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Research Article

Electrical Conductivity Modulation of Crosslinked Composite Nanofibers Based on PEO and PEDOT:PSS

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The aim of this work is to investigate the development of nanofiber mats, based on intrinsically conductive polymers (ICPs), which show simultaneously a high electrical conductivity and mandatory insoluble water properties. In particular, the nanofibers, thanks to their properties such as high surface area, porosity, and their ability to offer a preferential pathway for electron flow, play a crucial role to improve the essential characteristics ensured by ICPs. The nanofiber mats are obtained by electrospinning process, starting from a polymeric solution made of polyethylene oxide (PEO) and poly(styrene sulfonate) (PEDOT:PSS). PEO is selected not only as a dopant to increase the electrical/ionic conductivity, as deeply reported in the literature, but also to ensure the proper stability of the polymeric jet, to collect a dried nanofiber mat. Moreover, in the present work, two different treatments are proposed in order to induce crosslinking between PEO chains and PEDOT:PSS, made insoluble into water which is the final sample. The first process is based on a heating treatment, conducted at 130°C under nitrogen atmosphere for 6 h, named the annealing treatment. The second treatment is provided by UV irradiation that is effective to induce a final crosslinking, when a photoinitiator, such as benzophenone, is added. Furthermore, we demonstrate that both crosslinking treatments can be used to verify the preservation of nanostructures and their good electrical conductivity after water treatment (i.e., water resistance). In particular, we confirm that the crosslinking method with UV irradiation results to being more effective than the standard annealing treatment. Indeed, we demonstrate that the processing time, required to obtain the final crosslinked nanofiber mats with a high electrical conductance, results to being smaller than the one needed during the heating treatment.

1. Introduction

Nowadays, intrinsically conductive polymers (ICPs) represent an intriguing class of functional materials, thanks to their unique properties that combine (i) good chemical resistance, (ii) low weight, and low production cost to good mechanical, optical, and electrical characteristics [1–3]. Among conductive polymers, poly(3,4-ethylenedioxythiophene) doped with poly(styrene sulfonate) (PEDOT:PSS) results in being the most attractive among ICPs, due to its high stability, high electrical conductivity, and larger processability [4]. In particular, due to its properties and versatility of use, the PEDOT:PSS polymer is widely used in several applications, such as electrochemical (bio)sensors [1, 3, 5], organic electrochemical transistors (OECT) [6, 7], and volatile organic compound (VOC) sensors, where PEDOT:PSS is directly used to design highly sensitive chemiresistors [8–10].

