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Hem Raj Pant
Engineering Science and Humanities Department, Pulchowk Campus, IOE, Tribhuvan Univesity, Kathmandu, Nepal

Correspondence
Hem Raj Pant
Engineering Science and Humanities Department, Pulchowk Campus, IOE, Tribhuvan Univesity, Kathmandu, Nepal

Synthesis, Characterization, and antibacterial investigation of copper nanoparticles incorporated PAN nanofibers via electrospinning technique

Hem Raj Pant

Abstract
Composite PAN nanofibers containing well decorated Cu NPs on there surface as well as inside the fibers are easily fabricated using electrospinning process. Well desperced sol gel system of Cu powder and PAN viscus solution was directy allowed to electrospinning. The morphological characterization, carried out using SEM and TEM, showed that the Cu NPs were distributed on the surface as well as incorporated inside the PAN nanofibers. The as-synthesized hybrid composite fibers showed remarkable antibacterial activity and can be used as antibacterial membrane.

Keywords: Electrospinning; PAN nanofiber; Cu NPs, Antibacterial

1. Introduction

During the past few decades, electrospinning has been considered as a promising technique for the production of fibers having a diameter in the range of a few microns to nanometer scale. Electrospinning has been studied widely because of its efficiency and simplicity for the fabrication of nanofibrous structures. The electrospun nanofibers exhibit outstanding characteristics, such as very large surface-area-to-volume ratio and high porosity with very small pore size. Due to these properties, the electrospun nanofibers can be applied in various fields such as filtration, protective clothing, catalysis, energy storage, biomedical, sensor, etc. [1]. Furthermore, scientific research has been concerned about the development of the organic/inorganic composite fibers via electrospinning in order to achieve superior optical, mechanical, electrical as well as magnetic properties which further widens the application of the electrospun nanofibers [2].

In recent years, great interest has been devoted to the synthesis of metallic nanoparticles in order to explore the special properties which are considerably different from those of their bulk counterparts. Metal nanoparticles such as Ag, Au, Al, Pd, Zn, Cu have been used in a wide range including biomedical, catalysis, super capacitor, optical and sensor [3]. Compared with other metallic nanoparticles, Cu NPs are specifically focused due to their low processing cost, high conductivity, and high catalytic activity. Nowadays, bacterial contaminations are still a major issue in environmental and biomedical field. Lately, Cu nanoparticles have attracted significant interest due to their broad-spectrum and highly effective antibacterial activity with relatively low cost and high scalability [4]. It is well known that the smaller sized metal nanoparticles possess higher antibacterial activity, however, as the size decreases, the metal nanoparticles very easily aggregate which results in a significant reduction of the antibacterial efficiency. Furthermore, the easily oxidized nature of copper also limits its utilization for various applications [5]. Supporting of the nanoparticles in a proper matrix might be an efficient strategy to overcome the problem of aggregation [6]. Accordingly, the electrospun nanofibers can be considered as a good supporter for the metal nanoparticles. Cu NPs can be incorporated into polymer matrices, including polymer nanofibers. The incorporation of Cu NPs into a nanofibrous structure is an attractive method, because Cu NPs distributed evenly in a nanofibrous structure can be used as an antibacterial and antifungal agent.

In this contribution, we synthesized Cu decorated Polyacrylonitrile (PAN) nanofibers by electrospinning technique.

In the Cu/PAN composite nanofiber, PAN act as a polymer template and Cu NPs have been incorporated completely and densely on the fiber surface without agglomeration. As-synthesized composite fibers were tested for antibacterial properties against *E. coli* bacteria.

2. Experimental

2.1. Materials

Polyacrylonitrile (PAN, MW= 150,000, medical grade) was purchased from Sigma- Aldrich. Copper powder (Cu, 99.9% purity analytical grade) was obtained from Osaka Chemicals Ltd., Japan. N, N-dimethylformamide (DMF) (analytical grade; Showa Chemicals Ltd., Japan) was used as solvents. All chemicals were analytic grade as used without further purification.

2.2. Fabrication of Cu/PAN hybrid nanofiber

First of all, 10% PAN solution containing 0.25 g of copper particle was prepared by dissolving into N, N-dimethylformamide (DMF). After stirring at room temperature for 12 h, the electrospinning was carried out in which the applied current was 15 kV and distance from the tip of the nozzle to the collector was 15 cm. As-spun Cu/PAN nanofibers were collected in aluminum foil and; vacuum dried at 70 °C for 12 h before analyses. For comparison, pristine PAN nanofibers were also fabricated without using Cu NPs. Fig. 1 shows the schematic diagram for the preparation of nanofibers by electrospinning.

