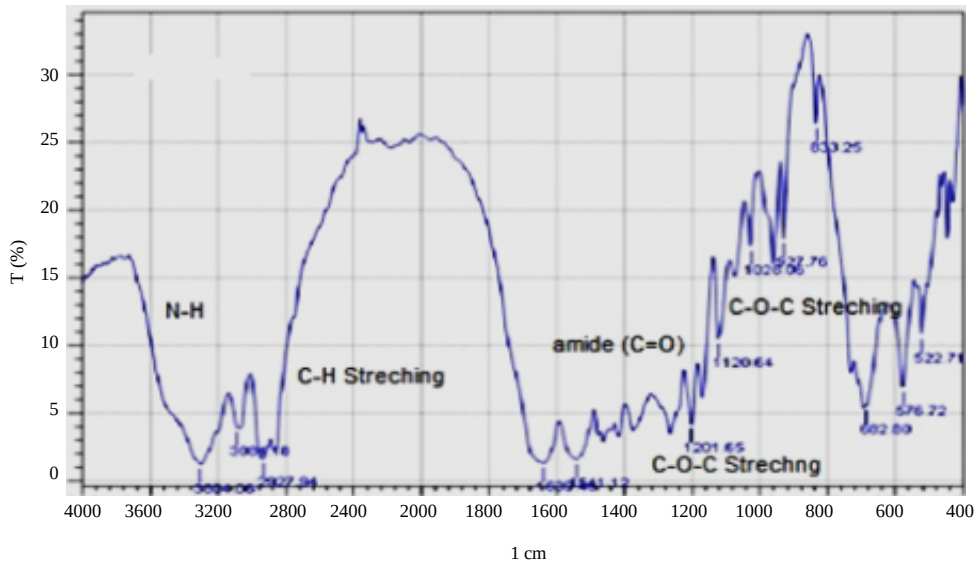

Fig. 1: FTIR analysis for PA6 powder

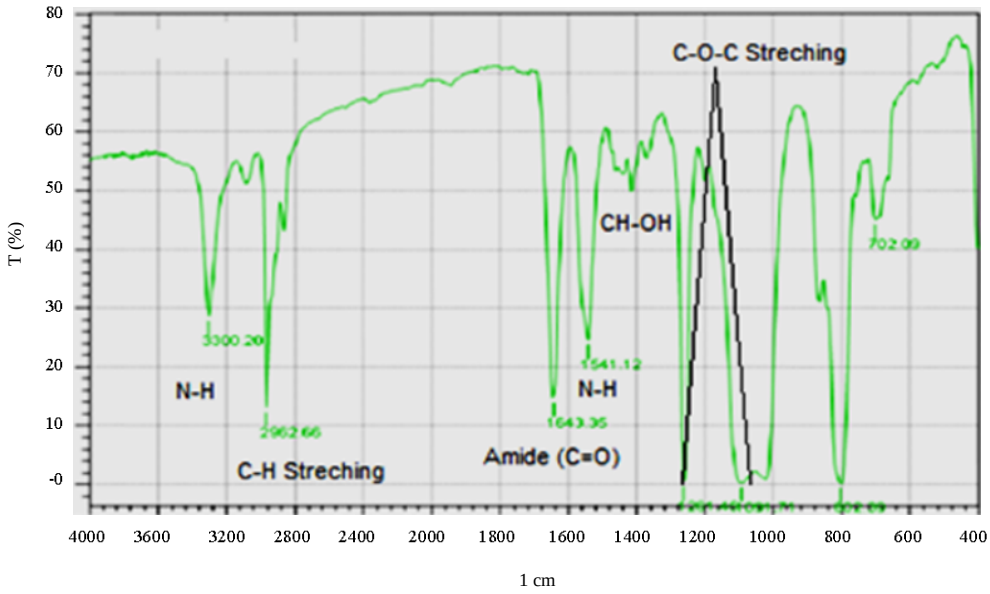

Fig. 2: FTIR analysis for PA6/CS nanofibers

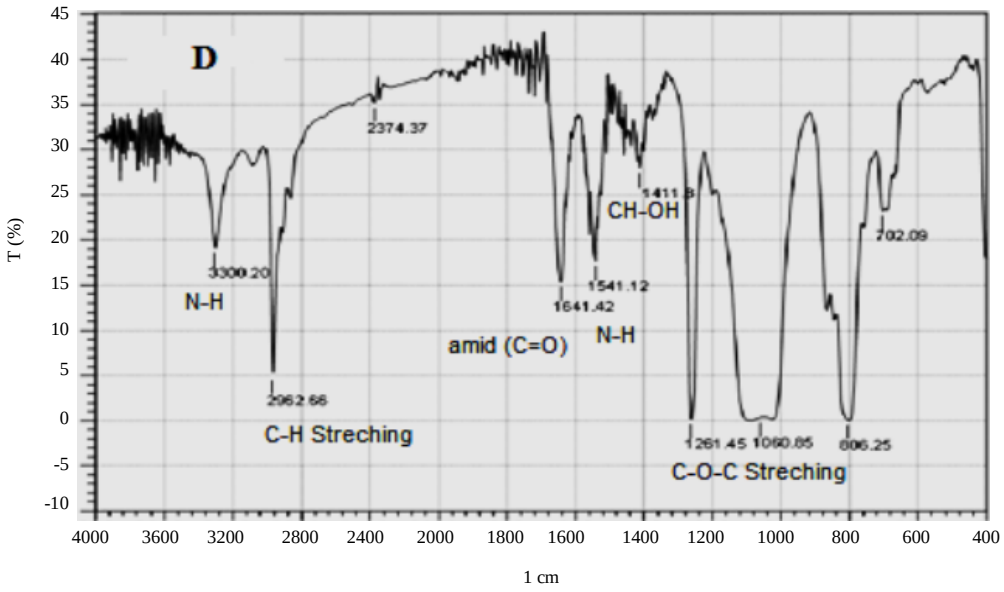

Fig. 3: FTIR analysis for (PA6/CS)-Ag-NPs nanofibers

PA6 are at 1265 and 1122 cm^{-1} indicating the bending vibration of C-O-C stretching which characterized PA6 functional groups (Ghani *et al*., 2014).

Figure 2 shows the proposed interaction of (PA6/CS), as illustrated previously in the presence of additional peaks and the variation in some of the peaks wavelengths and intensities. There are two peaks appear for chitosan at 1541 and 1401 cm^{-1} for N-H and CH-OH, respectively as well as the peak at 3300 cm^{-1} increased in its intensity due to the interaction between (PA6/CS) (Abdelgawad *et al*., 2014).