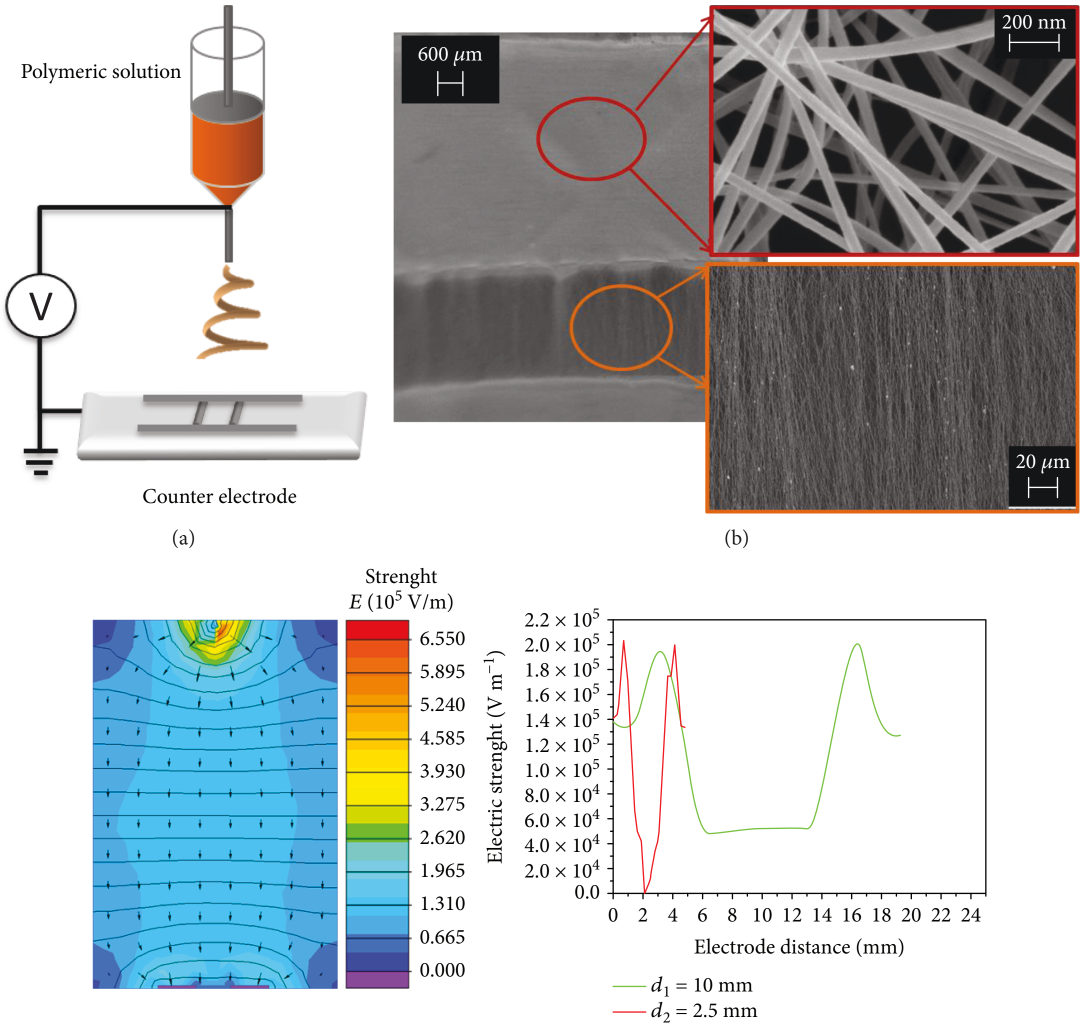
ICP polymers are usually processed in the form of thin films that can be obtained by different approaches, such as inkjet printing, dip-coating, doctor blading, and screen printing [11]. In the last years, an ever increasing interest has emerged to extend ICP processing also to other morphologies, and nanofibers by electrospinning are among the most interesting nanostructures so far. Indeed, as 1D nanomaterials, electrospun nanofiber mats can enhance the intrinsic properties of this kind of ICPs [8–10]. The set of properties shown by nanostructured samples obtained by electrospinning, such as high surface area, high porosity, and their ability to offer a preferential pathway for electron flow, play a crucial role to enhance the overall performances of ICP materials in electrochemical applications [5, 8–13]. For example, the sensitivity of (bio)sensors can be deeply improved by enhancing the surface area of samples [12, 13]. The key limit of ICP polymers is represented by their poor rheological properties, especially in terms of their low viscosity, making them unsuitable to be directly processed by electrospinning. Thus, a blending polymer has to be introduced allowing the formation of nanofibers. Unfortunately, the major part of polymers that can be used for this purpose, such as polyvinyl alcohol (PVA) and polyvinylpyrrolidone (PVP) [10], is electrically nonconducting. In the last decades, many works in the literature focused their attention on the development of PEDOT:PSS nanofibers, starting from a polymeric blend solution, where dopant compounds are added to enhance their electrical conductivity. Among all employed dopants, such as organic solvents (DMSO and EG) [14–17], anionic surfactants [18], and salts [19], polyethylene oxide (PEO) has demonstrated to be extremely effective in ensuring nanofiber formation and enhancing electrical conductivity. Indeed, Wang et al. [20] demonstrated a higher electrical conductivity reached by PEO/PEDOT:PSS nanofiber mats than the ones with poly(vinyl alcohol) PVA/PEDOT:PSS. The only disadvantage is due to the fact that the final nanofiber mats obtained by PEO/PEDOT:PSS blends are water-soluble, and this behaviour dramatically limits the possibility to use them in several fields requiring exposure to water-based solutions or moisture. Therefore, processes able to induce crosslinking, thus leading to enhancing the stability of these kinds of samples in water, are required. In the present work, we investigate nanofibers' water resistance, the latter property defined as proposed in the literature [17–24]. Indeed, water resistance can be defined as the capability of crosslinked samples to preserve their nanostructures and/or modify their morphological features in a reversible way, recovering their starting status after exposure to water or moist environment. Moreover, in the specific case of ICP polymers, water resistance also implies that they can retain their good electrical conductivity. In this work, we propose a direct comparison between two different crosslinking methods, employed to make insoluble the final electrospun samples. The first process is based on a heating treatment conducted at 130°C under nitrogen atmosphere, known as annealing treatment [25]. The second process is a photocrosslinking induced by UV irradiation in the presence of benzophenone, used as the photoinitiator. We especially demonstrate the effectiveness of both crosslinking processes, to improve the water resistance of crosslinked nanofibers, and analyse the morphology and the electrical conductivity of samples obtained by thermal- and photo-induced crosslinking. In particular, we focused our attention on the photo-induced crosslinking, which requires only 15 minutes to provide the expected result, thus leading to ensuring the preservation of the starting nanostructuration. Moreover, PEDOT:PSS NFs by UV crosslinking show a higher electrical conductivity than the one achieved/reached by a-NFs. In the present work, both crosslinking process are applied on PEO/PEDOT:PSS nanofiber mats characterized by an ordered distribution of nanofibers, with the aim to improve the electrical conductivity of the material [21–24]. Indeed, these patterned substrates are modified by depositing interdigitated electrodes, based on platinum, whose distance varies in the range from 10 mm to 2.5 mm. We then focused on the main target of the present work, which is to investigate the enhancement of water resistance of these kinds of crosslinked nanofiber mats.

