Characterization of ZnO/Polyaniline Nanocomposites Prepared by Using Surfactant Solutions as Polymerization Media

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ABSTRACT: We report the preparation and characterization of pure polyaniline nanoparticles (PANI_NPs), zinc oxide nanoparticles (ZnO_NPs), and ZnO_NPs/PANI hybrid inorganic/organic nanocomposites (HION) dispersed in aqueous sodium dodecyl sulfate solutions. We have characterized the HION, which were prepared by the polymerization of aniline in presence of sodium dodecyl sulfate and ZnO_NPs, by dynamic light scattering, zeta potential, Fourier transform infrared (FTIR), X-ray diffraction (XRD), and scanning electron microscope (SEM). We have obtained ZnO_NPs, PANI_NPs, and ZnO_NPs/PANI with negative values of zeta potential and average diameters of 4.6 nm, 3.6 nm and 8.1 nm, respectively. XRD results reveal that the polymer exhibits a well-defined crystalline phase, as well as the presence of ZnO in a hexagonal phase. The observed changes in the FTIR spectra of the HION are associated to the formation of hydrogen bonding between the ZnO_NPs and the N—H groups present in the PANI chains. Heterogeneous structures with fibrous characteristics are identified in the SEM images. Electrical measurements indicate that the ZnO_NPs/PANI HION exhibit a noticeable light-sensitive ohmic behavior. We suggest that the preparation method here described offers an easy route of synthesis for the development of water-soluble ZnO/PANI-based nanostructures with high colloidal stability and varied functionality. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 125: E141–E147, 2012

Key words: conducting polymers; surfactants; composites; nanoparticles

INTRODUCTION

Hybrid inorganic/organic nanocomposites (HION) normally exhibit a set of physical and chemical properties, which are markedly distinct from those of the isolated original components.[1,2] In recent years, these characteristics have been increasingly exploited in innovative applications, which range from active materials for electromagnetic interference shielding and microwave absorbers to biological uses as contrast agents for differentiating between distinct tissues and structures and in intracellular hyperthermia treatment of cancer tumors.[3] (Metal oxide)/(conducting polymer) HION, in special, are shown to combine the electrical properties of their polymeric outer shell to the magnetic, optical, and (eventual) catalytic characteristics of the metal oxide core: in the latter case, for instance, conducting polymers have been used in different composites as supporting matrices for the intercalation of catalytic nanoparticles.[4]

In fact, recently polymer-ZnO nanoparticles have been used to modify graphite electrodes to be used as electrochemical sensors for immobilized DNA layers.[5] Zinc oxide nanoparticles (ZnO_NPs) present wide band gap and large excitation energies, and exhibit interesting catalytic, electrical, optical and electronic properties;[6–8] also, they are amenable to mass production by simple and relatively affordable methods.[9–11] At the same time, cationic conducting polymers offer the combined advantages of biocompatibility[12] and special affinity for DNA chains;[13] amongst them, polyaniline (PANI) deserves special attention because of its distinct electrochromic behavior and electrochemical characteristics,[14] and the fact that PANI samples prepared by facile and low-cost processing methods[15,16] can attain high conductivity while maintaining a good environmental stability. Although several examples of hybrid structures of PANI and different inorganic nanoparticles[17–20] can be found in the literature, no

studies seem to have been directed so far to the preparation and characterization of water-soluble ZnO_NPs/PANI composites. Also, although there are reports of preparation of pure PANI fibers by electrospinning in the literature,[21,22] to the best of our knowledge no previous report has been presented on the use of this method to obtain PANI/ZnO composite fibers. By use of the electrospinning technique, one can easily form polymeric fibers through an electrically charged jet of the appropriate polymer solution or polymer melt.[23] Although it has been introduced in the 1930s, more recently electrospinning is becoming an increasingly popular method of obtaining polymer fibers, either in the form of individual fibers or as nonwoven fiber mats.[24] In this article, we have used this method to synthesize ZnO_NPs/PANI fibers.