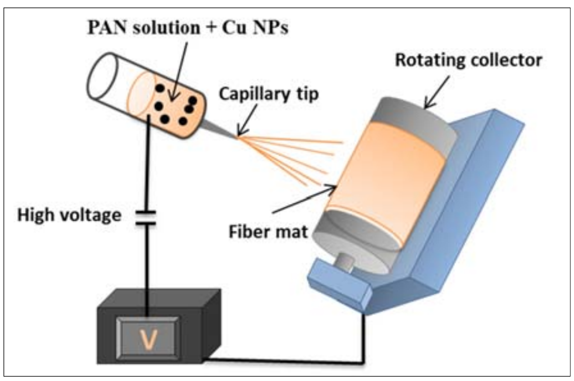

Fig 1: Schematic diagram showing the electrospinning setup.

2.3. Characterization

The morphology of the electrospun nanofibers was observed using field-emission scanning electron microscopy (FE-SEM, Hitachi S-7400, Hitachi, Japan) and scanning electron microscopy (SEM, Hitachi S-7400, Hitachi, Japan). The EDS of the as synthesized mat was also recorded with the same FESEM instrument. The size, shape and manner of deposition of the Cu NPs throughout the fiber body was investigated by transmission electron microscopy (JEM-2010, JEOL, Japan).

2.4. Antibacterial test

The antimicrobial properties of the mats were quantitatively evaluated using zone inhibition test against Gram-negative bacteria. The bacteria used in this study were *Escherichia coli* ATCC 52922 as model organisms. Bacteria were cultured in a tryptone soy broth medium at 37° C for 8 h, and pellets were suspended in an isotonic solution. Finally, the bacterial cell suspension was diluted to obtain cells of 10 CFU/mL. The electrospun samples were cut into a circular disk shape of 7 mm diameter, washed with PBS solution, and sterilized in UV light for 3 h. 100µ l of bacterial suspension was spread on a tryptone soy agar plate and the sterilized-samples were gently placed over the plate-containing respective bacteria. The plates were incubated for 24 h at 37 °C. Later, each agar plate was visually inspected for the presence of bacterial growth around the sample, and the diameter of the inhibition zone was measured.

3. Results and discussion

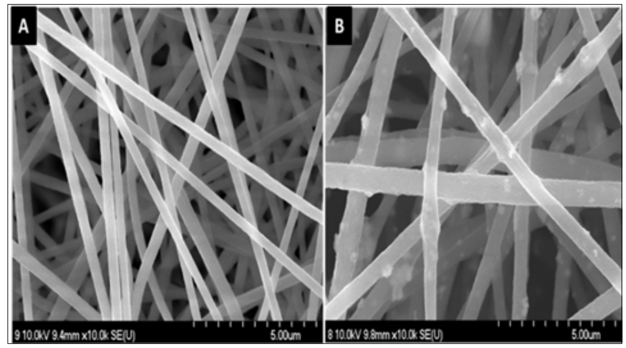

Fig 2: FE-SEM images of pristine PAN nanofiber (A) and Cu/PAN nanofiber (B).

Fig. 2 shows the morphology of as synthesized pristine PAN and Cu/PAN mats. It is clear that the pristine PAN nanofibers exhibited smooth, bead free, and continuous morphology. Figure 2(B) revealed Cu/PAN nanocomposite fiber in which copper nanoparticles are homogeneously and strongly attached throughout the surface of the PAN nanofibers by any physical and chemical means. After incorporation of Cu NPs to the PAN nanofiber, the resulting composite nanofiber still preserved its fiber morphology. The deposition of Cu NPs to the PAN nanofibers via electrospinning led to the increase in the diameter of the resulting hybrid nanofiber. The average diameter of the pristine nanofiber was found to be 500 ± 20 nm whereas the diameter of the Cu/PAN nanocomposite fiber was recorded to be 750 ± 20 nm. The presence of Cu on the PAN nanofiber was confirmed by the EDS analysis. As in figure 3, the EDS study showed that the composite fiber contains carbon, oxygen and copper. No further elements were detected as shown in the figure. To clearly assess the assembling structure of the Cu NPs on the surface of PAN nanofibers, TEM images were taken. Fig. 4 shows that a large number of Cu NPs with spherical shape are distributed homogeneously on the PAN nanofibers. It can be clearly seen that the Cu NPs are incorporated inside the nanofibers with good distribution. This strategy might be helpful for the long term utilization of Cu NPs as the NPs are inside the fibers.

