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Short communication

Novel preparation and characterization of human hair-based nanofibers using electrospinning process

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ABSTRACT

Human hair-based biocomposite nanofibers (NFs) have been fabricated by an electrospinning technique. Aqueous keratin extracted from human hair was successfully blended with poly(vinyl alcohol) (PVA). The focus here is on transforming into keratin/PVA nanofibrous membranes and insoluble property of electrospun NFs. The resulting hair-based NFs were characterized using Fourier transform infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD), differential scanning colorimetry (DSC), and thermogravimetric analysis (TGA). Toward the potential use of these NFs after cross-linking with various weight fractions of glyoxal, its physicochemical properties, such as morphology, mechanical strength, crystallinity, and chemical structure were investigated. Keratin/PVA ratio of 2/1 NFs with 6 wt%-glyoxal showed good uniformity in fiber morphology and suitable mechanical properties, and excellent antibacterial activity providing a potential application of hair-based NFs in biomedical field.

1. Introduction

One problem we encounter in our everyday lives due to technological development is the contamination of our environment. Although mankind benefited from technological innovation during this phase of history, even after the Industrial Revolution, we are still suffering from a by-product of industrialization, the pollution of nature. Nowadays, we have attempted to use natural resources which can protect the nature. One of the most environmentally-friendly materials is keratin. Keratin is three-dimensional mesh structures associated structural fibrous proteins, moreover the hard keratins found in hair, wool, feather, nails, and horns have a sulphur content >3 wt% [1]. Keratins are composed of quite a number of disulfide bonds which have cysteine-rich structural proteins and significant mechanical properties due to its hard fibrous structure. Because of its insolubility in polar solvents like water, weak acids and bases, as well as non-polar solvents, limited numbers of methods are available for the extraction of keratin [2–4]. More than 300,000 ton of protein-rich hair wastes produced around the world each year. Hair wastes can be usable for regenerating water-soluble compounds through the reduction of disulfide bonds [2]. To date, most of reports provide the broad overview of keratin based material obtained in various forms for biomedical applications [5] or for the fabrication of electrospun blend NFs by mixing with other polymers [6].

Electrospinning is a well-known and versatile technique to fabricate micro and nanofibers with high porosity and many biomedical applications such as wound dressing, tissue engineering scaffolds, and drug release [7]. A number of researches have been studied on the function and composition of biomaterials produced by keratins [8–10]. Here we present the fabrication of human hair-based keratin/PVA NFs by electrospinning. In our previous work, we have successfully prepared the biocompatible hydrogels made by human hair [11]. Because of its low molecular weight (65–11 kDa) and poor mechanical properties [12], regenerated keratin is so difficult to handle that poly(vinyl alcohol) (PVA) was blended with keratin aqueous solution.

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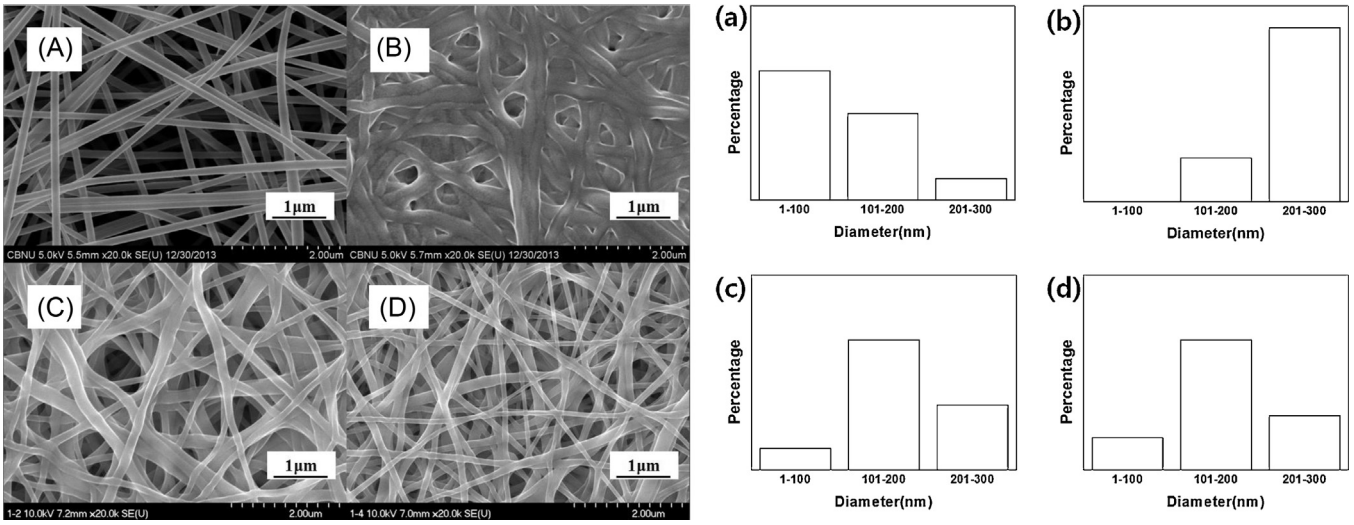

Fig. 1. SEM micrographs of (A, a) human hair and keratin NFs blended with PVA (keratin/PVA = 2/1) after dipping in water with various weight fractions of glyoxal: (B, b) 3 wt%, (C, c) 6 wt%, and (D, d) 12 wt%.