We first describe the use of a wet chemistry route to control aniline polymerization[25] and prepare water-soluble PANI nanoparticles (PANI_NPs), ZnO_NPs, and hybrid ZnO_NPs/PANI (using sodium dodecyl sulfate as surfactant), and then discuss the characterization of these systems by use of zeta-potential measurements, UV–visible spectrophotometry, infrared spectroscopy, X-ray diffraction, and scanning electron microscopy (SEM). The electrospinned fibers of ZnO_NPs/PANI HION exhibit a light-sensitive electrical charge transport. We suggest that the development of photodetectors operating in the UV region may be one of the possible uses of these fibers.

EXPERIMENTAL

Chemicals

The aniline (C_6H_7N, VETEC, Brazil) monomer was distilled under vacuum and, subsequently, stored in a dark recipient under refrigeration until use. All other reagents of analytical grade, zinc nitrate hexahydrate [$Zn(NO_3)_2.6H_2O$, VETEC, Brazil], ammonium persulfate [$(NH_4)_2S_2O_8$, Merck, Brazil], chloridic acid (HCl, Quimex, Brazil), and sodium dodecyl sulfate (SDS, Sigma–Aldrich, USA), were used with no further purification.

Synthesis of ZnO_NPs, ZnO_NPs/PANI HION, and PANI_NPs

To obtain ZnO_NPs, we added 25 mL of a $0.5M$ $Zn(NO_3)_2.6H_2O$ solution, slowly and under vigorous stirring, to 25 mL of a $1.0M$ NaOH solution, which was prepared at 80°C, and maintained the temperature and the stirring for 2 h.[26] After this period of time, the white precipitate formed was collected in a filter paper and generously washed with ethanol and, then, deionized water in abundance. The resulting
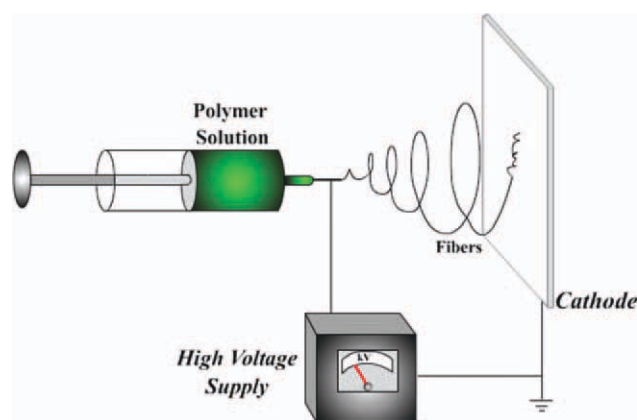

Figure 1 Schematic representation of the main components of an electrospinning apparatus. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

material was dried at a temperature of 60°C during 48 h and finally stored under vacuum.

The ZnO_NPs/PANI HIONs were prepared by a direct *in situ* chemical polymerization of aniline.[25] For this, initially a small amount (35 mg) of dried ZnO_NPs and 0.7 g of SDS were added to 20 mL of a $0.1M$ HCl aqueous solution, and then the resulting mixture was stirred at room temperature for 20 min, before the introduction of 50 mM of aniline. Finally, 2 mL of a $0.1M$ HCl solution containing 30 μM of the oxidant [$(NH_4)_2S_2O_8$] was slowly added, and stirring continued for 24 h. No precipitate was observed during the preparation of any of the colloidal dispersions described above, and a final pH of 1.8 was found for all samples. A similar procedure, but this time without the introduction of ZnO_NPs, was observed for the synthesis of pure PANI_NPs.

Preparation of the ZnO_NPs/PANI fibers

ZnO_NPs/PANI composites in the form of fibers were prepared by the electrospinning technique.[23] For this, 1 mL of a ZnO_NPs/PANI aqueous solution was placed in a hypodermic syringe whose needle was connected to copper foil cathode maintained at 15 kV and situated at 10 cm from the tip of the needle (see the corresponding scheme in Fig. 1). The resulting fibers were collected on a Petri dish and dried under vacuum at room temperature.[27]