2. Materials and Methods

2.1. Materials and Nanofiber Synthesis. All polymer-based solutions are based on polyethylene oxide (Mw = 600000 Da, purchased from Sigma-Aldrich) and PEDOT:PSS aqueous dispersion (Heraeus Clevios™ PH1000). Since the main target of the present work is to investigate the electrical conductivity of the final nanofiber mats, correlating it with their water resistance, two polymeric solutions (named P1 and P2) were prepared to obtain nanofiber mats. The first P1 contained 5 wt% PEO and 2.5 g of PEDOT:PSS solution in order to have a final content of 5 wt% of PEDOT:PSS with respect to the PEO amount. The second solution, P2, had the same composition of P1, expect for the addition of 0.1 wt% of benzophenone, an onion salt used as the photoinitiator to crosslink the PEO chains.

For ageing, both polymeric solutions were left under continuous stirring overnight. Electrospinning process was performed by NANON 01A (from MECC), starting from both polymeric solutions. In order to obtain a stable charged polymeric jet, a positive voltage of 23 kV was applied between the needle and the counter electrode, spaced 15 cm one from each other. Moreover, a flow rate of 0.1 $\mathrm{mL\,h^{-1}}$ was defined by a syringe pump. Samples of electrospun nanofiber mats were named PEO/PEDOT:PSS NFs. To achieve the water resistance behaviour, two different approaches were followed. The electrospun nanofibers, obtained from the P1 solution, were annealed at 130°C for 6 h under nitrogen atmosphere [25], and the resulting samples were named a-NFs, while the electrospun nanofibers obtained from P2 were exposed to UV light for photocrosslinking (35 $\mathrm{mW\,cm^{-2}}$, as measured by means of a UV Power Puck® II radiometer, EIT), obtaining the so-called PEDOT:PSS NF samples. The immersion into water for 15 minutes was carried out in order to verify the insolubility of both sets of nanofiber mats, a-NFs and PEDOT:PSS NFs.

