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Short communication

Inductive heating of electrospun Fe_2O_3/polyurethane composite mat under high-frequency magnetic field

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Abstract

This study reports on the effect of high-frequency magnetic field on the heating of electrospun polyurethane (PU) nanofibers decorated with Fe_2O_3 nanoparticles (NPs). The morphological and thermal properties were checked by several characterization techniques. Here, the effect of Fe_2O_3 NP concentration and intensity of magnetic field on the inductive heating of the composite mat were investigated. Fe_2O_3 nanoparticles were confirmed to be embedded on/in the nanofibers and their presence has increased the thermal stability of the composite nanofibers compared to the neat PU nanofibers. The composite nanofibers under high-frequency magnetic field showed a temperature increase of 3–17% with respect to the neat PU nanofibers depending on the Fe_2O_3 NP concentration and magnetic field strength. The present results suggest the potential use of electrospun nanofibers as good substrate for magnetic nanoparticles, and when exposed to external magnetic field, can induce heating that may be useful for localized hyperthermia application.

Keywords: Hyperthermia; Fe_2O_3; Electrospinning; Magnetic field; Nanoparticles

1. Introduction

In recent years, research on magnetic nanoparticle-based materials has been increasing for different biomedical and cancer therapy applications [1,2]. Magnetic nanoparticles (NPs) show promise in their use for magnetic hyperthermia application, wherein ferromagnetic or superparamagnetic NPs are introduced onto or in close proximity with tumor tissue and are heated up by external magnetic field. In hyperthermia therapy, the tumor is heated in the temperature range of 41–47 °C, causing death of the cancerous cells, but not the healthy cells [3,4]. Several nanoparticles have been investigated for their inductive heating properties, such as $CoFe_2O_4$, $MnFe_2O_4$, Fe_2O_3, Fe_3O_4, etc. [5–8]. However, only very few studies have been conducted on the heating of magnetic NPs that are incorporated in a polymeric nanofibrous matrix [9,10].

For in vivo application, polymeric nanofibers with magnetic NPs can find potential application as cover for non-vascular stents. A non-vascular stent is a man-made wire-mesh that is inserted inside the body to create an artificial passageway or support structure, and restore flow conditions in a blocked hollow lumen organs such as in esophagus, biliary areas, lungs, urethra, and colon with minimal invasion [11]. A non-vascular stent can have bare or covered design. However, the use of bare stents has an issue of re-occlusion of passageway

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due to tumor in-growth. So that, film-coated (i.e., covered) stents are now increasingly being developed in order to prevent restenosis due to the proliferation of cancer cells. Many studies [12–15] have been carried out on the use of polymeric films as cover for nonvascular stents in order to inhibit the overgrowth of smooth muscles, such as malignant tissues in bile duct cancer or esophageal cancer, which infiltrate the lumen and causes blockage in the passageway. One simple yet powerful technique to cover nonvascular stents is with the use of electrospinning technique. Electrospinning is an efficient and easy technique for the fabrication of polymeric nanofibers, which has many applications such as in tissue and biomedical engineering, filtration, clothing and textiles, and composite materials. Only a few groups have researched on the use of electrospun nanofibers as substrate material for magnetic nanoparticles to be used as cover for nonvascular stents that has potential for magnetic hyperthermia applications. Thus, this paper presents a preliminary study on the magnetic hyperthermia application potential of electrospun nanofibers decorated with Fe_2O_3 nanoparticles. Fe_2O_3 has been used in many catalytic and biomedical applications [16].

In this paper, we incorporated Fe_2O_3 nanoparticles in/on polyurethane nanofibers by electrospinning and exposed the composite materials to alternating magnetic field for inductive heating. The heating effect depends on the size as well as the frequency and amplitude of the applied alternating magnetic field [17]. Here, we investigated the effect of nanoparticle concentration and intensity of magnetic field on the inductive heating of Fe_2O_3/PU composite nanofiber.