Characterization

The colloidal samples were placed in quartz cuvettes, and their absorption spectra in the 300–900 nm range were collected by use of a Cary 5E spectrophotometer (Varian, Australia) with an optical length of 1 cm. For the identification of the relevant vibrational modes, samples were prepared in the
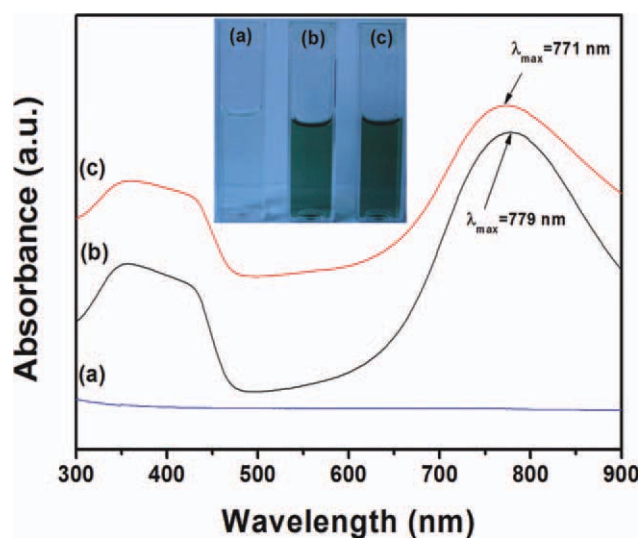
Figure 2 Absorption spectra of ZnO_NPs (a), PANI_NPs (b), and ZnO_NPs/PANI (c) colloidal dispersions. Insets: photograph of the corresponding colloidal dispersions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

form of KBr pellets, and the corresponding spectra were collected in an ABB FTLA 2000 Fourier transform infrared spectrometer (Bomem, Canada) operating in the 4000 cm^{-1} to 400 cm^{-1} region, with a resolution of 4 cm^{-1} and at a rate of 200 sweeps/spectrum.

A ZS90 NanoZetasizer instrument (Malvern, UK) was used to investigate the colloidal samples dispersed in water at a temperature of 25°C; size was determined by the dynamical light scattering of a λ = 633 nm laser at a 90° scattering angle, whereas the electrophoretic method was used for the determination of the zeta-potential of the particles. All reported results correspond to the average of three independent measurements.

The crystalline structure of powdered samples was analyzed in a D5000 diffractometer (Siemens AG, Germany), using the CuKα (λ = 1.54) radiation. The patterns were recorded in the 2θ range from 30° to 70°, in steps of 0.02 and counting time of 1 s per step.

We have used a JSM-5900 (JEOL, Japan) SEM to investigate the morphology of the samples, which were mounted on a small glass slide and covered with a thin gold surface by use of a SDS050 Bal-Tec metallizer (Japan). We obtained qualitative information about the chemical composition of the samples by use of an energy dispersive spectroscopy (EDS) accessory coupled to the JSM-5900 instrument.

The current–voltage (I–V) curves of fibers ZnO_NPs/PANI were recorded on an HP 4155A semiconductor analyzer at room temperature, after applying silver conducting paint to establish the electrical contacts disposed with an average separation of 1 mm.

RESULTS AND DISCUSSION

UV–vis spectrophotometry

In Figure 2, we present the corresponding absorption spectra for the SDS colloidal dispersions of ZnO_NPs, PANI_NPs, and ZnO_NPs/PANI nanocomposites. The PANI_NPs absorption spectrum reveals the presence of three bands, in agreement with well-known results for the emeraldine salt form of doped PANI;[28] the first one is centered at 350 nm and associated to the π–π* electronic transitions in the quinoidal and benzenoid aromatic rings, whereas the second (at 425 nm) and the third (at 779 nm) features are related to the polaronic bands of lower and higher energy, respectively.[29] For the ZnO_NPs/PANI composites, a hypsochromic shift of the lower energy band to 771 nm can be taken as an indication that in the hybrid structures the polymer is in its lower oxidation state.[29] Note, however, that while Hosono et al.[9] identified an absorption band centered at 380 nm for ZnO_NPs in an ethanolic medium, no similar absorption bands were observed in this work (which describes the synthesis of ZnO_NPs in aqueous solution).