2.2. Counter Electrode Modification. In the present work, we used platinum strips with dimensions from few tens of microns up to some millimeters, sputtered on a microscopic glass (carried out by Q150T ES by Quorum Technologies, applying a current of 50 mA for 120 s), in order to induce an aligned distribution of nanofibers between two adjacent metallic strips [21–24]. Indeed, platinum strips on a dielectric substrate can act as conductive protrusions, thus leading to locally enhance the electric field and consequently help in obtaining a selective patterned deposition of nanofibers. Moreover, to deeply understand the electric field distribution close to metallic strips, a free student software was implemented (QuickField Student Edition).



(a) (b)

(c)

Figure 1: (a) Sketch of the electrospinning process. (b) FESEM image of aligned nanofibers obtained between two metallic strips in as-spun PEO/PEDOT:PSS NFs. In the orange box, a higher magnification is proposed. (c) The simulation of the electric field intensity during the process is represented, and the detail of the simulation output at the electrode is proposed.

2.3. Characterizations and Measurements. Field effect scanning microscopy (FESEM, Zeiss MERLIN), operating in the voltage range from 5 kV to 10 kV, is used to evaluate the morphological properties of composite nanofibers. Optical microscopy (Nikon Eclipse ME600) is used to evaluate the morphological properties of both nanofiber mats (a-NFs and PEDOT:PSS NFs) before and after their exposure to water, thus leading to verify how their morphology can change and to demonstrate the preservation of nanostructures. To evaluate the electrical conductivity of all samples, electrical characterizations are performed by means of a Keysight B2912A source measure unit. First of all, I-V characterizations are obtained by polarizing all samples. A voltage range from −1 V to +1 V with a rate of 10 mV s^{-1} is applied, and consequently, the corresponding current (I), flowing through the materials, is measured. Then the resistivity and successively the electrical conductivity are defined, taking into account the thickness of all samples, measured by a surface profiler (TENCOR P-10).

3. Results and Discussion

3.1. Electrospinning on Electrode Assembly. During the electrospinning process, different substrates were used to induce a proper distribution with all aligned fibers; all of them are made of metallic strips sputtered on dielectric glass slides, as sketched in Figure 1(a). Since, as deeply investigated in the literature [21–24], metallic strips can locally modify the electric field distribution (E) during the process, enhancing its intensity in proximity of metals and then stretching nanofibers across them, two different distances between platinum strips are studied: (i) $d_1 = 10$ mm and (ii) $d_2 = 2.5$ mm. As previously mentioned, through this approach, aligned nanofibers are directly deposited on the counter electrode during electrospinning step, as represented in Figure 1(b). A random distribution of nanofibers is achieved on platinum strips, as highlighted by the red box in Figure 1(b). Electrostatic simulations, which permit analysis of the electric field distribution close to the metallic strips, confirm

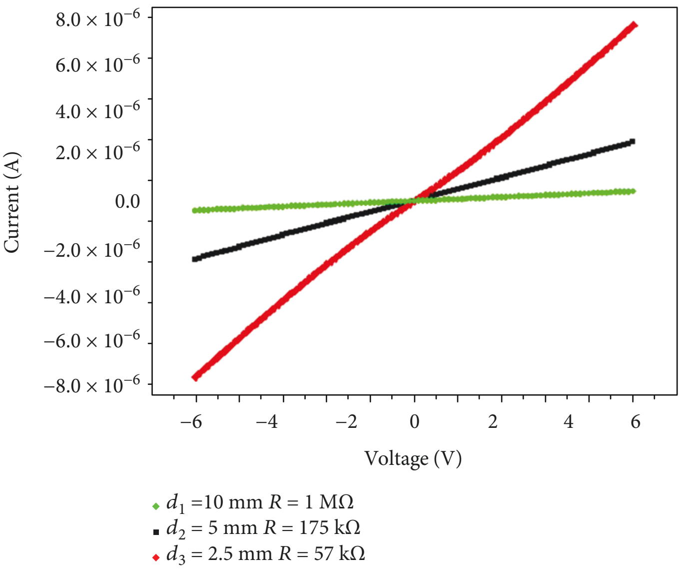


Figure 2: I-V characterizations performed on PEO/PEDOT:PSS nanofibers, obtained by providing three different distances between metallic strips: $d_1 = 10$ mm, $d_2 = 5$ mm, and $d_3 = 2.5$ mm.

these morphological properties. Furthermore, as reported in Figure 1(c), the electric field lines are divided in proximity of metallic strips and an enhancement of E is induced on them.