2. Experimental

Urea, sodium disulfite ($Na_2S_2O_5$), sodium dodecyl sulfate (SDS), acetone (CH_3COCH_3), ethanol (C_2H_5OH), glyoxal solution (40 wt% in H_2O), and PVA (Mw = 85,000–124,000 g/mol, 87–89% hydrolyzed) were purchased from the Sigma Aldrich, Co. (St. Louis, USA). Human hair was obtained from local hair salons for free and double-distilled water was used for creating aqueous solutions. Keratin was extracted by sulfitolysis [13]. The cleaned and dried hair (150 g) was placed into 1.5 L of an aqueous solution that contained 8 M urea, 75 g of SDS and 150 g of $Na_2S_2O_5$ and heated to 100 °C, shaken for 30 min and then cooled in a water bath at 30 °C. The filtrate was dialyzed against 15 L of water that contained 0.1 wt% $Na_2S_2O_5$ using cellulose tubing (molecular-weight cutoff of 12,000 Da) for 3 days. (In our previous report [11], the protein concentration in the extracted keratin solution was 5.0 wt% on average). Then 10 wt% PVA was mixed with different weight ratios (3:1, 2:1, 1:1, and 1:2). Glyoxal solution was added as a cross-linker (3, 6, 12 wt%) while the pH of the system was adjusted at 2–3 by phosphoric acid. The solution was electrospun at 18 kV by maintaining a tip-to-collector distance of 16 cm. As-spun hair/PVA NFs were collected in Teflon paper, put into the oven under alcohol vapor at 50 °C for 24 h, and then cured at 120 °C for 10 min.

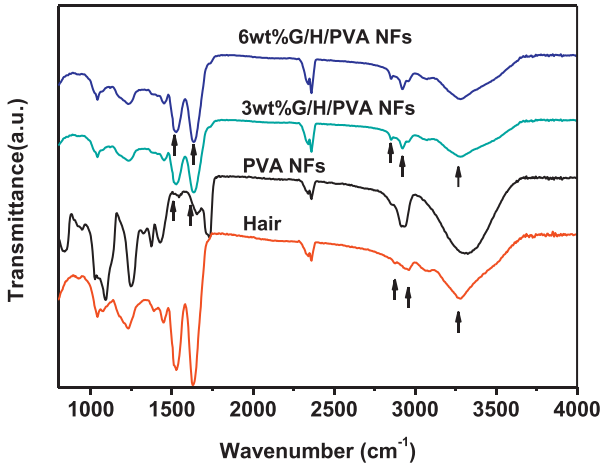

Fig. 2. FT-IR spectra of human hair, PVA NFs, and keratin NFs blended with PVA (keratin/PVA = 2/1) in the presence of 3 wt%- and 6 wt%-glyoxal.

The chemical structures were confirmed by FT-IR (Varian 1000 Scimitar series) and the surface morphologies were determined by SEM (JSM-5900JEOL Co.). Information about the crystallinity was obtained by XRD (Rigaku Co.) with Cu Kα (λ = 1.540 Å) radiation. DSC (Perkin-Elmer, USA) and TGA (Perkin-Elmer, USA) were carried out under nitrogen ambient with a flow rate of 20 mL/min at a scanning rate of 10 °C/min. Mechanical properties were measured by a universal testing machine (UTM, AG-5000G, Shimadzu, Japan) according to ASTM D638. The disk diffusion susceptibility test for *Staphylococcus aureus* and *Escherichia coli* was performed on the MHA plate at the incubation temperature of 37 °C. The bacterial activity of the nanofibers produced was tested using growth inhibition studies against Gram-positive and Gram-negative bacteria. The pathogens tested in this study were *S. aureus* ATCC 29231 and *E. coli* ATCC 52922 purchased from the American Type Culture Collection (ATCC). The zone of inhibition was observed after 24 h of incubation and the diameter of the zone was measured.

3. Results and discussion

Fig. 1 presents the SEM micrograph and diameters of the keratin/PVA blend NFs varying weight fractions of glyoxal. In this work, the protein concentration in the extracted keratin solution was 5.0 wt% on average using the BCA quantitation method [11]. When 10 wt% PVA was mixed with different weight ratios (3:1, 2:1, 1:1, and 1:2) with keratin solution, 2:1, 1:1, and 1:2 were successfully electrospun. Therefore we focus on the keratin NFs blended with PVA (H/PVA NFs, keratin/PVA = 2/1) with various weight fractions of glyoxal as a cross-linker due to the high solubility of H/PVA NFs in water. PVA is a water-soluble polyhydroxy polymer. Even though PVA has excellent properties, its applications are limited due to its high hydrophilicity [14]. Thus PVA NFs are cross-linked to improve their water resistance [15]. In Fig. 1, H/PVA NFs and diameters of all fibers were distributed 100–300 nm. As seen in (B, b), (C, c) and (D, d), H/PVA NFs with 3 wt%-glyoxal (3 wt%G/H/PVA) were swollen while H/PVA NFs with 6 wt%-glyoxal (6 wt%G/H/PVA) and 12 wt%-glyoxal (12 wt%G/H/PVA) did not show any significant differences after dipping in water, whereas the fiber diameters of 6 wt%G/H/PVA are slightly bigger than those of 12 wt%G/H/PVA after swelling process. Although not fully understood, this result suggests that the glyoxal influence significantly the fabrication of insoluble NFs as a cross-linker during electrospinning. However, homogeneous and bead-free nanofibers