Study on Morphology and Size Distribution of Electrospun NiO-GDC Composite Nanofibers

Raziyeh Ghelich, Mansoor Keyanpour Rad, Amir Ali Youzbashi

Materials and Energy Research Center (MERC), Tehran, IRAN

Correspondence to:
Raziyeh Ghelich email: r.ghelich@yahoo.com

ABSTRACT

Nickel oxide-gadolinium doped ceria (NiO–$Ce_{0.8}Gd_{0.2}O_{1.9}$) deserves special attention because of its high ionic conductivity. The nanocomposite fibers of this material are suitable as anode materials for low-temperature SOFCs. The composition and morphology of the nanofibers which prepared via electrospinning method were characterized by SEM, TEM and EDS. It was noticed that the fiber diameters gradually decreased from 190±77 nm to 75±27 nm by increasing the calcination temperature from 800 ˚C to 1000 ˚C. Further, the influence of the different parameters such as concentration of the polymeric pre-spinnable solution (CPS), concentration of the cationic solution (CCS), the applied voltage, tip-target distance, feed rate, diameter of the needle and the calcination temperature on the morphology of the fibers were investigated.

Keywords: NiO-GDC; Nanofiber; Composite material; Morphology; Size distribution

INTRODUCTION

One-dimensional (1D) materials such as nanofibers, nanotubes and nanocomposites have been widely investigated because of their applications in gas sensors, super-capacitors, field-emission microelectronic devices, catalysts, lithium-ion batteries and solid oxide fuel cells [1, 2]. There are several methods for fabrication of nanofibers, such as making use of chemical, thermal and electrostatic principles. The electrospinning process has attracted much attention for the production of composite nanofibers, as it can produce them with diameters in the range from several micrometers down to nanometers, depending on the polymer solution and processing conditions [3, 4]. Electrospinning is simple, inexpensive, versatile, and effective technique to make nanofibers with high surface-to volume ratio, and high porosity depending on the solution properties, equipment and processing variables [5, 6]. One of the most important quantities related with electrospinning is the fiber diameter. Systematic investigations on the effect of electrospinning parameters on the diameter and the morphology of the fibers have been reported by several researchers [7]. Wide range of parameters can play important roles in obtaining the desired nanofiber size and microstructure, namely solution viscosity, voltage, feed rate, solution conductivity, capillary-to-collector distance and capillary tube [8, 9]. Great efforts have been made to study the effects of processing parameters on the structure and morphology of electrospun fibers [10, 11]. Recently a new class of nanocomposites materials based on organic and inorganic species combined at a nanoscale has obtained more attention [12, 13]. The properties of these nanocomposites are strongly depended on the size, dispersion efficiency and morphology of the sample [14]. For the first time Larsen et al. [15] combined electrospinning with sol–gel method to prepare nanofibers made from inorganic oxides and hybrid (organic/inorganic) materials with diameters in the micrometer and submicrometer range. Many applications of electrospun nanofibers could be greatly improved by increasing the surface area and porosity of the fibers. So producing porous nanofibers in a versatile and inexpensive way was tried by a number of researchers [16, 17].

Materials based on nanosized cerium dioxide (CeO_2) have many potential applications in high-storage capacitor devices, buffer layers for conductors, fuel cells, polishing materials, and optical devices [18, 19]. The synthesis of CeO_2 nanofibers with a diameter of 50–150 nm after calcinations of as-spun polyvinyl alcohol/cerium nitrate composite was reported by other researchers [20]. The NiO-GDC material is a potential candidate for an anode material for the low-temperature SOFCs [21]. A maximum conductivity was usually observed at a doping concentration between 10 and 20% [22].

In this work, by applying the electrospinning method to prepare high quality NiO-GDC nanofibers, the effects of instrument parameters, such as applied voltage, tip–target distance, flow rate, calcinations temperature and also solution parameters, such as concentration of the polymeric pre-spinnable solution (CPS) and concentration of the cationic solution (CCS), on the morphology and diameter of the electrospun fibers were systematically evaluated.

