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Conductive polypyrrole nanofibers via electrospinning: Electrical and morphological properties

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Abstract

Conductive polypyrrole nanofibers with diameters in the range of about 70–300 nm were obtained using electrospinning processes. The conductive nanofibers had well-defined morphology and physical stability. Two methods were employed. Electrospun nanofibers were prepared from a solution mixture of polypyrrole (PPy), and poly(ethylene oxide) (PEO) acted as a carrier in order to improve PPy processability. Both the electrical conductivity and the average diameter of PPy nanofibers can be controlled with the ratio of PPy/PEO content. In addition, pure (without carrier) polypyrrole nanofibers were also able to be formed by electrospinning organic solvent soluble polypyrrole, $[(PPy3)^{+} (DEHS)^{-}]_x$, prepared using the functional doping agent di(2-ethylhexyl) sulfosuccinate sodium salt (NaDEHS) [Jang KS, Lee H, Moon B. Synth Met 2004;143:289–94. [24]]. Electrospun blends of sulfonic acid (SO_3H)-bearing water soluble polypyrrole, [PPy(SO_3H)–DEHS], with PEO acting as a carrier, are also reported. The factors that facilitate the formation of electrical conduction paths through the electrospun nanofiber segments are discussed.

Keywords: Nanofibers; Electrospinning; Conducting polymer; Polypyrrole

1. Introduction

Electrospinning is being used to an increasing extent to produce ultra thin fibers from a wide range of polymer materials [1–9]. This non-mechanical, electrostatic technique involves the use of a high voltage electrostatic field to charge the surface of a polymer solution droplet and thus to induce the ejection of a liquid jet through a spinneret. In a typical process, an electrical potential is applied between a droplet of a polymer solution held at the end of a capillary tube and a grounded target. When the electric field applied overcomes the surface tension of the droplet, a charged jet of polymer solution is ejected. The single jet initially formed is divided into multiple filaments by radial charge repulsion, which results in the formation of solidified ultra thin fibers on the target. A characteristic feature of the electrospinning process is the extremely rapid formation of the nanofiber structure, which is on a millisecond scale [2]. Other notable features of electrospinning are a huge material elongation rate on the order of 1000 s^{-1}, and a cross-sectional area reduction on the order of 10^5 to 10^6, that have been shown to affect the orientation of the structural elements within the fiber [2].

Polymer nanofibers have a diameter that ranges from an order of a few nanometers to several micrometers and are remarkable for their very high surface area per unit mass, their small pore size and very high porosity, and a low basis weight. Optimal nanofibers can be prepared by controlling electrospinning process parameters, including the strength of the applied electric field, the deposition distance, and the properties of the polymer solution [3,5,6].

Moreover, this process is highly versatile and allows the processing not only of many different polymers into polymeric nanofibers but also the co-processing of polymer mixtures, and mixtures of polymers and low molecular weight non-volatile materials, simply by using ternary solutions of the components for electrospinning to form a combination of nanofiber functionalities [3,5].

Electrically conductive polymers have attracted much interest in the past 20 years because they simultaneously display the physical and chemical properties of organic polymers and the electrical characteristics of metals. Further,

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fabrication of nanofibers made of conductive electronic polymers has recently been demonstrated in the design and construction of nanoelectronic devices [10,11].

The main work in the electrospinning of conductive polymers focuses on polyaniline (PANi) and blends thereof [10–15]. Mac Diarmid et al. [11] prepared highly conductive sulfuric acid-doped PANi electrospun fibers using a mixture of PANi and different conventional polymers such as PEO, polystyrene, polyacrylonitrile, etc. Electrospinning of conductive nanofibers using poly(3,4-ethylenedioxythiophene), PEDOT, a commercial polythiophene derivative (Baytron P type from Bayer), has also been reported [16]. In this study, poly(3,4-ethylenedioxythiophene)/poly(styrene sulfonate) (PEDOT/PSS) dispersions were used using polyacrylonitrile as carrier.