These results demonstrate that platinum strips, during the electrospinning process, play a crucial role into the modulation of E. Indeed a maximum value of E is reached on the metallic strips, while the minimum values are achieved in the gap. It is possible to evaluate how the electric field across the metals decreases as the distance between strips decreases. According to the Coulomb interactions, the induced electrostatic forces are inversely proportional to the square root of the gap, so that the lower the distance is, the higher the electrostatic force is, which causes a strong alignment of nanofibers across metallic strips in the direction perpendicular to the strips. Therefore, the nanofibers move towards an aligned parallel distribution, and they are positioned perpendicular to the edge of metallic strips [26].

The enhancement of E on platinum strips induces at the same time two effects: the first one, correlated with the increase of E intensity on metals, induces a random distribution on them, as shown in the red box of Figure 1(b). At the same time, a second effect is produced, which concerns the stretching of nanofibers between two adjacent metallic electrodes, thus leading to obtain aligned distributions of nanofibers across platinum, as highlighted in the orange box of Figure 1(b).

3.2. Electrical Characterizations on Patterned Nanofibers. Different distributions of nanofibers, induced by a modulation of the electric field during the electrospinning process, can deeply influence the electrical conductivity of final samples.

As reported in Figure 2, it is indeed possible to appreciate how the resistance of all nanofiber mats decreases more than proportionally as the distance among metallic strips decreases. In particular, experimentally, we defined three distances among the metallic strips, on which nanofiber mats are directly collected: (i) $d_1 = 10$ mm, (ii) $d_2 = 5$ mm, and (iii) $d_3 = 2.5$ mm.

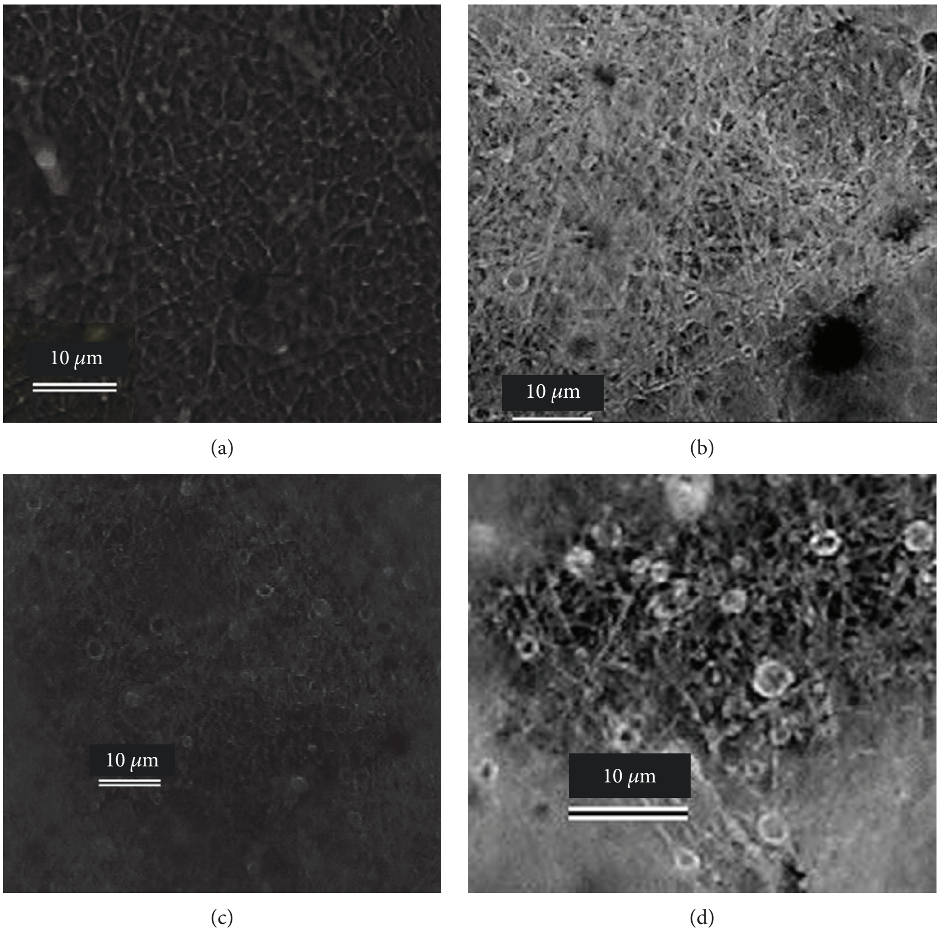
These results confirm that the nanofibers' distribution plays an important role in the modification of their electrical behaviour. In particular, a better alignment of nanofibers was reached with the lowest distance (d_3) among the metallic strips, creating a preferential pathway for electron flow, thus leading to improve the electrical conductivity even if the sample thickness, close to 10 μm, remains constant. For this reason, this sample offers a lower resistance value close to 57 kΩ.