2. Materials and method

2.1. Materials

Fe_2O_3 nanoparticles (Iron (III) oxide, red) were purchased from Samchun Chemicals (Korea). High molecular weight polyester grade thermoplastic polyurethane pellets (Estane Skythane X595A-11) were bought from Lubrizol Advanced Materials, Inc., USA. N,N dimethylformamide (DMF) and methyl ethyl ketone/2-butanone (MEK, extra pure) were purchased from Showa Chemical Co., Ltd., Japan and Junsei, Japan, respectively, and were used as received.

2.2. Electrospinning of Fe_2O_3/PU nanofibers

Neat PU solution was prepared by dissolving polyurethane pellets (10 wt%) in DMF/MEK (50/50, wt:wt%) solvent system by magnetic stirring. To prepare Fe_2O_3/PU solution, appropriate amounts of Fe_2O_3 NPs were mixed in the PU solution by magnetic stirring. The concentration of Fe_2O_3 NPs (i.e., 10 wt%, 20 wt%, and 30 wt%) was based from the weight of PU.

Electrospinning was carried out using the setup used in our previous study [18]. It was composed of a high-voltage power supply, a cylindrical aluminum collector covered with polyethylene sheet, 10-ml plastic syringes, and metal capillary (nozzle) with $d_i = 0.51$ mm (21 G), and a syringe pump. Solutions were collected in the plastic syringe connected to nozzle holder. The syringe was fixed in a syringe pump set at a constant feed rate of 0.3 ml/h. The nozzle tip was connected to a power supply at a voltage of 20 kV. The grounded cylindrical collector rotating at 835 rpm was placed 200 mm away from the nozzle tip. After electrospinning, the nanofibrous membrane was dried in an oven at 80 °C for 48 h to remove the residual solvents, and then characterizations and measurements followed.

2.3. Characterization

The surface morphological structure of the samples was characterized by field emission scanning electron microscopy (FESEM, Hitachi S-4800) and transmission electron microscopy (TEM, JEM-2010, JEOL). The elemental composition of the surface was checked using an energy dispersive spectrometer (EDS) connected to SEM. The TEM samples of NPs were prepared by immersing a copper grid mesh into a Fe_2O_3 sample dispersed in alcohol for a few seconds and air-dried. FTIR spectra of the samples were obtained using a Paragon 1000 Spectrometer (Perkin Elmer). The signal resolution of the FTIR was 1 cm^{-1} and a minimum of 16 scans was obtained and averaged within the range of 400–4000 cm^{-1}. X-ray powder diffraction (XRD) analysis was carried out by a Rigaku X-ray diffractometer (Cu Kα, $\lambda = 1.54059$ Å) over Bragg angles ranging from 20 to 65°. The thermal degradation of the nanofibers was measured using TGA (Q50, TA Instruments) at a heating rate of 10 °C/min from room temperature to 600 °C.

2.4. Magnetic heating experiment

Magnetically induced heating treatment was performed with a magnetic hyperthermia system (OSH-120, Osung, Korea). The water-cooled induction coil is made of copper, with an inner diameter of 60 mm. The induction coil is driven by the inverting power supply and produces an AC magnetic field strength of 1 kA m^{-1} (approximately 12.57 Oe) and a frequency of 368 kHz [19]. Nanofibrous membrane samples (i.e., neat and composite nanofibers) in cylindrical form were placed in between the copper coil, and type-T thermocouples were attached to the surface of the nanofibers. A real-time data acquisition system (NI-DAQ^R, National instrument, USA) was used to automatically record the temperature change of the membranes through LabVIEW program. Before each inductive heating test, calibration and stabilization of temperature for 10 min was always conducted.