EXPERIMENTAL

The NiO–$Ce_{0.8}Gd_{0.2}O_{1.9}$ (NiO:GDC=50:50 mass%) nanocomposite fibers were synthesized by electrospinning method and characterized in our previous work [23]. 50 wt.% NiO was chosen for attaining 39 vol.% Ni in the final cermets, this amount was adequate for obtaining good electrical conductivity in the anode of SOFCs [24]. Briefly, the nitrates of Ce, Gd and Ni were dissolved into adequate amount of distilled water to form homogeneous solution. For the electrospinning experiment, PVA solution was slowly dropped into the cationic solution of the starting materials, to form a homogeneous viscous gel by heating the gel at 80˚C. For the electrospinning process, the above solution was supplied through a plastic syringe attached to a metal needle with a special feed rate via a syringe pump. A high-voltage power supply (Glassman High Voltage, USA); capable of generating voltages up to 60 kV, was used as the source of the electric field. A flat aluminum foil used to collect of the as-spun PVA/NiO-GDC fibers. The polymeric nanofiber webs were air dried for 12 h and then subjected to calcination for removing the polymer to obtain the NiO–GDC cermets.

The morphology of the composite nanofibers was examined with SEM (S360 Cambridge, England) and TEM (CM200, Philips, Germany) attached with an energy dispersive spectrometer (EDS). The fiber diameter distribution was calculated with the Image J software from the SEM images, and also image processing software (Origin Pro 8) used to draw the diameter distribution histograms.

RESULTS AND DISCUSSION

Effect of Polymer Concentration Solution

A series of samples with different PVA concentration (8, 10 and 15 % w) were electrospun, resulting in various fiber morphologies, as shown in *Figure 1*. Tacx et al. [25] have obtained the following Mark–Houwink relationship for PVA in water:

$$[\eta]=6.51\times10^{-4}\ M_W^{0.628} \quad (1)$$

Using this equation, [η]C was calculated for the PVA (M_W=72000) used in this study. The calculated values of [η]C for stable fibrous structures were typically between 5 and 12, indicating that a minimum degree of chain entanglement was needed for producing fibrous structures [26]. Therefore, the PVA concentration was chosen to be 6.8-16.4 for obtaining uniform fibers. By keeping the processing variables as the fixed parameters, a morphological transformation of electrospun products was seen as the round fibers at a concentration of 8 wt% to the ribbon-like at the high concentrations. It was also noticed that uniformity of the fibers was decreased with increasing CPS. This happens when the molecular weight of the polymers and CPS were both high. This is because the solvent evaporation was reduced when the solution was in high viscosity, and as a result, those wet fibers which could be reached to the collector are flattened due to the impact [27].

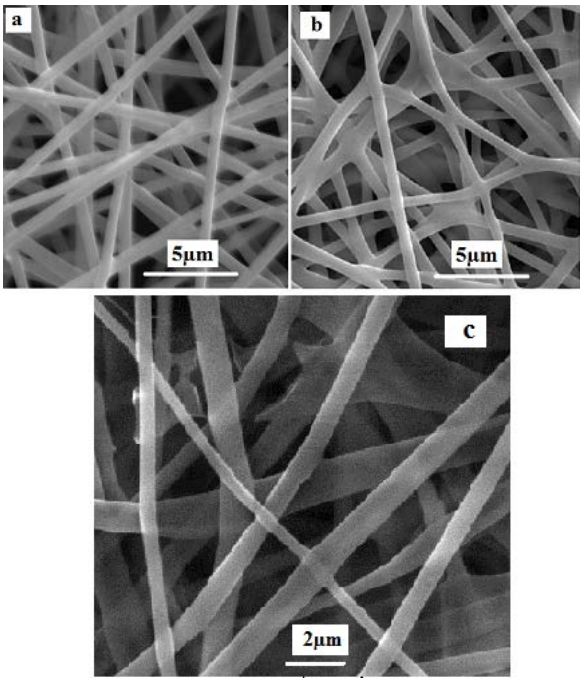

FIGURE 1. Morphology of the as-spun PVA/NiO-GDC fibers obtained with traveling distance of 100 mm, needle diameter of 0.6 mm, voltage of 10kV and flow rate of 0.1 ml $h^{-1}$and a polymer concentration with: (a)8 %w, (b)10 %w, (c)15 %w.(SEM MAG:10 kx).