Based on the results of these electrical characterizations, we decided to provide the same distance between metallic strips ($d_3 = 2.5$ mm) obtaining the final set of samples of a-NFs, PEDOT:PSS NFs, and PEO/PEDOT:PSS NFs.

3.3. Morphological Properties of Nanofiber Mats. As deeply investigated in the literature [25–31], thermal treatment and UV-curing with benzophenone as the photoinitiator are carried out in order to induce a proper crosslinking among PEO chains, ensuring then their insoluble properties. The target is to investigate the use of these processes in nanofiber-based samples, verifying if modifications can be induced on the morphology of nanostructures. In Figures 3(a) and 3(b), the morphologies of both a-NFs and PEDOT:PSS NFs after annealing and UV-curing, respectively, are shown.

In particular, from Figures 3(a) and 3(b), it is possible to appreciate that both postprocess treatments can induce the formation of a two-layer configuration. Indeed, a first layer can be observed at the bottom of the sample, and it is made of a thin film, while a second layer is present on its top, made of nanofibers, thus leading to verify the preservation of nanostructures after thermal treatment and UV-curing.

Moreover, Figures 3(c) and 3(d), referring to the samples after immersion in water, highlight the absence of morphological modification of a-NFs and PEDOT:PSS NFs due to



(a) (b) (c) (d)

Figure 3: Morphological properties of (a) a-NFS and (b) PEDOT:PSS NFs after UV-curing treatment, (c) a-NFs after immersion into water, and (d) PEDOT:PSS NFs after immersion into water.

dissolution of PEO and/or swelling phenomenon in the nanofiber mats, induced by the interaction of PEO/PEDOT:PSS with water. All these results demonstrate that both processes are effective in order to provide water-resistant samples, made of a-NFs and PEDOT:PSS NFs.

3.4. Electrical Characterizations on PEO/PEDOT:PSS Nanofibers.

Since one of the main targets of the present work is to establish how the exposure to water can modify the functional behaviour of a-NFs and PEDOT:PSS NFs, electrical characterizations are provided.

As previously introduced in Figure 2, the electrical characterization of the starting PEO/PEDOT:PSS NFs clearly shows that the designed nanofiber mats are able to conjugate two main features: the intrinsic conductivity, ensured by the PEDOT:PSS polymer, and optimal electron pathways along the nanofiber axis thanks to their aligned distributions. Consequently, the electrical conductivity of the final nanofiber mats result to being very close to 13.82 mS cm^{-1}. However, both crosslinked nanofiber mats, a-NFs and PEDOT:PSS NFs, are characterized by lower conductivity values, as summarized in Table 1. This result can be attributed to two main reasons. Firstly, the presence of a thin film layer at the bottom of a-NF and PEDOT:PSS NF samples (see Figures 3(c) and 3(d)) can offer a higher resistance to the electron flow, inducing consequently a worsening of the electrical conductivity of samples.