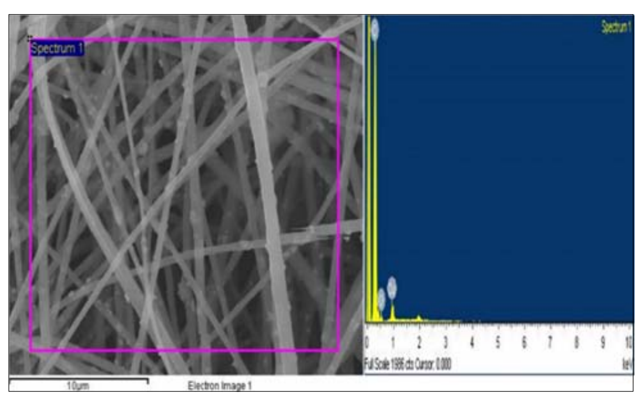

Fig 3: EDS spectra of the Cu/PAN composite nanofiber.

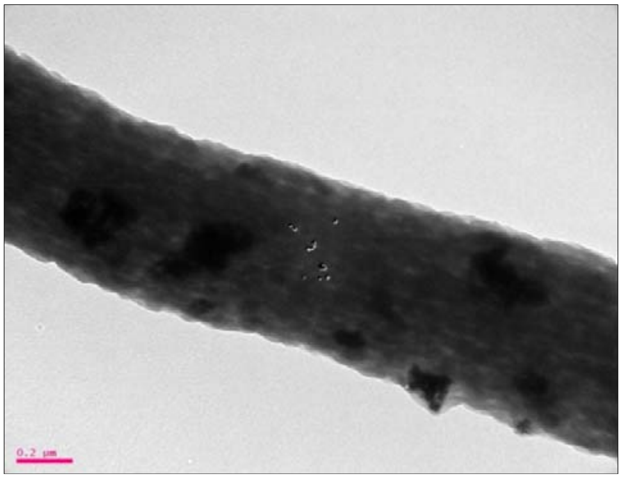

Fig 4: TEM image of Cu/PAN composite nanofiber.

The effect of the obtained composite nanofibers on the antibacterial efficiency was measured using zone of inhibition method of a bacterial solution at room temperature and compared with pristine PAN nanofibers. Among microorganisms, *E coli* is the most common etiological agents for water borne infections. Therefore, it was selected as a model organism for antibacterial study of the prepared nanostructures. Fig. 5 shows the optical images showing the zone of inhibition for *E. coli* incubated petri plates, grown in the presence of nanofibers. In the figure, it can be clearly seen that, in the case of pristine PAN nanofibers there is no zone of inhibition, which suggests that pristine PAN nanofiber mat lacks antibacterial property. However, the clear zone of inhibition was observed in the case of Cu/PAN composite fiber. The zone of inhibition in the case of composite nanofiber is due to the presence of Cu NPs inside the nanofibers. Therefore, it clearly reveals the role of Cu NPs in bactericidal activity. Several previous studies show that copper and its forms are potent antimicrobial agents; however, the exact mechanism of antibacterial action is not clear yet [7]. The possible antibacterial effect of the Cu NPs may be the multiple todxic effects of Cu such as generation of reactive oxygen species, lipid peroxidation, protein oxidation, and DNA degradation in the *E. coli* cells [7, 8].

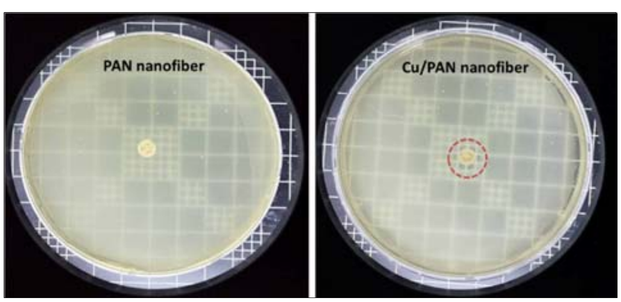

Fig 5: Antibacterial performance of different samples against *E. coli* bacteria.

4. Conclusions

Cu NPs /PAN colloidal soloution can be easily electrospun into smooth nanofibers. The morphological characterization showed that the Cu NPs were incorporated inside the PAN nanofibers. The as-synthesized hybrid composite fibers showed remarkable antibacterial activity against *E. coli* bacteria. This observation supports the applicability of the Cu/PAN nanofiber mats useful for various applications as a broad range of antibiotics.

5. References

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