3. Results and discussion

3.1. Characterization

Fig. 1 shows the FESEM images of the fabricated neat PU and Fe_2O_3/PU composite nanofibers. Neat PU nanofibers (Fig. 1a) showed smooth and bead-free nanofibers with high porosity and wide fiber diameter distribution. The incorporation of Fe_2O_3 nanoparticles (Fig. 1b–d) did not change the

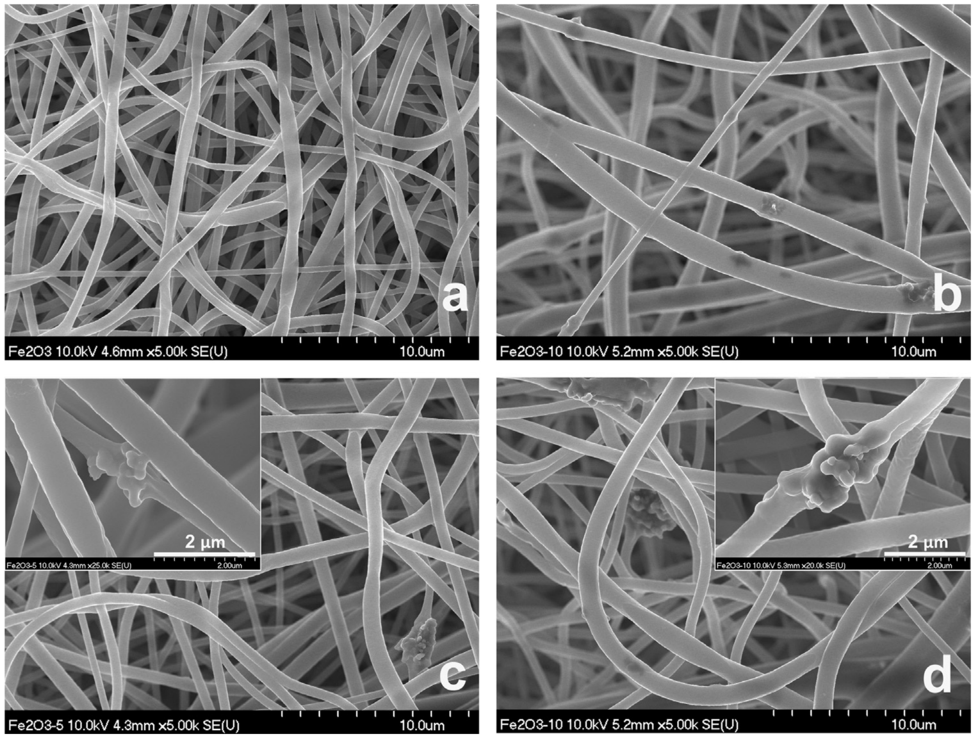

Fig. 1. FESEM images of the (a) neat PU and Fe_2O_3/PU composite nanofibers with Fe_2O_3 NP concentration of (b) 10 wt%, (c) 20 wt%, and (d) 30 wt%.

geometry and structure of the fibers of the composite nanofibers compared to neat PU. However, it can be seen that beads were formed on the surface of PU nanofibers after nanoparticle incorporation. Fe_2O_3 NPs were decorated in/on the PU nanofibers, but more agglomeration of particles are noticed when the NP concentration was increased (Fig. 1c and d). It should be noted that no surfactant for dispersion of Fe_2O_3 NPs was used, so that due to the high surface-to-volume ratio of Fe_2O_3 NPs, they tend to agglomerate at higher content. As checked, by EDS, the particles were confirmed to be Fe_2O_3 showing Fe peaks in the EDS spectra of 30 wt% Fe_2O_3/PU composite (Fig. 2). Due to the very small dimension of Fe_2O_3 nanoparticles (diameter~150–250 nm as checked by TEM), electrospun nanofibers could provide excellent substrate material that has very high surface-area-to-volume ratio and high porosity, is light weight and is mechanically-flexible. Compared to the use of solid films as substrate, nanofibers provide much higher surface area, thus giving more reactive sites. Through proper dispersion and immobilization of Fe_2O_3 NPs in/on the nanofibers, we can maximize their magnetic heating effect upon exposure to magnetic fields. From Fig. 1, it could be seen that the NPs are embedded firmly in/on the nanofibers, thus preventing their possible migration when used as cover for stent. This also provides reusability of the composite material. Several researchers [20–25] have utilized electrospun nanofibers as substrate material of nanoparticles to provide added functionalities for biomedical applications.