Dynamic light scattering and zeta potential measurements

As noted before, all colloidal dispersions presented a good stability, with no evidence of precipitates or formation of agglomerates. The average diameters of the pure PANI colloidal particles and of the ZnO_NPs in solution were determined to be of the order of 3.6 nm and 4.6 nm, respectively, but a larger value (8.1 nm) was found for the hybrid ZnO_NPs/PANI composites. Therefore, it is reasonable to suppose that ZnO_NPs are enveloped by a PANI shell. For the zeta-potential, the average values obtained for ZnO_NPs (−42.88 mV), PANI_NPs (−66.05 mV), and ZnO_NPs/PANI composite (−46.68 mV) aqueous solutions indicate the presence of well-defined Gouy-Chapman layers that confers stability to the colloidal suspensions.[30]

X-ray diffraction

The corresponding X-ray diffraction patterns are shown in Figure 3. For ZnO_NPs, the main 2θ features are present at 31.8° (d = 0.282 nm), 34.4° (d = 0.260 nm), 36.2° (d = 0.247 nm), 47.5° (d = 0.191 nm), 56.6° (d = 0.162 nm), 62.9° (d = 0.147 nm), 66.4° (d = 0.141 nm), 67.9° (d = 0.137 nm), and 69.1° (d = 0.136 nm), which correspond to the (100), (002), (101), (102), (110), (103), (200), (112), and (201) Bragg reflections, respectively. These results are consistent with a ZnO nanocrystalline hexagonal structure, in agreement to the literature.[6,31,32] For pure PANI_NPs
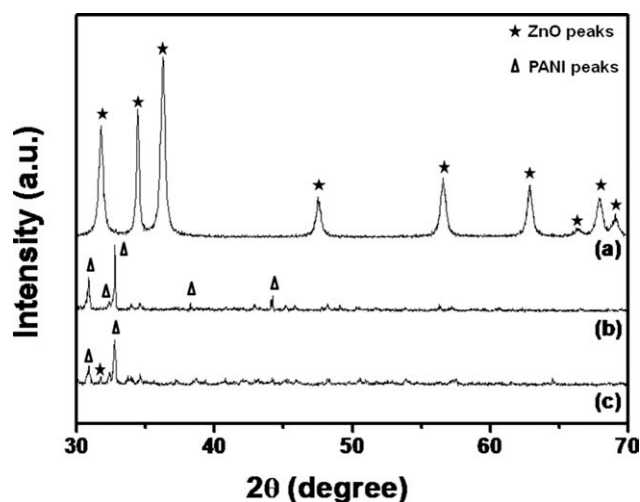
Figure 3 Representative X-ray diffractogram of ZnO_NPs (a), PANI_NPs, (b) and ZnO_NPs/PANI (c) powdered samples.

samples, the principal peaks are observed at $2\theta = 30.8°$, $32.3°$, $32.7°$, $38.2°$, and $44.3°$, which correspond to the existence of (202), (020), (120), (310), and (130) reflections of the pure emeraldine organized in an orthorhombic structure ($a = 7.65$ Å, $b = 5.75$ Å, and $c = 10.22$ Å).[33,34] Finally, we have identified peaks at $2\theta = 30.8°$, $31.8°$, $32.3°$, and $32.7°$ in the ZnO_NPs/PANI diffractogram, in an indication that the PANI crystalline structure is essentially retained. Possibly because the ZnO_NPs are well embedded in a PANI envelope, we were not able to identify all of the Bragg reflections seen in the pure ZnO_NPs but only the presence of the $2\theta = 31.8°$ peak (corresponding of the (100) reflections), in a situation similar to that reported by Tang et al.[35] in their study of preparation and properties of maghemite/PANI nanocomposites. Also, we must recall that the ZnO_NPs/PANI systems were prepared using very small amounts of ZnO_NPs, relative to those of aniline (0.43 mM/50.0 mM).