Effect of Cationic Concentration of Starting Materials

Adding proper amounts of cationic precursors into the polymer solution is another simple and effective way to inhibit the bead formation in the electrospinning process and favors the fiber formation. To examine the effect of precursor

concentration on the morphology of NiO-GDC nanofibers, three solutions with various molarities (0.5, 1 and 2 M) were prepared, while other processing parameters such as, feed rate, nozzle to collector distance, voltages were kept constant. *Figure 2* shows SEM images of the samples before and after calcination. As could be seen in this figure, the fibers which derived from the solution having concentration of 0.5 M were perfectly flat and smooth. With increasing the solution concentration, the fibers were tangled together and it could be seen that after calcination morphology was changed from fibrous like to flat materials. The electrospinning of the samples having 1 and 2 molarities was difficult, due to the instability of the jet and sticking the viscous gel inside the needle tip. When the concentration of the solution was too high, the relatively high molarity could also inhibit the electrospinning process so the fibers were highly interconnected (*Figure 2e*). The addition of cations resulted in a higher charge density on the surface of ejected jet during spinning, thus more electric charges are carried by the electrospinning jet. As the charges carried by the jet increase, fibers were drawn faster to the collector and there was not enough time for removing solvent. Therefore the diameter of the final fibers also becomes substantially larger [9].

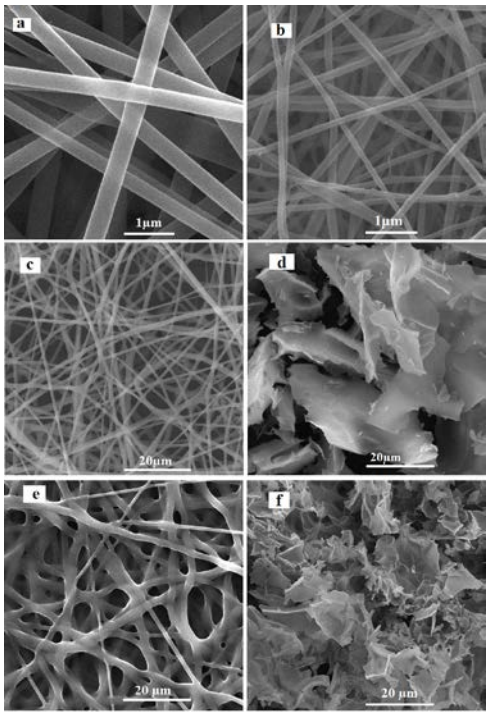

FIGURE 2. Morphology of the samples obtained with a 10% (w) polymer solution at a traveling distance of 100 mm, needle diameter of 0.6 mm, voltage of 10 kV, flow rate of 0.1 ml h^{-1}, and cationic concentration (a,b): 0.5 M, (c,d): 1 M , (e,f): 2 M before and after calcinations in right and left, respectively.

Effect of Calcination Temperature

All the electrospinning experiments were performed at room temperature (~25°C) and below 30% relative humidity. The heating program started from room temperature to 300˚C to omit PVA for 1 h (heating rate: 5˚C /min), and then calcinated at 800˚C, 900˚C and 900˚C for 2 h (heating rate: 10 ˚C /min) to form the NiO‑GDC composite nanofibres.

It was interesting to observe that the fibrous nature of the original composite fibers was retained, even though PVA was removed. It is noticed that the average fiber diameter decreased with increasing calcination temperature. The average diameter of as-prepared (550±216 nm) and calcinated (75±27 nm) fibers at a temperature of 1000 ˚C indicated shrinkage of the fiber structure during calcination as shown in *Figure 3*. This is due to the decomposition of the organic materials, which belonged to PVA and also the NO_3 group of the nitrate components and the other volatiles materials such as H_2O, CO_x and etc. As previously described, the neat composite fibers that had been prepared from the chosen spinning condition were smooth. After calcinations processes, the shrinkage of the fibers was evident, and the extent of shrinkage becoming more pronounced with an increase in the calcination temperature. Specifically, with increasing the calcinations temperature from 800 ˚C to 1000 ˚C, the average fiber diameters were gradually decreased from 190±77 nm to 75±27 nm. Also with increasing calcinations temperature, the resulting nanofibers became rougher, which was believed to be caused by crystallization of the GDC solid solution. These results were consistent with the findings of other researchers [28, 29].