Polypyrrole (Fig. 1) is one of the most widely investigated conductive polymers because of the aqueous solubility of the monomer, the low oxidation potential and the high conductivity. It has also been investigated for many industrial applications such as antistatic [17], electromagnetic shielding [18], actuators [19] and polymer batteries [20] because of its easy synthesis and long-term ambient stability [21,22]. However, the inherently poor solubility in common solvents, which originates from the strong inter- and intra-chain interactions, has limited practical applications of polypyrrole in many areas.

To our knowledge, only very recent work has attempted to electrospin polypyrrole (first chemically polymerized using ammonium persulfate as the oxidant and dodecylbenzene sulfonic acid as the dopant source) alone and in mixtures with a carrier such as poly(vinly cinnamate) [23]. The electrospun polypyrrole fibers exhibited a circular cross-section. Their diameter was about 3 μm and the electrical conductivity of the compressed polypyrrole non-woven web was reported to be about 0.5 S/cm.

In the present study, conductive polypyrrole nanofibers were obtained using polyethylene oxide (PEO) as the carrier through electrospinning of aqueous solutions. We also prepared pure (without carrier) polypyrrole conductive nanofibers by electrospinning organic solvent soluble polypyrrole using the functional doping agent di(2-ethylhexyl) sulfosuccinate sodium salt (NaDEHS), synthesized at Sogang University (South Korea) [24–27]. Electrospun blends of water soluble polypyrrole [24] using the functional doping agent NaDEHS with PEO are also reported. The electrical and morphological characteristics of these polypyrrole nanofibers were investigated.

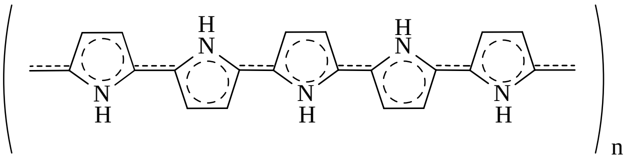

Fig. 1. The chemical structure of polypyrrole.

2. Materials and methods

2.1. Materials

Poly(ethylene oxide) (PEO), with an average molecular weight (M_w) of about 400,000 g/mol, water soluble polypyrrole solution (Product No 48 255-2, 5 wt% in water doped with proper dopants) and Triton-X 100 non-ionic surfactant from Sigma-Aldrich (S-A) were used. According to the supplier, the conductivity of the polypyrrole (PPy) films was 0.025 S/cm.

High-molecular weight (M_w 62,300 g/mol) polypyrrole that is highly soluble in both water and organic solvent was synthesized using the functional doping agent di(2-ethylhexyl) sulfosuccinate sodium salt (NaDEHS). The sample was kindly donated by the laboratory of Prof B. Moon, Department of Chemistry, Sogang University, South Korea [24]. The solubility of water soluble polypyrrole $PPy(SO_3H)$–DEHS was about 3 wt%/vol, while the solubility of organic solvent soluble polypyrrole $[(PPy3)^+ (DEHS)^-]_x$ was about 9–10 wt%/vol in polar dimethylformamide (DMF). The electrical conductivity of the polymer films of $PPy(SO_3H)$–DEHS cast from aqueous solutions was 2×10^{-1} S/cm [24] and the conductivity of the $[(PPy3)^+ (DEHS)^-]_x$ films prepared using DMF as solvent was reported to be 2.3 S/cm [27].

2.2. Electrospinning

PEO solutions were prepared at room temperature by dissolving the polymer in deionized water. PEO solution was added to PPy polymer (S-A) and the solution was stirred overnight to ensure complete dissolution. Solutions of polymer blends with PEO and PPy were prepared with concentrations of PEO of 1.5 and 2.5 wt% and an amount of PPy solution ranging from 20 to 80 wt% (thus giving a ratio of 1.0–4.0 PPy/PEO content in the mixed solution). Polypyrrole solutions of $[(PPy3)^+ (DEHS)^-]_x$ dissolved in (DMF) and $PPy(SO_3H)$-DEHS blend solutions with PEO were also prepared for electrospinning. PPy solutions (S-A) with lower water contents (dried in an oven at 40 °C), and PPy solutions blended with typically spin-enhancing solvents such as chloroform or dimethylformamide at different ratios, with or without the presence of salts (LiCl, NaCl) were also examined with electrospinning.