FTIR spectroscopy

In Figure 4, we present the infrared spectra of KBr pellets containing pure PANI_NPs and ZnO_NPs/PANI HION. The characteristic PANI peaks at 1469 cm^{-1} and 1560 cm^{-1} are assigned to C=C stretching of the benzenoid and quinoid rings, respectively. As observed for emeraldine salts, the benzenoid band at 1469 cm^{-1} is more intense than that of the quinoid band at 1560 cm^{-1}. In the two types of nanostructures, one can note the presence of a band at 2925 cm^{-1}, which corresponds to the symmetric and asymmetric stretching of the —CH$_2$— groups, respectively.[36] However, the N—H stretching vibration band (3210 cm^{-1}) appears in the FTIR spectrum of PANI.[36] Although the peak at 1139 cm^{-1} for the

PANI_NPs and ZnO_NPs/PANI systems is related to the plane bending vibration of C—H, which is formed during protonation,[37] from the presence of the peak at 1224 cm^{-1} (which is associated to the in-plane C—N stretching mode), we can infer that the PANI chains are in their conducting (i.e., emeraldine salt) form.[38] The observation of a vibrational band in the 400–600 cm^{-1} region[39] for the ZnO_NPs/PANI composites suggests the presence of a core of ZnO_NPs in the hybrid structures, whereas the bands at 3200–3600 cm^{-1} correspond to the —OH mode of vibration on the ZnO surface.[40] The stretching mode of vibration of C=O is observed at 875 cm^{-1} and 1730 cm^{-1}. We note, however, that for the ZnO_NPs/PANI nanocomposites, the characteristic peaks of PANI at 1560, 1469, 1224, and 1139 cm^{-1} are shifted to 1457, 1386, 1251, and 1095 cm^{-1}, a fact that can be attributed to an interaction between the ZnO_NPs surface and the N—H groups in the polymeric chains.[41] Also, the band at 1078 and 590 cm^{-1} was ascribed to the absorption of SO$_3^-$ groups, suggesting that PANI is also doped with a little SDS.[42,43]

Scanning electron microscopy

In Figure 5, we show SEM images of the surface of the films obtained by depositing the colloidal dispersions, and after evaporation of the solvent. In Figure 5(a), one can observe the formation of a homogeneous film of PANI particles, whereas in the Figure 5(b), we present a SEM image of freshly prepared ZnO_NPs samples, where rod-like structures suggestive of the existence of a wurzite order[44] can be identified. However, as it can be seen in Figure 5(c), a more fibrous structure seems to be associated to the ZnO_NPs/PANI composites.
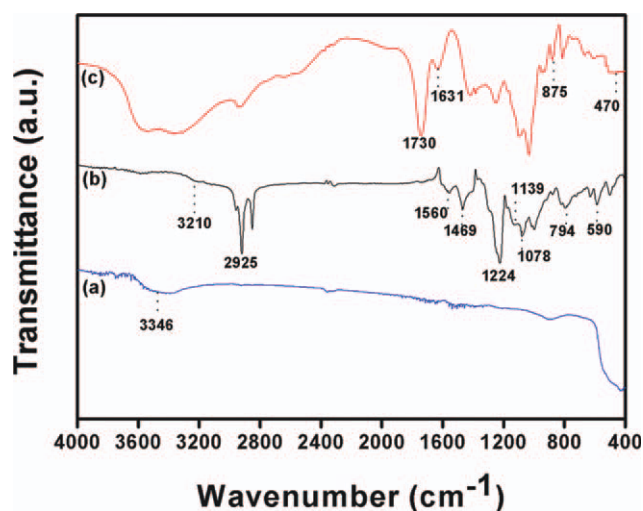