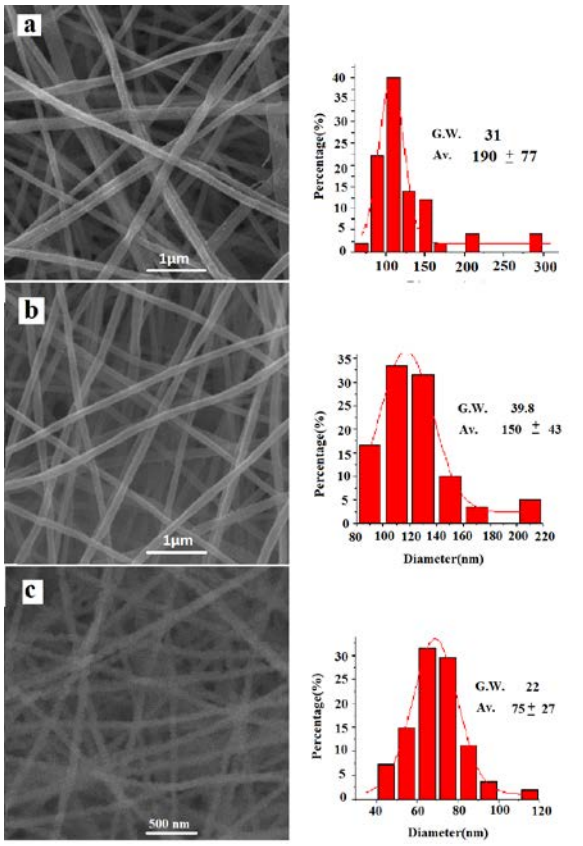

FIGURE 3. Morphology of the NiO-GDC nanofibers calcined at (a):800 ˚C, (b): 900 ˚C, (c): 1000 ˚C.

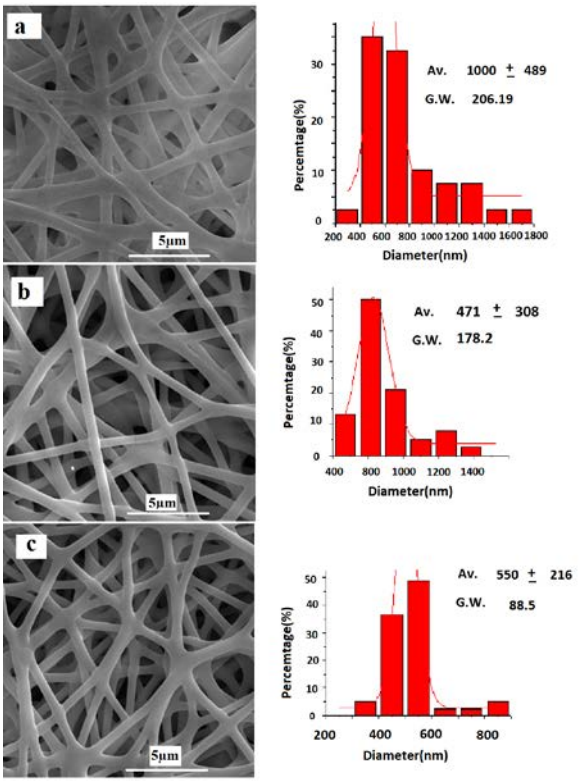

FIGURE 4. Morphology of the as-spun PVA/NiO-GDC mats for the samples obtained with a 10% (w) polymer solution at a traveling distance of 100 mm, needle diameter of 0.6 mm, flow rate of 0.1 ml h^{-1} and a voltage of (a) 8 kV, (b) 10 kV, (c) 15 kV.