Figure 3 shows FTIR analysis for (PA6/CS)-Ag-NPs nanofibers. It is noticeability that when adding nanosilver particles, the intensity decreased from 55-31% to the fact that silver nanoparticles absorb the most of the radiation energy falling on the sample (Abdelgawad *et al*., 2014), also can notice from the figure that there is no new peak appears.

Atomic Force Microscopy (AFM): The surface morphology of samples by using AFM test were studied.

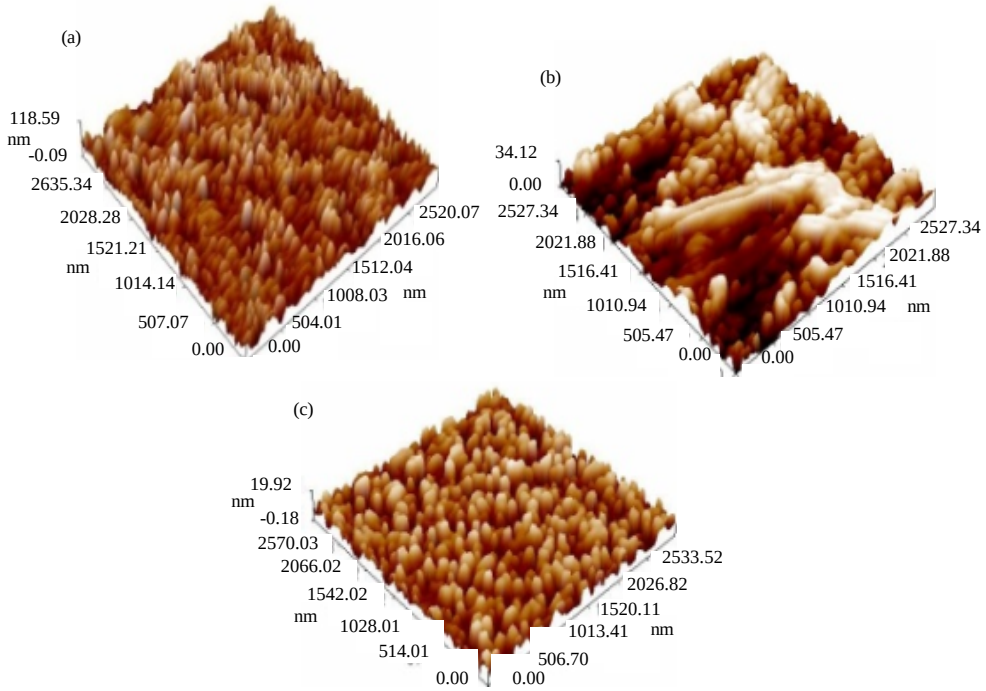

Fig. 4: AFM sample 3-D morphology for: a) PA6/CS (95/5); b) PA6/CS (90/10) and c) (PA6/CS)/Ag-NPs (90/10)/2%

Average fiber diameters are measured in this study to characterize the nanofibers membranes. Figure 4 shows 3-D morphology for PA6/CS(95/5), PA6/CS(90/10) and (PA6/CS)/Ag-NPs(90/10)/2%, respectively.