The polymer solutions were electrospun at room temperature at driving voltages of 30 kV (HV Power Supply, Gamma High Voltage Research, Ormond, FL). The syringe used in this experiment had a capillary tip diameter of 0.8 mm that contained an attached copper wire that was used as the positive electrode. A grounded metal screen covered by a paper sheet was used as the counter electrode and was placed 20 cm from the tip of the capillary. Continuous nanofibers were deposited on the paper target and collected in the form of non-woven, fibrous mats. The final nanofiber mats had a uniform thickness of about 10–20 μm and were stored in a desiccator. The thickness was measured using a live camera in an optic microscope Nikon SMZ-U, with Easy Image Measurement software.

2.3. Extraction of PEO

Accelerated extraction of PEO from the nanofibers structure was done with Dionex ASE200 Accelerated Solvent Extractor (Sunnyvale, CA) using ethanol as solvent at a temperature of 60 °C and a pressure of 2000 psi for 20 min.

3. Characterization

3.1. SEM microscopy

The morphology and diameter of PET nanofibers were determined with a scanning electron microscope SEM (Jeol JSM-T300). A small section of the fiber mat was placed on the SEM sample holder and sputter-coated with gold.

3.2. Electrical conductivity

Conductance was measured using a two-point method in accordance with ASTM 4496-04. An Oltronix D400-007D voltage supply was used to create a voltage, and the current through the sample was measured with a 602 Solid State Electrometer (Keithley Instruments), which is able to measure currents as low as 10^{-11}.

Before measuring the conductivity, the fiber samples (dimensions $2\times2\ \text{cm}^2$) were conditioned for 24 h in 23 ± 1 °C and 25% relative humidity. Each sample was measured ten times in different directions by applying a potential of 100 V. Average values are reported. Note that we have tested the electrical conductivity of the nanofiber samples randomly collected on the paper sheet, while the previous publication [23] reported the electrical conductivity of a ‘compressed non-woven web’ (of unspecified thickness).

4. Results and discussion

4.1. Polypyrrole/poly(ethylene oxide) nanofibers

Doped PPy aqueous solutions (S-A) without or with the presence of typically spin-enhancing solvents and salts cannot be processed into fibrous forms. To improve the processability of PPy, solutions of PPy with poly(ethylene oxide) (PEO) were electrospun into ultrafine fibers. A critical concentration of 1.5 wt% PEO polymer in solution was required in order to achieve electrospun PPy/PEO nanofibers (Figs. 2 and 3). Below this concentration, electrospray took place as chain entanglements are insufficient to stabilize the jet, resulting in the spraying of droplets that coalesced into ill-defined shapes (data not shown). The long electrospun PPy/PEO nanofibers exhibited a cylindrical morphology and were randomly distributed in a fibrous mat with very uniform and dense structures that adhered to each other. It should be noted that the addition of PPy reduced the minimum concentration necessary for electrospinning a PEO–water solution to 1.5 wt%, much lower than the 7 wt% found for the preparation of pure PEO nanofibers from aqueous solutions (using similar M_w PEO) [28].