Effect of Applied Voltage

The influence of applied voltage in the average size of the electrospun fibers was also studied. A series of experiments were carried out when the applied voltage was varied from 8-15 kV and the tip to target distance was held at 100 mm. It could be seen in *Figure 4* that the electrospun fibers contained many branches and were highly interconnected. Also it was noticed that all the samples were free of beads, indicating that the tested NiO electrospinning conditions provided sufficient chain entanglement for fiber formation. A narrow distribution of fiber diameters was observed at 15 kV, while broad distribution in the fiber diameter was obtained at 8 kV. Increasing the applied voltage would increase the electrostatic repulsive force on the fluid jet which favors the thinner fiber formation [9].

It can be seen that by increasing the voltage, the diameters of the fibers became non-uniform. Also with increasing the voltage, the spaces between the fibers were increased due to increasing the electrostatic repulsive force on the fluid jet which favors the thinner fiber formation. It can be seen that split fibers were formed by the separation of a primary jet into two smaller jets. The elongation of the jet and the evaporation of the solvent both change the shape and the charge per unit area carried by the jet. The balance between the surface tension and the electrical forces could collide so that the shape of a jet became unstable. Such an unstable jet could reduce its local charge per unit surface area by ejecting a smaller jet from the surface of the primary jet or by splitting apart into two smaller jets. Due to several branches of a scrub brush, and bond with other fibers, the morphology of the mat was generally felt to be a woven network. Individual fibers form when all or most of the solvent evaporate before reaching to the collector [27].

Effect of the Tip–Target Distance

The influence of the distance between the needle tip to the grounded collector on the fiber average diameter and distribution was also analyzed. *Figure 5* shows the SEM images of the samples which were electrospun at various distances from the collector (8, 10 and 15 cm). Increasing of the tip–target distance had no significant effect on the electrospun fiber diameter, as shown in *Figure 5*. As can be seen, with increasing distance from 8 cm to 10 cm there was no change in the average fiber diameter. When the distance between the needle tip and the collector was low (e.g. 8 cm), the solution traveled less distance to reach the collector plate, therefore the solvent had found less time to evaporate. Residual solvent remained in the mats caused formation of the bind between the inner and outer layers of the fibers and therefore a network was generated which could increase the strength of the mat. When the distance was fixed at 10 cm, the solvent had enough time to evaporate and separate fibers sit on collector. With increasing distance to 15 cm, the average fiber diameter was increased to 2750 ±1369 nm and again fibers were tangled together. The increase in diameter of the fibers was caused by reduction of the electrostatic field strength and hence the fibers were less stretched [7, 30]. The above results showed that in order to prepare single fibers, optimization of the needle to collector distance is required.

Effect of Feed Rate

It can be seen from *Figure 6* that with increasing feed rates, the average fiber diameter increased, and this was because a greater volume of liquid was drawn out of the tip. Average fiber diameter and standard deviation for the feed rate of 0.1ml/h, 0.3 ml/h and 0.5 ml/h were calculated to be 671±104 nm, 700±302 nm, and 1000±489 nm, respectively. On the other hand, uniformity of the fibers decreased with increasing the feed rate. This is because high injection rate and hence greater volume of solution being drawn from the tip of the needle, the more time was taken to dry the accelerated solution. Constant tip-target distance and also not having enough time to form thinner fiber in higher feed rate, fibers were tangled together to form straps [31].

