Colloids and Surfaces A: Physicochem. Eng. Aspects 395 (2012) 94–99

Contents lists available at SciVerse ScienceDirect

Colloids and Surfaces A: Physicochemical and Engineering Aspects

journal homepage: www.elsevier.com/locate/colsurfa

Characterization and antibacterial properties of Ag NPs loaded nylon-6 nanocomposite prepared by one-step electrospinning process

Bishweshwar Pant[a], Hem Raj Pant[a,b], Dipendra Raj Pandeya[c], Gopal Panthi[a], Ki Taek Nam[d], Seong Tshool Hong[c], Cheol Sang Kim[a,e], Hak Yong Kim[d,*]

[a] Department of Bionano system Engineering, Chonbuk National University, Republic of Korea
[b] Department of Engineering Science and Humanities, Pulchowk Campus, Tribhuvan University, Nepal
[c] Department of Microbiology and Immunology, Chonbuk National University, Republic of Korea
[d] Department of Organic Materials and Fiber Engineering, Chonbuk National University, Republic of Korea
[e] Division of Mechanical Design Engineering, Chonbuk National University, Republic of Korea

ARTICLE INFO

Article history:
Received 23 September 2011
Received in revised form 9 November 2011
Accepted 7 December 2011
Available online 16 December 2011

Keywords:
Electrospinning
Nanofibers
Ag NPs
Reduction
Antibacterial

ABSTRACT

A facile one-step approach to fabricate nylon-6 nanofibers decorated with silver nanoparticles by electrospinning has been reported. The method did not need post-treatments and could be carried out at ambient room condition. It employed the electrospinning solvents [formic acid and methoxy poly(ethylene glycol)] as a reducing agent for the conversion of $AgNO_3$ into Ag NPs during the solution preparation for electrospinning. The resultant Ag/nylon-6 hybrid nanofibers showed a smooth fibrous structure, with controlled size of Ag NPs uniformly dispersed throughout the nylon-6 matrix. The size of Ag NPs could be controlled by regulating the standing time duration of electrospinning solution. These hybrid nylon-6 composite nanofibers exhibited antibacterial activity against both Gram-negative *Escherichia coli* and Gram-positive *Staphylococcus aureus*. Therefore, the obtained nylon-6 nanofibrous mats loaded with Ag NPs can be used in different areas such as wound dressing, water filters etc.

1. Introduction

Electrospinning is regarded as a simple and versatile technique for fabricating continuous fibers not only from polymers [1,2] but also from inorganic [3] and hybrid (organic–inorganic) compounds [4–6]. Recently, unique mechanical, electrical, chemical and optical properties have been achieved by decorating polymer fibers with metal nanoparticles (NPs) [7–10], which have different applications in a wide range of areas, e.g., filtration, protective clothing, catalysis, sensors, energy storage, biomaterials [6,9,10]. Amounts, size and proper decoration of metal NPs on the polymer matrix can effectively control the properties of these hybrid nanocomposites [11]. Recently, the utilization of electrospun polymer nanofibers with embedded silver NPs has attracted much attention mainly due to its antimicrobial activities [12–15]. Silver NPs show antibacterial activity toward germs on contact without release of toxic biocides [16,17]. Therefore, the antimicrobial properties of Ag NPs can be considered as non-toxic and environmental friendly material in biomedical application. The Ag NPs filled polymeric materials have to release the Ag ions to a pathogenic environment continuously in order to be efficacious [18]. The loading of Ag NPs on biocompatible polymer matrix was usually achieved either by reducing $AgNO_3$ into Ag NPs in polymer solution prior to electrospinning [19] or by using post treatments process, such as UV radiation, thermal or chemical reduction of the electrospun composite fibers [20]. Mixing of Ag NPs in polymer solution prior to electrospinning causes the aggregation of NPs, whereas post treatment reduction of Ag NPs on electrospun mats is time and energy consuming. Furthermore, the reducing agents used for post treatment are toxic. Therefore, a facile and feasible approach to attain good dispersion of Ag NPs in the polymer fibers is highly desirable.

Here, we demonstrated a novel one-step route for uniformly assembling the in situ formed Ag NPs by solvent reduction of polymer solution on the surface of electrospun nylon-6 nanofibers driven by either coordination bond or interfacial hydrogen bond formation. In particular, we have investigated the effect of the standing time duration of polymeric solution containing $AgNO_3$ on the assembly of Ag NPs on nylon-6 nanofibers by influencing the combined reduction efficiency of formic acid (HCOOH) and methoxy poly(ethylene glycol) (MPEG) (used as solvent). Formic acid only acts as reducing agent whereas MPEG can act as both reducing as well as capping agent. In case of nylon-6 electrospun nanofibers obtained from the solution of short standing time, the attachment of Ag NPs on the surface of nylon-6 fibers may cause

* Corresponding author. Tel.: +82 632702351; fax: +82 632704249.
E-mail address: khy@jbnu.ac.kr (H.Y. Kim).