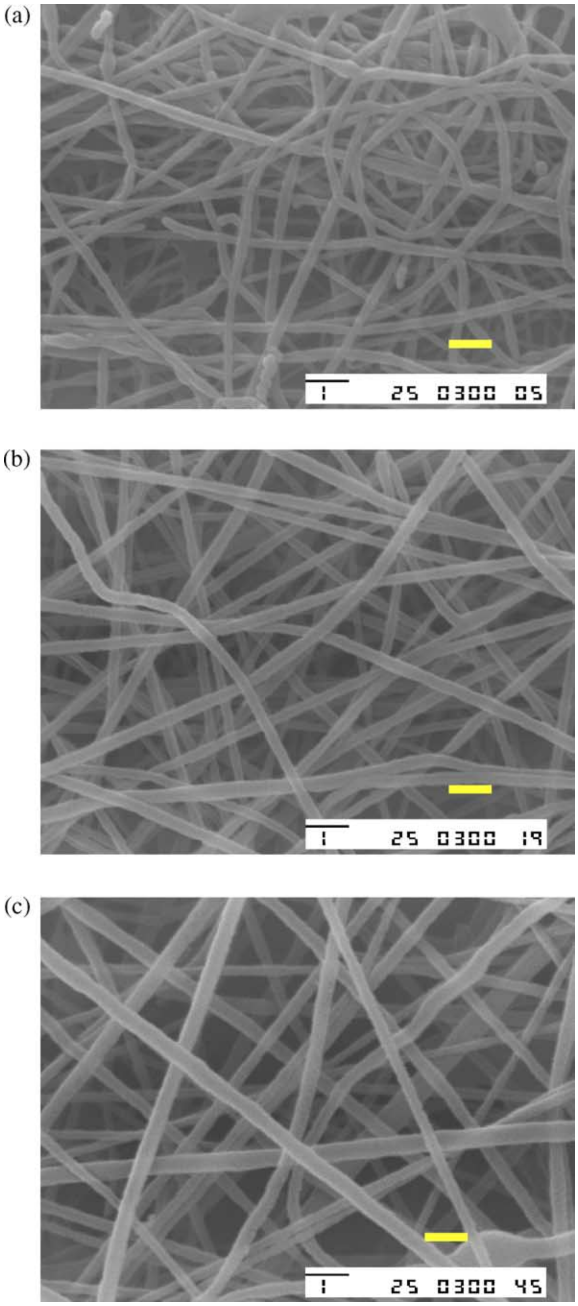

Fig. 2. SEM micrographs of electrospun nanofibers from aqueous solutions of 1.5 wt% PEO as carrier with various PPy concentrations. The PPy content of the nanofibers is (a) 45.5 wt%, (b) 62.5 wt% and (c) 71.5 wt%. The scale bar is 1 μm.

Overall, the size, but not the morphology, of the nanofibers was affected by the ratio of the PPy/PEO content. The average diameter of the nanofibers was in the range of about 200–300 nm and increased with increasing PPy concentration (Fig. 4). It is also evident that decreasing the PEO content also decreased the diameter of the nanofibers. The average nanofiber diameter as a function of PPy at low-PEO content