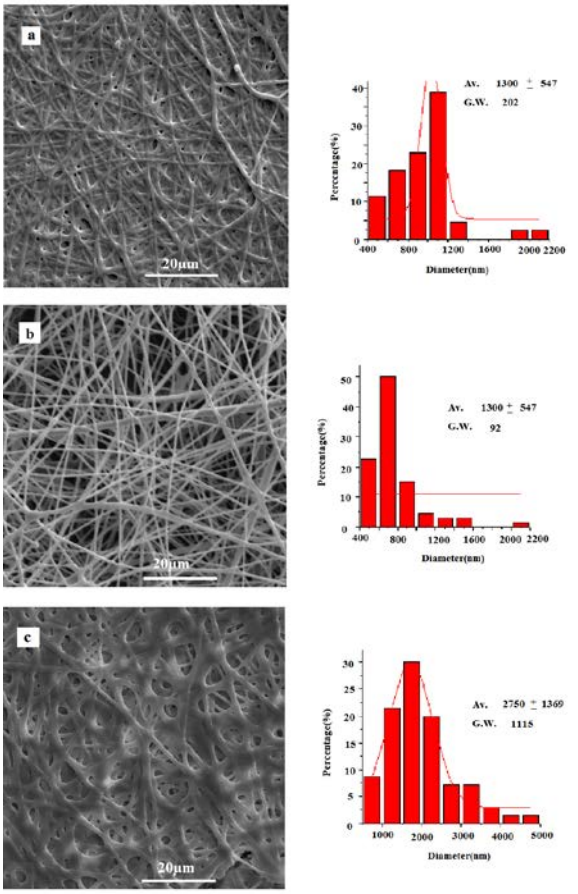

FIGURE 5. Morphology of the as-spun PVA/ NiO-GDC mats for the samples obtained for a 10% (w) polymer solution, and a needle diameter of 0.5 mm, flow rate of 0.1 ml h−1, a voltage of 15 kV and a distance between the needle tips to collector of (a) 80 mm, (b) 100 mm, (c) 150 mm.

Effect of the Needle Diameter

As it can be seen in *Figure 7*, with the increasing internal diameter of the needle from 0.6 mm to 0.8 mm, the average diameter of the fibers increased. When the internal diameter decreased, the droplet size of the tip of the needle became smaller and the surface tension of the droplet increased, so the larger columbic force was needed to start the accelerated solution flow. This is because, the solution was ejected from the smaller needle would have more time for stretching and elongation, so the fiber diameter was decreased by reducing the internal diameter of the needle. Also the samples obtained with a larger needle diameter showed a more uniform fiber size distribution.

TEM Analysis

In order to further identify the nano-phase nature of the NiO-GDC particles contained in the composite fibers, the TEM image and EDS pattern of composite fibers calcined at 900˚C are shown in *Figure 8*. The resulting fibers were composed of very small grains uniformly linked with an average diameter of 9 nm, indicating the nanostructure of the fibers. Energy dispersive spectrum collected on the calcined NiO-GDC fibers distinctly identifies the presence of Ce, Gd, Ni and O as the elemental component in the fibers.

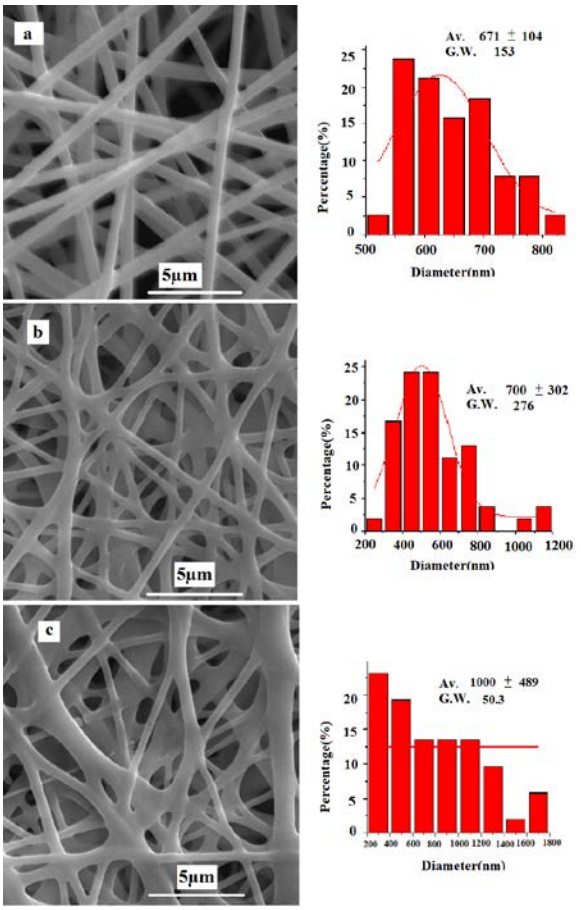

FIGURE 6. Morphology of as-spun PVA/ NiO-GDC mats for the samples obtained with a 10% (w) polymer solution at a traveling distance of 100 mm, needle diameter of 0.6 mm, voltage of 10 kV and flow rate of (a): 0.1 ml h^{-1}, (b):0.3 ml h^{-1}, (c): 0.5 ml h.