Fig. 3 shows the crystalline structures of the electrospun nanofibers by XRD analysis. One or two broad peaks in the XRD results indicate a typical pattern for a low crystalline material, which suggests that the present PU nanofiber has low crystallinity (see Fig. 3a). However, many new peaks are observed for the composite nanofiber, which clearly indicates the presence of Fe_2O_3 nanoparticles (Fig. 3b). The existence of the several new peaks is attributed to the peaks of Fe_2O_3 nanoparticles as seen in Fig. 3c. To characterize the molecular nature of a material, Fourier transform infrared (FT-IR) spectra of the samples were taken (see Fig. 4). The absorption peaks of neat PU (Fig. 4a) at 3328 cm^{-1}, 2923 cm^{-1}, 1700 cm^{-1} to 1732 cm^{-1}, 1532 cm^{-1}, 1063 cm^{-1}, and 762 cm^{-1} are assigned to N–H stretching, CH_2 stretching, C–H asymmetrical flexing vibration, $>C=O$ stretching vibrations, amide II band, C–O stretching, and CH_2 rocking, respectively [26–28]. When Fe_2O_3 nanoparticles were incorporated, no pronounced changes in absorption peaks were observed compared to neat PU. However, we observed a slight shift at 1063 cm^{-1} for all composite nanofibers indicating an increased hydrogen bonding between PU and Fe_2O_3 NPs.

The TGA weight loss vs. temperature plot of neat PU and composite nanofibers is shown in Fig. 5. The onset decomposition temperature of the composite samples (~280 °C) was lesser than that of neat PU (~305 °C). The neat PU (Fig. 5a) showed a steady degradation from 305 °C to 450 °C and its complete degradation occured at 500 °C. On the other hand, the composite nanofibers (Fig. 5b–d) showed better thermal stability than neat PU with increasing Fe_2O_3 NP content. The better thermal stability of the composite nanofibers is attributed to the successful dispersion of Fe_2O_3 NPs, which has good thermal stability in the nanofibers and the interaction between PU and the Fe_2O_3 NPs.

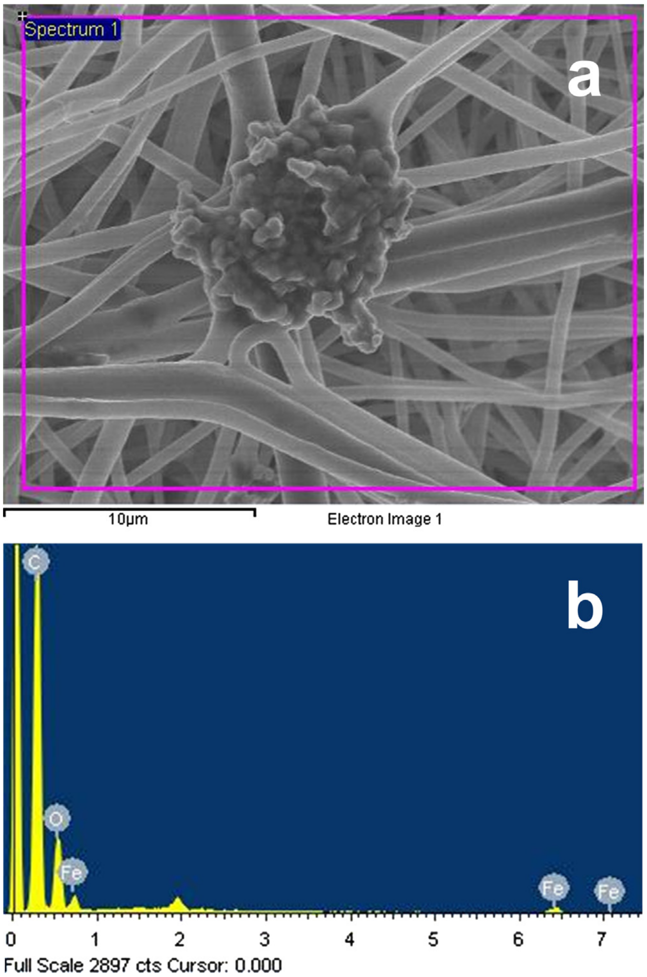

Fig. 2. SEM image and EDS spectra of 30 wt% Fe_2O_3/PU composite nanofibers.