0927-7757/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.colsurfa.2011.12.011

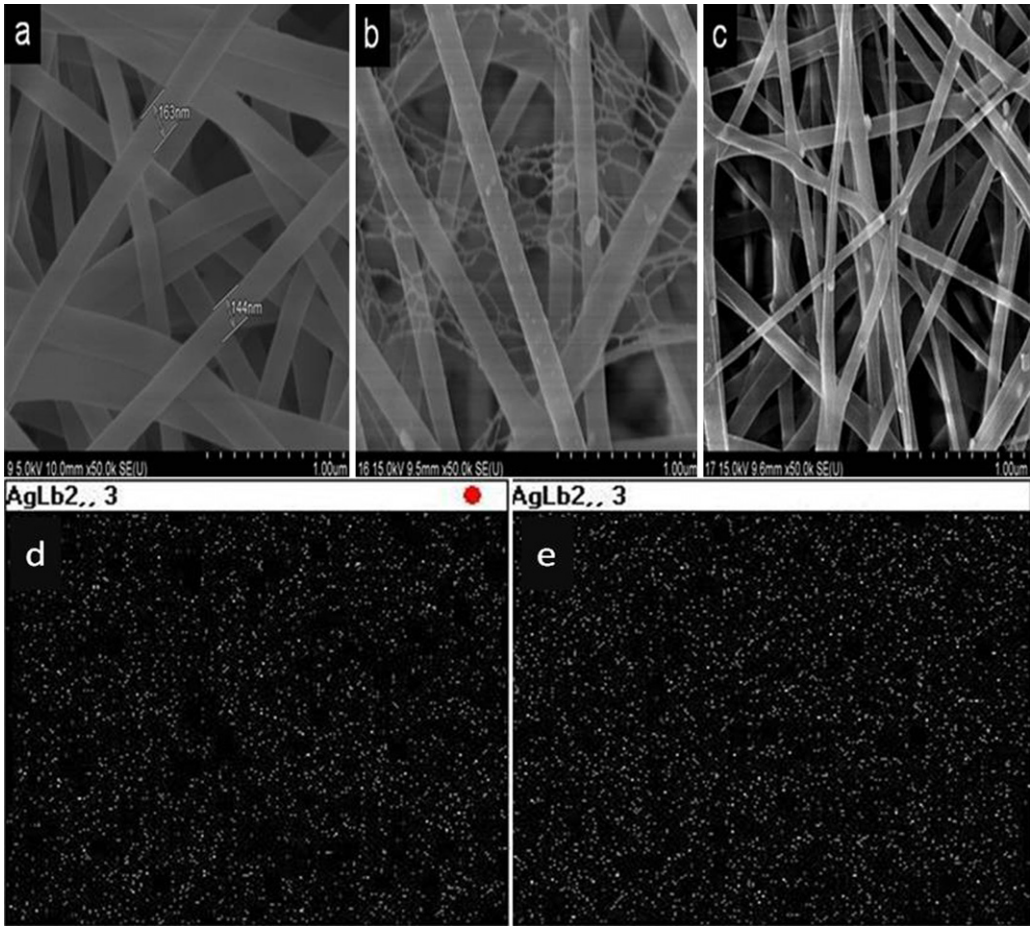

Fig. 1. FE-SEM images of pristine nylon-6 (a), m1 (b), and m2 (c) mats, and FE-SEM metal mapping images of m1 (d) and m2 (e).

by the formation of coordination-bond between the lone-pair of electrons (present in amide group of nylon-6) and Ag metal. However, the attachment of Ag NPs on the surface of nylon-6 fibers obtained from longer standing time solution may be due to the formation of interfacial hydrogen-bonding interactions between amide groups of nylon-6 and hydroxyl group of MPEG. Because silver NPs have long been known to exhibit strong inhibitory and bactericidal effects as well as this effect depends upon their size and shape, the antibacterial activity of these Ag/nylon-6 nanocomposites was evaluated against Gram-negative and Gram-positive microorganisms.

2. Experimental

2.1. Material and methods

Nylon-6 (medium molecular weight of KN 120 grade, Kolon, Korea) was used for this study. Formic acid/acetic acid (Showa, Japan) as well as Methoxy poly(ethylene glycol) oligomer (MW = 550, Aldrich, USA) were used as solvent without any purification. Silver nitrate (Showa, Japan) was used as metal precursor.

Ag/nylon-6 hybrid mats were prepared from the solution containing 2.5 g of $AgNO_3$ solution (10 wt% $AgNO_3$, 40 wt% MPEG, and 50 wt% CH_3COOH) and 25 g of 22 wt% nylon-6 solution prepared in 4:1 ratio by weight of HCOOH and CH_3COOH. $AgNO_3$ was dissolved in CH_3COOH/MPEG followed by mixing with nylon-6 solution and standing for different time duration. Electrospinning was carried out at 20 kV (applied voltage) and 18 cm (working distance) by taking the fraction of this solution at two interval of time i.e., after the standing time of 5 h and 20 h. The mats obtained from 5 and 20 h standing time of the same solution were named as m1 and m2, respectively.

2.2. Characterization

The morphology of the electrospun mats was investigated using FE-SEM (S-4700, Hitachi, Japan). The EDX spectrum of an Ag/nylon-6 nanocomposite mat was also recorded with the same FE-SEM instrument. The size, shape, and manner of deposition of Ag NPs on the fiber surface were investigated via transmission electron microscopy (JEM-2010, JEOL, Japan). Information about the phase and crystallinity was obtained with a Rigaku X-ray diffractometer (XRD, Rigaku, Japan) with Cu K_α (λ = 1.540 Å) radiation over Bragg angles ranging from 10° to 80°. The stability of Ag NPs on the surface of nylon-6 nanofibers was studied by the extraction of mats into the water for one week.

2.3. Antibacterial performance

The antimicrobial properties of the mats were quantitatively evaluated using Gram-positive and Gram-negative bacteria. The bacteria used in this study were *Escherichia coli* ATCC 52922 and *Staphylococcus aureus* ATCC 29231 as model organisms. Bacterial inoculum was prepared in Tryptic Soy broth (medium pH 7.3) containing 17 g/l Pancreatic Digest of Casein, 3 g/l Papaic Digest of Soybean, 2.5 g/l Dextrose, 5 g/l NaCl and 2.5 g/L Dipotassium phosphate to give a bacterial concentration of about 4.5×10^8 CFU/ml, which was incubated at 37 °C for 12 h with shaking. Furthermore,