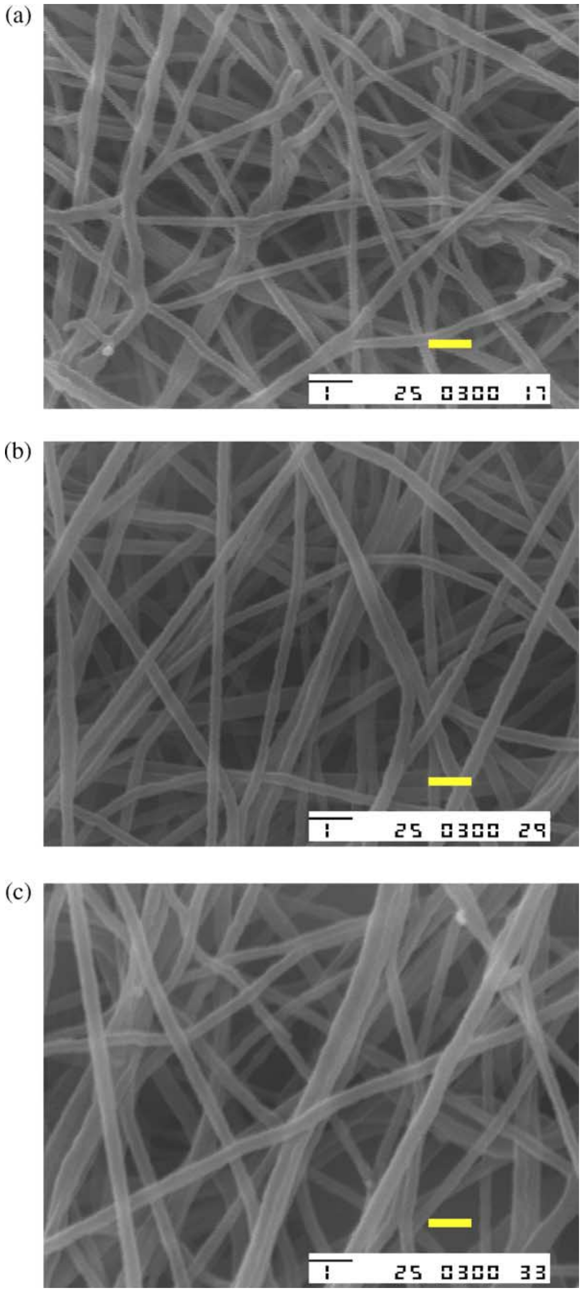

Fig. 3. SEM micrographs of electrospun nanofibers from aqueous solutions of 2.5 wt% PEO as carrier with various PPy concentrations. The PPy content of the nanofibers is (a) 37.5 wt%, (b) 50 wt% and (c) 55 wt%. The scale bar is 1 μm.

showed a steeper slope (≈ 3.6) than the diameter of nanofibers at high-PEO content (a slope of ≈ 1.6).

It has been shown earlier that the diameter of the fiber depends on the surface tension, flow-rate and electrical conductivity of the solution [5]. Thus, introducing a dispersant in the (1.5 wt%) PPy/PEO mixture, such as non-ionic surfactant Triton, resulted in a significant decrease in the nanofiber diameter, ranging from about 120 to 220 nm (Figs. 4 and 5). The resulting thinner, smooth fibers can be attributed to a decreased surface tension that tends to decrease the surface area per unit, hence changing the jets.

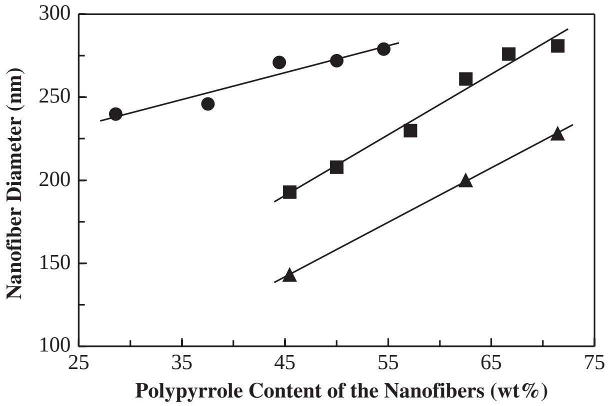

Fig. 4. Nanofiber diameter as a function of polypyrrole content. Solutions of PPy with 2.5 wt% PEO (●), 1.5 wt% PEO (■) and 1.5 wt% PEO with 0.5 wt% Triton-X100 (▲) as carrier were used.

4.2. $[(PPy3)^{+}\ (DEHS)^{-}]_x$ and $[PPy(SO_3H)–DEHS]$ nanofibers

Pure (without carrier) polypyrrole nanofibers with an average diameter of approximately 70 nm were formed using $[(PPy3)^{+}\ (DEHS)^{-}]_x$ dissolved in DMF (Fig. 6). This low average nanofiber diameter probably results from the relatively low molecular weight of the conducting polymer. The cylindrical nanofibers obtained are primarily randomly oriented, composed of many smaller fibers adhered to each other and are much thinner than PPy/PEO nanofibers. To the best of our knowledge this is the first conducting polymer with such a small average nanofiber diameter that has been electrospun without the use of a carrier.

Water soluble polypyrrole $[PPy(SO_3H)–DEHS]$ cannot be processed into fibrous forms without a carrier. In this case, the solution jet disintegrated into droplets and no polymer fibers formed. As seen in Fig. 7, however, continuous nanofibrous structures could be obtained from aqueous solutions of $[PPy(SO_3H)–DEHS]$ with 1.5 wt% PEO or 2.5 wt% PEO. The micrograph reveals the formation of a dense network of fibers with a cylindrical morphology and even distribution with numerous polymer–polymer junctions. Furthermore, these nanofibers were much shorter and thinner than the PPy/PEO nanofibers discussed above (Figs. 2 and 3). The average diameter of the nanofibers was approximately 100 and 150 nm formed via electrospinning a solution of $[PPy(SO_3H)–DEHS]$ with 1.5 or 2.5 wt% PEO, respectively, (Fig. 7).