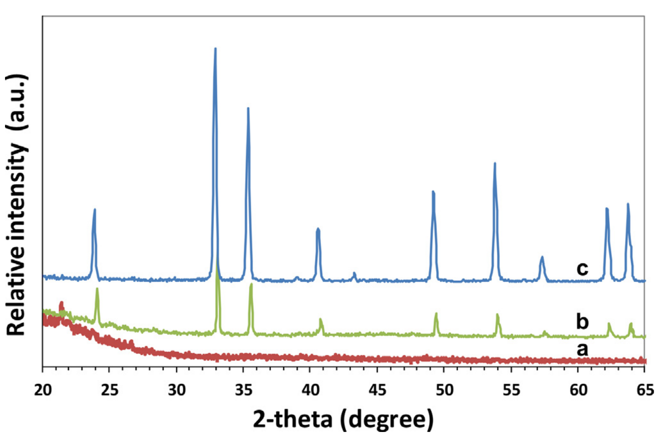

Fig. 3. XRD spectra of (a) neat PU, (b) 30 wt% Fe_2O_3/PU composite nanofibers, and (c) Fe_2O_3 nanoparticles.

3.2. Magnetic heating

The inductive heating of magnetic particles is primarily due to magnetic losses associated with the magnetization/demagnetization cycling. Magnetic iron oxide nanoparticles, are good candidates for hyperthermia tumor therapy due to their excellent high-frequency

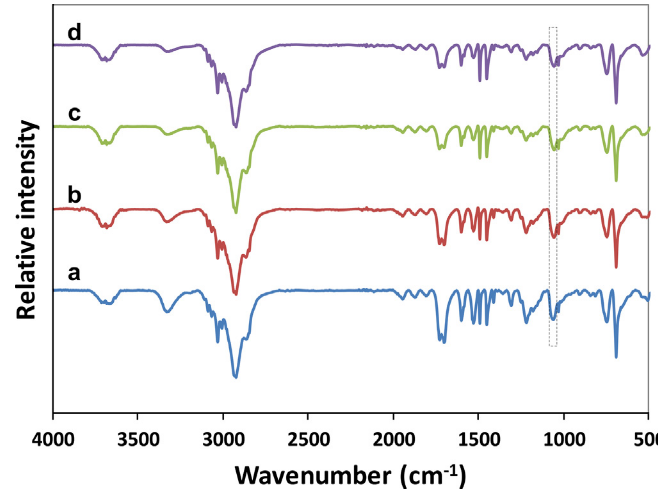

Fig. 4. FTIR spectra of (a) neat PU and Fe_2O_3/PU composite nanofibers with Fe_2O_3 NP concentration of (b) 10 wt%, (c) 20 wt%, and (d) 30 wt%.

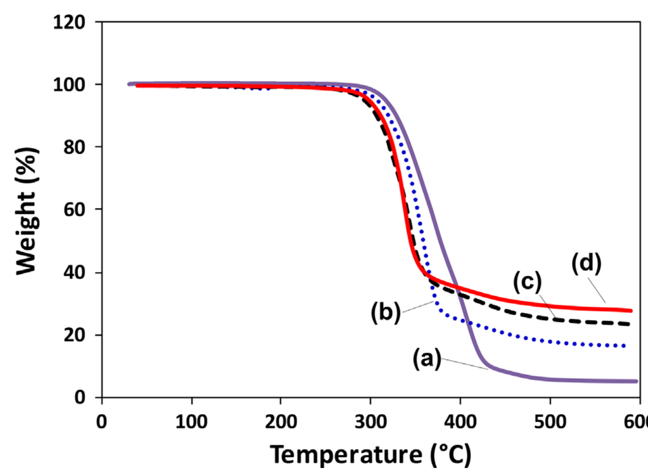

Fig. 5. Thermogravimetric curves of (a) neat PU and Fe_2O_3/PU composite nanofibers with Fe_2O_3 NP concentration of (b) 10 wt%, (c) 20 wt%, and (d) 30 wt%.