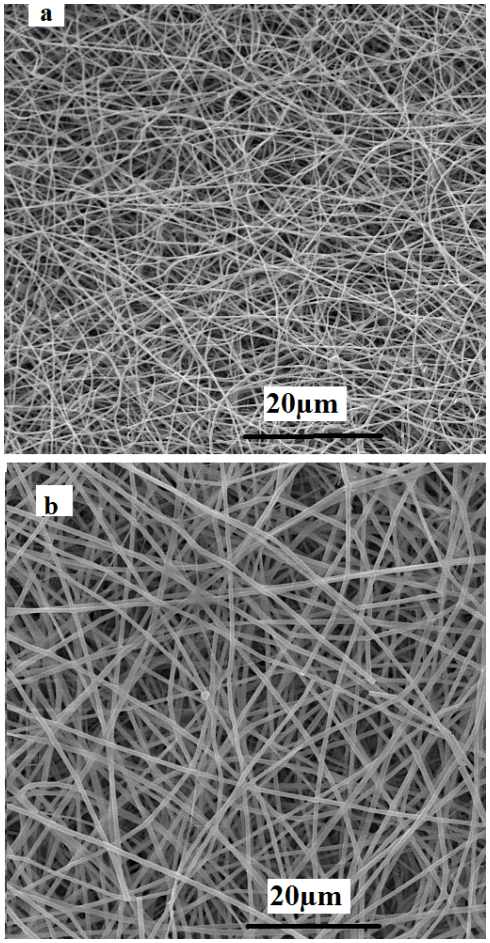

FIGURE 7. Morphology of the as-spun PVA/ NiO-GDC mats for the samples obtained with a 10% (w) polymer solution at a traveling distance of 100 mm, voltage of 10 kV, flow rate of 0.1 ml h^{-1} and the diameter of the needle (a) 0.6 mm, (b) 0.8 mm.(SEM MAG:2kx).

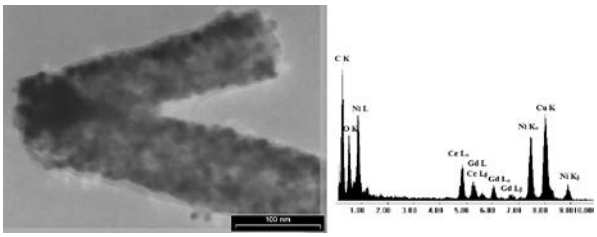

FIGURE 8. TEM image of a NiO-GDC nanofibers and EDS pattern.

CONCLUSION

In this paper, continuous NiO-GDC composite nanofibers were prepared by electrospinning different concentrations of aqueous PVA solutions. The effect of the concentration of the solutions and electrospinning parameters were studied on the morphology and diameter of NiO-GDC nanocomposite. It was observed that by increasing the CPS, the morphology of the fibers was changed from circular cross-section to ribbon shape. It was noticed that decrease of the tip-target distance caused the diameters of the fibers to become smaller and an increase of the applied voltage also decreased the nanofibers diameter. There was a slight increase in average fiber diameter with increasing feed rate of the solution. Needle diameter did not have significant effect on the fiber diameter; however the morphology of the nanofibers was slightly changed. It was also found that smooth fibers were changed to agglomerated fibers when the distance between the needle tips to the sample collector was increased. It was generally observed that the calcinations temperature had a great influence on both the crystalline phase and the surface morphology of the fibers. As the calcination temperature increased the nanofibres became smaller and the surface rougher.

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AUTHORS' ADDRESSES
Raziyeh Ghelich
Mansoor Keyanpour Rad
Amir Ali Youzbashi
Materials and Energy Research Center (MERC)
PO Box 14155-4777
Tehran
IRAN