For samples PA6/CS(95/5) and PA6/CS(90/10), fiber diameters increasing from 93-97 nm as chitosan content increased from 5-10 wt. %, respectively. This increasing behavior is due to the increasing of polymer solution viscosity with the increasing of chitosan content which have high viscosity (Jia *et al*., 2007). At very high viscosity values there is difficulty in the ejection of jets from polymer solution and it results in larger fiber diameter. Moreover, the desirable morphology converted to defective structure with increasing the chitosan content in the blend solution as shown in Fig. 4. The diameter of nanofibers in the case of adding Ag-NPs is larger than without Ag-NPs (107 nm), this might be due to the fact that during electrospinning, the Ag-NPs are united or agglomerate in the syringe tip and sometime it abruptly drop down with nanofiber ejection, however, the presence of Ag-NPs in the blend improve its morphology and good uniformity (Jia *et al*., 2007).

Metals absorption result: The result shows that, the metal absorption efficiency decrease by increasing of chitosan in polymer blend nanofibers from 5-10%, this due to surface activity to absorb metal ions decreased (Sewvandi and Adikary, 2011). The non-homogenousity of the nanofibers obtained as well as the agglomeration of the nanofibers is the cause of the decreasing the surface active to the Pb^{+} ions as shown in Fig. 4 which shows that PA6/CS (95/5) sample is more homogenous and has uniform surface than PA6/CS(90/10) sample were the fibers agglomeration is observed (Table 1).

Table 1: The result of Pb^{+} absorption test

Samples	Total Pb^{2+} in original solution (mg/L)	Total Pb^{2+} in solution after filtration process (mg/L)
PA6/CS (95/10)	5.7	2.0
PA6/CS (90/10)	5.7	3.9

Table 2: Antibacterial activity of PA6/CS and PA6/CS/AgNPs against *S. aureusand E. coil*

Materials	Inhibition zone (mm) for *S. aureus*	Inhibition zone (mm) for *E. coil*
PA6/CS	56	35
PA6/CS/AgNP	56	35

The mechanism of absorption is by the amine group of chitosan polymer initiates a coordinate bond with the metallic ions. The bond is formed between the free electron pairs of nitrogen in the amine group and the void orbitals of the metal (Sewvandi and Adikary, 2011).

Anti-bacterial results: Table 2; Fig. 5 and 6, respectively shows the antibacterial activity of electrospinning fibers obtained from (PA6/CS) blend, the bacteria growth decreased in the state of (PA6/CS) blend because the chitosan disturbs polycation interaction "Protonated amino groups" with the bacteria surfaces that have negative charge. This results in loss permeability of membrane with cell leakage and lastly the cell will (Hang *et al*., 2010; Ignatova *et al*., 2006).

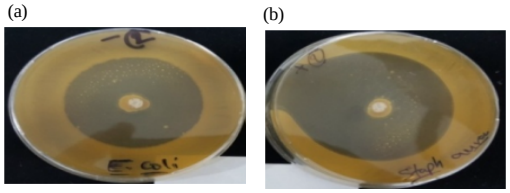

Fig. 5: Antibacterial activity of PA6/CS nanofibers for: a) *E. coil* and b) *S. asureus*

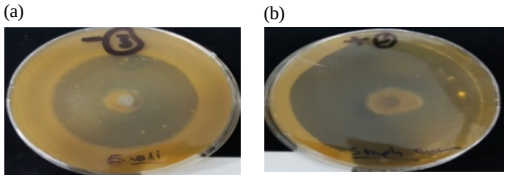

Fig. 6: Antibacterial activity of PA6/CS/AgNPs nanocomposite fibers for: a) *E. coil* and b): *S. aureus*

Figure 6 shows the antibacterial activity of (PA6/CS)/Ag-NPs electrospinning fiber mats. Notice the antibacterial activity of (PA6/CS)/Ag-NPs fiber mats increased, this due to the Ag-NPs attack the walls of cell and damage the permeability of cell's wall. Also, Ag-NPs murder the microbes (bacteria, viruses and fungi) through hindering the enzyme's effectiveness and halt the uptake of oxygen which necessary to microbes live (Sileikaite *et al*., 2006).

CONCLUSION

The nanofibers polymer blend which produced in this search by electrospinning process (PA6/CS)(95/5) wt. % and (PA6/CS)(90/10)wt. % are very effective for heavy metal (Pb^{+} ions) absorption as well as have antibacterial activity for *E. coli* as a negative type and *S. aureus* as a positive type. The antibacterial activity of PA6/CS increased with adding Ag-NPs to the nanofibers polymer blend. The preparing fibers can use in different application as air filtration for the industrial and medical field.

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