4.3. Electrical conductivity

The electrical conductivity of these nanofiber structures can be varied by controlling the ratio of PPy/PEO. As shown in Fig. 8, the conductivity through the thickness of the electrospun PPy/PEO nanofibers increased by two orders of magnitude from the lowest to the highest concentration of PPy and ranged from about 4.9×10^{-8} to 1.2×10^{-5} S/cm. Obviously, the higher the PPy content of the PPy/PEO nanofibers, the higher the electrical conductivity, as contacts between conducting

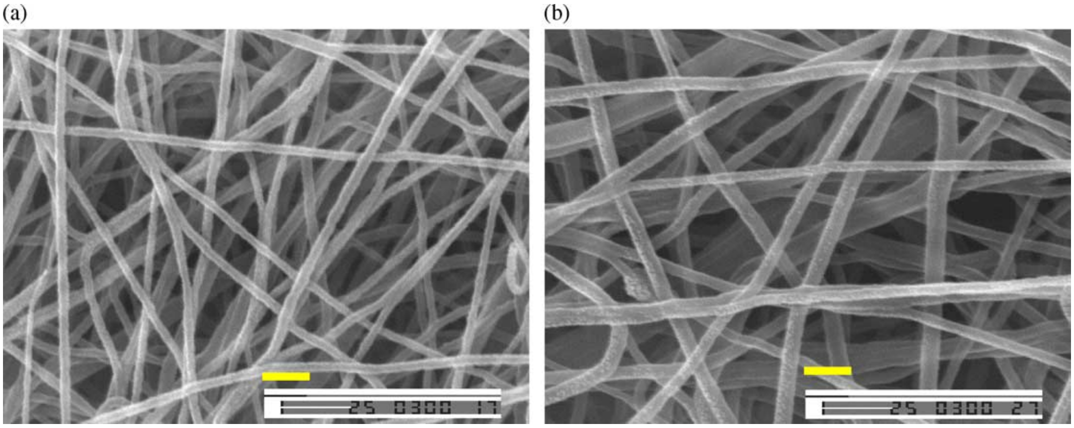

Fig. 5. SEM micrographs of electrospun nanofibers from aqueous solutions of 1.5 wt% PEO as carrier, with (a) and without (b) 0.5 wt% TritonX-100 surfactant. The PPy content of the nanofibers is 71.5 wt%. The scale bar is 1 μm.

polymer regions remain 'less isolated' from non-conducting regions and facilitate electrical conduction. In addition, the lower the (insulating) PEO concentration of the nanofibers, the higher is the conductivity, owing to the increase in the continuous domains of the conductive PPy molecules in the fiber structure. Further, it seems that, for the present polymer blends, the higher concentration of PEO in the initial solution is advantageous for forming a matrix nanostructure that provides higher conductive pathways or charge-carrier mobility of PPy molecules along the fibers.

We can thus imagine that a substantial fraction of conductive PPy molecules remains in connection to form continuous conduction paths. Moreover, the stretching of fibers in the electrospinning process may orient PPy molecules along the longitudinal fiber direction, thus also potentially increasing its charge-carrier mobility [29,30]. It is mainly the low initial PPy conductivity and the insulating PEO molecules between conducting polymer domains that explain the relatively low conductivity observed. As can be seen in the SEM micrographs of the electrospun nanofibers (Figs. 2 and 3), the fibers are highly porous, thus making the 'fill factor' of the PPy fibers less than that of a cast film [10]. In addition, it must be noted that measuring the conductivity of the very thin non-woven mat is considerably more difficult than measuring the conductivity of a cast film, as discussed in recent publications [10,16]. It is nevertheless reasonable to expect that the conductivity of an individual electrospun fiber will be higher than that of the non-woven mat.