Table 1: Conductivity values of all samples: as-electrospun PEO/PEDOT:PSS NFs, annealed nanofiber a-NFs, and UV-curing PEDOT:PSS nanofibers. The conductivity values, measured before and after water treatment, are reported.

Electrical conductivity (mS cm^{-1})		
Nanofiber mats	Before water treatment	After water treatment
PEO/PEDOT:PSS NFs	13.82 ± 0.03 mS cm^{-1}	—
a-NFs	0.59 ± 0.01 mS cm^{-1}	0.57 ± 0.01 mS cm^{-1}
PEDOT:PSS NFs	2.61 ± 0.01 mS cm^{-1}	1.63 ± 0.04 mS cm^{-1}

A second reason to explain the lower performance of crosslinked samples can be attributed to PEO, according to the findings of Subramanian et al. [32]. That work investigated the condensation reaction occurring between PEO and PSS in films, during the annealing treatment. That reaction ensures the crosslinking reaction itself, and it occurs at the interface between PEDOT:PSS and PEO, thus leading to maintain unmodified the central part of PEO crystals [33].

The presence of intact PEO crystals inside the samples can reduce the electrical conductivity, acting as an insulating fraction inside the sample [33]. Electrospinning itself is well recognised as a process favouring the formation of crystals inside the nanofibers [34]. The presence of PEO crystals in PEO/PEDOT:PSS nanofibers can therefore even be increased. Secondly, as confirmed by several works in the literature [16], the electrical conductivity can decrease as a consequence of uncontrolled variations of the sample thickness, during both processes.

It is interesting to notice that PEDOT:PSS NFs processed by photocrosslinking exhibit a better electrical conductivity than thermal-annealed a-NF samples. Moreover, it is possible to appreciate how the electrical conductance still remains the same, after the exposure of a-NFs and PEDOT:PSS NFs to water, thus leading to demonstrate the water resistance properties induced in all samples by the two treatments.

4. Conclusion

In the present work, we investigated the fabrication of nanofiber mats through the electrospinning process, starting from a water-based solution, containing PEO and PEDOT:PSS.

Furthermore, we optimized the distribution of nanofibers on patterned substrates, thus leading to demonstrate the possibility to align nanofibers that result in ensuring a better pathway for electron flow, offering a lower resistance.

We compared two different treatments, implemented in order to induce a crosslinking between the PEO chains and PEDOT:PSS polymer, thus avoiding several phenomena that can occur when PEO is exposed to water, such as swelling and dissolution. We demonstrated that both of the two processes are effective to preserve the nanostructured morphology of the original samples, after exposure to water, highlighting then the efficiency ensured by photocrosslinking treatment. In the present work, we demonstrated the occurred water resistance of both crosslinked samples. The water resistance, in particular, is defined as the capability of two crosslinked mats to preserve the nanostructures and their function properties (i.e., high electrical conductivity), after exposing them to water or to humidity environment. Indeed, we demonstrated that PEDOT:PSS NFs show a preserved nanostructure, after UV irradiation, which, at the same time, ensures an efficient crosslinking in a smaller period.

A high electrical conductivity has been demonstrated for PEO/PEDOT:PSS NFs, close to $13.82 \pm 0.03\,\mathrm{mS\,cm^{-1}}$. The electrical conductivity was also analysed for both sets of samples, a-NFs and PEDOT:PSS NFs. In particular, it still remains the same, after exposure of a-NFs and PEDOT:PSS NFs to water, thus leading to demonstrate the water resistance properties induced in all samples by the two treatments.

Data Availability

All data, regarding the electrical conductivity of nanofibers and morphological properties of materials, used to support the findings of this study are included within the article. All these data are the results of our experimental activity and they are deeply described in the Materials and Methods of the main manuscript.

Conflicts of Interest

All authors declare that there is no conflict of interest regarding the publication of this paper.

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