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# Preparation and Characterization of Ge/$SiO_2$ Nanocomposite Fibers

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## ABSTRACT

This research investigates incorporating nanoscale germanium (Ge) and silica ($SiO_2$) particles into nanofibrous structures via electrospinning for biomedical applications. Composite fibers containing Ge and $SiO_2$ were fabricated at various concentrations of Ge and/or $SiO_2$ and layered on polypropylene nonwoven. The morphological properties of Ge/$SiO_2$/PVA nanocomposite fibers were characterized using a field-emission scanning electron microscope and a transmission electron microscope. Layered fabric systems with electrospun PVA nanofibers containing Ge and $SiO_2$ nanoparticles showed significant increases in far-infrared emissivity and emissive power. The system exhibited a 99.9% bacterial reduction against both *Staphylococcus aureus* and *Escherichia coli*, and showed a 34.8% reduction in *Klebsiella pneumoniae.*

## 1. INTRODUCTION

Electrospinning is a fiber forming technique that uses electrostatic force to produce polymer fibers with diameters in the nanometer to micron range. One advantage of electrospinning is that additional functionalities can be imparted to polymer nanofibers by simultaneous spinning of the polymer material with a functional material (Zeng et al. 2006). The large surface areas of nanofibrous structure can maximize the functionalities as compared with those of conventional fibers with typical diameters of several microns. Furthermore, nanofibrous structures have numerous pores with pore dimensions that are small enough to prevent bacteria from entering but large enough for moisture vapor to diffuse through; thus they have been proposed for biomedical applications such as wound dressing, skin masks for therapeutical or medical treatments. Recently, germanium and ceramic powders have been applied to textile structures to exhibit far-infrared radiation effect (Fijino and Kabaya 1987, Li et al. 2007). It has been reported that infrared rays having a long wavelength such as far-infrared rays enhance blood circulation and metabolism and promote recovery of fatigue of muscles (Inoué and Honda 1986, Ise et al. 1987). In this study, nanocomposite fibers containing germanium and silica nanoparticles were fabricated by electrospinning to impart far-infrared radiation effect. Far-infrared radiation properties of Ge/$SiO_2$/PVA nanocomposite fiber webs with various concentrations of Ge and/or $SiO_2$ were

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assessed. In addition, antimicrobial activity of Ge/$SiO_2$/PVA nanocomposite fibers was examined.

## 2. EXPERIMENTAL

Poly(vinyl alcohol) (>99% hydrolyzed, Mw=89,000-98,000, Sigma Aldrich Co., USA) was used as a polymer system, and distilled water was used as a solvent. Water-based nanosized colloidal germanium with a mean particle size under 50 nm at 5,000 ppm was supplied by Nanopoly Co. (Korea). Water-based nanosized colloidal $SiO_2$ with a mean particle size under 70 nm at 200,000 ppm was also supplied by Nanopoly Co. (Korea).

Solutions of eleven weight percent of PVA with various weight percent of Ge and $SiO_2$ nanoparticles were prepared. Electrospinning was performed in a vertical electrospinning setup under a variety of conditions to find an optimum spinning condition. To form a layered fabric system, a 100% polypropylene, light-weight, highly porous spunbonded nonwoven was used as a substrate.

Morphology of electrospun nanocomposite fibers was examined using a field emission scanning electron microscope (FE-SEM) and a transmission electron microscope (TEM). Electrospun PVA nanocomposite fiber webs were heat-treated to stabilize PVA structures against dissolution in water.

Fourier Transform Infrared Spectrophotometer (FT-IR) was used to measure emissivity and emissive power of Ge/$SiO_2$/PVA nanocomposite fibers. Far-infrared emissive power was determined in the range 5-20μm of wavelength at 37°C. Emissivity was measured from the relative value assuming that the emissivity of the black body is 1. Antibacterial properties of the layered fabric systems were evaluated quantitatively according to ASTM E 2149-01 by measuring the bacterial reductions of *Staphylococcus aureus* (ATCC 6538, Gram-positive bacterium), *Klebsiella pneumoniae* (ATCC 4532, Gram-negative bacterium), and *Escherichia Coli* (ATCC 25922).