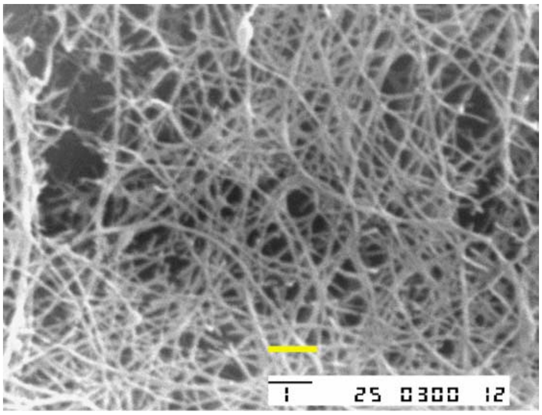

Fig. 6. SEM micrograph of electrospun nanofibers from 7.5 wt% $[(PPy3)^{+}(DEHS)^{-}]_x$ solution in DMF. The scale bar is 1 μm.

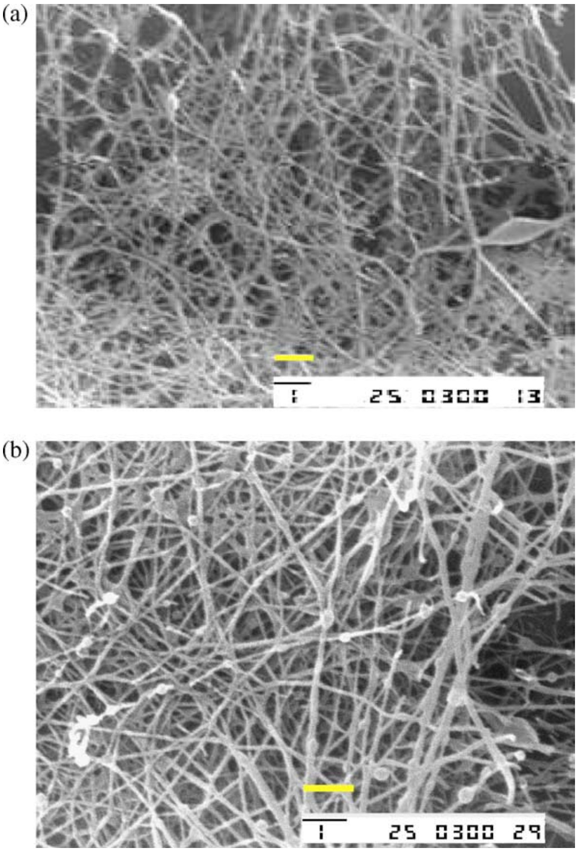

Fig. 7. SEM micrographs of electrospun nanofibers from aqueous solutions of [PPy(SO_3H)–DEHS] with PEO. (a) The [PPy(SO_3H)–DEHS] content of the nanofibers is 50 wt% (spun from a solution with 1.5 wt% PEO). (b) The [PPy(SO_3H)–DEHS] content of the nanofibers is 37.5 wt% (spun from a solution with 2.5 wt% PEO). The scale bar is 1 μm.

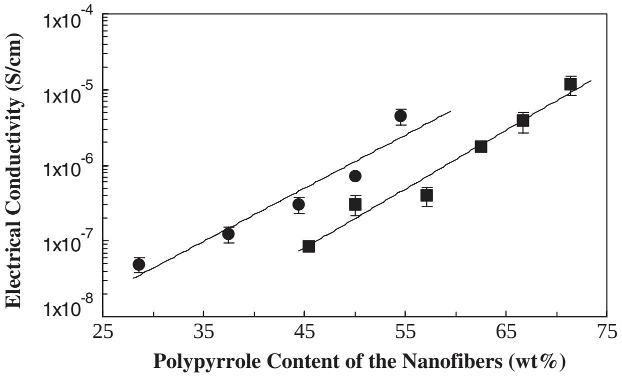

Fig. 8. Electrical conductivity of PPy/PEO nanofiber webs as a function of their polypyrrole content. Solutions of PPy with 2.5 wt% PEO (●), and 1.5 wt% PEO (■) as carrier were used (lines are visual guides).