response and high resistivity property, especially the possible large magnetic loss and high heating ability. In this study, we incorporated large amount of Fe_2O_3 nanoparticles to the PU nanofibers (i.e., 10 wt%, 20 wt%, and 30 wt%) so as to enable a better magnetic hyperthermia effect [29,30]. Fig. 6 shows the average temperature vs. magnetic field strength of the present samples after 10 min test. From Fig. 6, it can be seen that the composite nanofibers with 20 and 30 wt% Fe_2O_3 showed better heating effect compared to neat PU nanofiber. Increasing the magnetic flux also obtained the same increasing trend for all samples. At 20% magnetic flux, the composite nanofibers were only slightly higher by 1–1.5 °C compared to neat PU after 10 min of inductive heating. When the magnetic flux was increased to 40%, the composite nanofibers were 5–6.5 °C higher than the neat PU, obtaining up to 44 °C after 10 min heating or an increase of up to 17%. The highest amount of Fe_2O_3 NPs in the PU nanofiber (i.e., Fig. 6c) showed consistently higher heat effect compared to others. This is primarily because the heat source was the Fe_2O_3 NPs, thus in a given time the more the amount of the

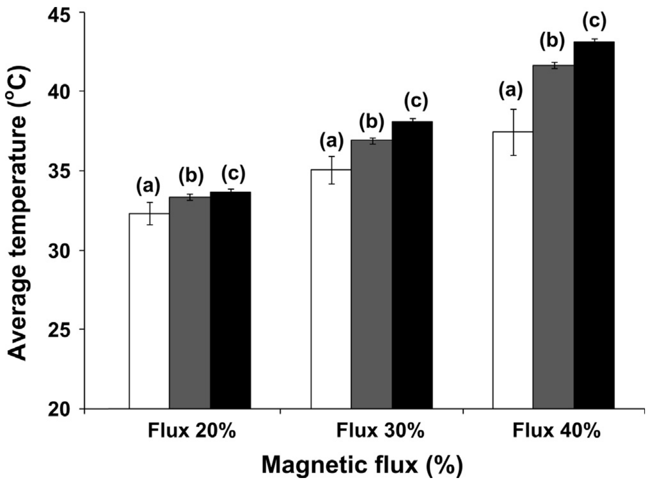

Fig. 6. Inductive heating of (a) neat PU and composite nanofibers with (b) 20 wt% and (c) 30 wt% of Fe_2O_3 NPs at different magnetic field strength.

magnetic NPs the more the heat could be generated [2]. Furthermore, controlling the magnetic flux could also result to more or faster heating time. Khot et al. [31] reported an increasing temperature with the increase of field amplitude and concentration of $MgFe_2O_4$ nanoparticles under alternating current (AC) magnetic field. In the present study, the heating of Fe_2O_3/PU nanofibers under high-frequency magnetic field could be attributed to both hysteresis loss, which refers to the loss of due to irreversible magnetization process in AC field, and residual loss or relaxation loss, which refers to various relaxation effects of magnetization in magnetic field as extensively explained by Le'vy et al. [32].

4. Conclusions

In this study, we have incorporated Fe_2O_3 nanoparticles into polymeric nanofibrous substrate by one-step electrospinning process. Morphological and surface characterizations have confirmed the presence of Fe_2O_3 nanoparticles that are well-distributed in/on PU nanofibers. The composite nanofibers produced better thermal stability compared to neat PU nanofibers. Through the application of external magnetic field, the Fe_2O_3/PU composite nanofibers provided better heating effect, with higher heating when more Fe_2O_3 nanoparticles are incorporated. The results also indicated the possible tuning of inductive heating of the composite nanofibers by controlling the Fe_2O_3 NP concentration and the applied magnetic flux. The present one-step fabrication of composite nanofibrous mat and its magnetic heating capability could find potential application in hyperthermia therapy.

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