## 3. RESULTS AND DISCUSSION

Germanium and silica nanoparticles were incorporated into PVA fibers by the inclusion of the nanoparticles in the electrospinning dope. Nanocomposite fibers with diameters ranging from 200 to 300 nm were obtained using electrospinning. The fibers were electrospun with a 30-gauge needle at a feed rate of 0.2 mL/hr, a voltage of 15-17 kV and a collecting distance of 13 cm. Nanoscale Ge and $SiO_2$ particles were observed inside the nanocomposite fiber as well as on the surface of the fiber.

Nanofibrous membranes containing Ge and $SiO_2$ were heat-treated at 210°C for 30 min. To confirm that the heat treatment stabilized the PVA nanocomposite fibers against dissolution in water, both heat-treated and untreated nanofiber webs were immersed in water at 18°C for 1 hr, and then the morphology of the membranes was examined using a FE-SEM. An untreated PVA nanofibrous membrane lost its fibrous structure after exposure to an aqueous environment, whereas the fibrous structure of the heat-treated Ge/$SiO_2$/PVA nanocomposite fibers was maintained after immersion in water.

Layered fabric systems with electrospun nanocomposite fiber webs were developed at various concentrations of Ge and/or $SiO_2$ to impart far-infrared radiation effect. An electrospun PVA nanofibrous membrane without nanoparticles showed a far-infrared emissivity of 0.881 and an emissive power of $3.40\times10^2$ W/m$^2$. An emissivity and emissive power of the nanofibers electrospun from 11 wt% PVA solutions containing 0.5 wt% Ge were 0.887 and $3.42\times10^2$ W/m$^2$. Nanocomposite fibers electrospun from 11 wt% PVA solutions containing 1 wt% $SiO_2$ showed a far-infrared emissivity of 0.890 and an emissive power of $3.43\times10^2$ W/m$^2$. Multi-component composite fibers containing both Ge and $SiO_2$ were also developed. Nanocomposite fibers electrospun from 11 wt% PVA solutions containing 0.5 wt% Ge and 1 wt% $SiO_2$ showed a far-infrared emissivity of 0.891 and an emissive power of $3.44\times10^2$ W/m$^2$.

We also examined antimicrobial properties of Ge/$SiO_2$/PVA nanocomposite fibers electrospun from 11 wt% PVA solutions containing 0.5 wt% Ge and 1 wt% $SiO_2$. The antimicrobial properties were evaluated quantitatively, and three microorganisms were used for assessment: *Staphylococcus aureus*, *Klebsiella pneumoniae* and *Escherichia Coli*. Layered fabric systems with the electrospun Ge/$SiO_2$/PVA nanocomposite fiber webs exhibited a 99.9% bacterial reduction against both *Staphylococcus aureus* and *Escherichia Coli*. On the other hand, the same system showed a 34.8% reduction in *Klebsiella pneumoniae*. The lower reduction of Gram-negative *Klebsiella pneumoniae* than Gram-positive *Staphylococcus aureus* and *Escherichia Coli* might be due to that Gram-negative bacteria have more complex cell wall structures than Gram-positive bacteria (Fu et al. 2005). The additional outer membrane of Gram-negative bacteria may protect these organisms, resulting in lower reduction of Gram-negative *Klebsiella pneumoniae*.

## 4. CONCLUSION

Layered fabric systems with nanofibrous membranes containing Ge and $SiO_2$ were developed at various concentrations of Ge and/or $SiO_2$ to impart far-infrared radiation effect and antibacterial property via electrospinning. Multi-component composite fibers electrospun from 11 wt% PVA solutions containing 0.5 wt% Ge and 1 wt% $SiO_2$ exhibited a far-infrared emissivity of 0.891 and an emissive power of $3.44\times10^2$ W/m$^2$. The same system exhibited a 99.9% bacterial reduction against both *Staphylococcus aureus* and *Escherichia Coli*. The results showed that far-infrared radiation effect and antibacterial properties were successfully imparted to PVA nanofibers by electrospinning mixtures of polymers and functional nanoparticles. The results indicate that PVA nanofibrous membranes containing Ge and $SiO_2$ have high potential for medical and healthcare applications.

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