Furthermore, since PEO has insulating properties, we anticipate that a higher order of PPy segments can be obtained by eliminating PEO from the fiber structure and that this would decrease the non-conducting barriers to charge transport between polypyrrole chains. It was tempting to reduce PEO from the nanofibers using a simple accelerated extraction treatment. We found that ethanol could provide satisfactory PEO removal, as determined using FT–IR spectroscopy, but extraction of PEO also caused morphological changes and the nanofiber matrix could finally not be maintained intact (microscopy data not shown). Thus the PEO domains impart the continuous fibrillar structure and provide sufficient mobility for the formation of conduction paths of matrix-dispersed and/or co-continuous PPy domains along the length of the nanofibers. The compatibility of the species prior to solidification owing to the low molecular weight of PPy (in comparison with PEO) and the extremely rapid solvent evaporation during the electrospinning process diminish the formation of phase-separated domains between conductive polymer molecules along the length of the nanofibers.

The electrical conductivity was approximately 3.5×10^{-4} S/cm for 50 wt% content of [PPy(SO_3H)-DEHS] in the nanofibers (electrospun from a solution with 1.5 wt% PEO) and about 1.1×10^{-4} S/cm for 37.5 wt% content of [PPy(SO_3H)-DEHS] in the nanofibers (electrospun from a solution with 2.5 wt% PEO), which is nearly three orders of magnitude higher than that of the PPy/PEO samples. This can be explained in part by the higher initial polypyrrole conductivity [24]. It can also be related to an enhanced 'orientation' of conductive polymer domains along the fiber segments. Note as well that the [PPy(SO_3H)-DEHS]/PEO nanofibers have a very uniform morphology (Fig. 7), composed of very thin fibers with numerous polymer–polymer junctions which apparently promotes a lower porosity and higher 'fill factor' effect.

The electrical conductivity of the pure $[(\mathrm{PPy3})^{+}(\mathrm{DEHS})^{-}]_x$ nanofiber web was about 2.7×10^{-2} S/cm, which is about four orders of magnitude higher than that of the PPy/PEO nanofibers. This can obviously be explained by both the high initial polymer conductivity and the molecular orientation [23] of conducting domains induced during electrospinning.

5. Conclusions

The above results are the first examples of ultrafine and conductive polypyrrole fibers with diameters in the range of about 70–300 nm. The electrospinning process could be used to prepare pure (without polymer carrier) polypyrrole and polypyrrole/poly(ethylene oxide) nanofibers with a well-defined morphology and physical stability. It appeared that the addition of polypyrrole to the PEO solution has effect on the diameter of the electrospun fiber. The electrical conductivity of the pure $[(\mathrm{PPy3})^{+}(\mathrm{DEHS})^{-}]_x$ nanofiber web was about three orders of magnitude higher than that of the PPy/PEO. Moreover, the higher the PPy content of the PPy/PEO nanofibers, the higher the electrical conductivity, as contacts between conducting polymer regions remain 'less isolated' from non-conducting regions and facilitate electrical conduction.

Our results show that the following factors facilitate the formation of electrical conduction paths throughout the electrospun nanofiber segments: (i) both the nature of the polymer solutions (compatibility in solution) of the conducting/carrier polymer blends prior to solidification and the extremely rapid structure formation of polymer nanofibers diminish the formation of phase-separated domains between conductive polymer molecules along the length of the nanofibers, (ii) the conductive polymer/carrier polymer ratio which controls the fraction of conductive molecules that remain connected to one another to form continuous conduction paths, (iii) the stretching of fibers in the electrospinning process, which may orient conductive polymer molecules along the longitudinal direction of fibers and potentially increasing their charge-carrier mobility.

Finally, in terms of the processing of polypyrrole, it should be pointed out that the fact that these conductive nanofibers were fabricated using a simple electrospinning technique expands the potential for low-cost practical applications of polypyrrole nanofibers in the area of construction of nanoelectronic devices. Moreover, by choosing different types of carrier polymers and dopants, we can flexibly control the electrical behavior and functionality of the resulting electrospun nanostructures.

Acknowledgements

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