Figure 4 FTIR spectra of pure ZnO_NPs (a), pristine PANI_NPs (b), and ZnO_NPs/PANI composites (c). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
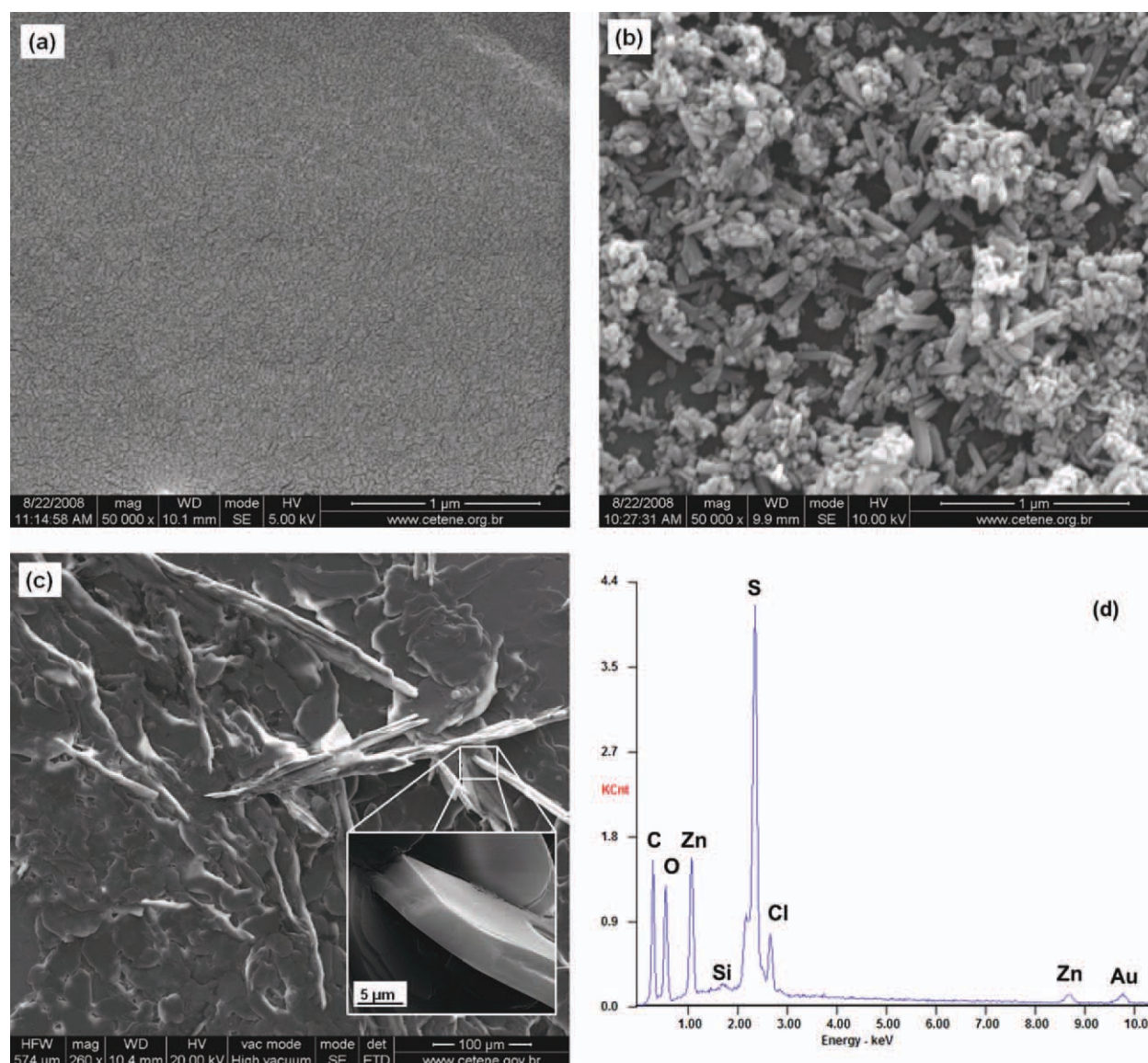
Figure 5 SEM images of pristine PANI_NPs (a), pure ZnO_NPs (b), ZnO_NPs/PANI composites (c), and EDS spectrum of ZnO_NPs/PANI composites (d). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

An EDS analysis of the latter [Fig. 5(d)] can provide relevant information about their chemical composition: while the gold and silicon peaks are associated to the metallization process and to the glass substrate used, respectively, the intense Zn and O peaks confirm the presence of ZnO_NPs in the hybrid nanostructure. We have also used SEM techniques to investigate the structure of the ZnO_NPs/PANI fibers prepared by electrospinning. The typical form of the fibers is shown in Figure 6.

Electrical characteristics

Considering, as a first approximation, each fiber as a cylinder of effective length L and cross sectional area A, we have estimated the corresponding resistivity by direct use of Ohm's law, that is, $\rho = (A/L)V/I$.

We examined the electrical characteristics of the ZnO_NPs/PANI fibers prepared by electrospinning, before and after we doped them by a 5 min exposure to HCl vapor. A characteristic I–V curve for a doped ZnO_NPs/PANI fiber, shown in Figure 7, should be compared to the corresponding curve for undoped ones (inset). An ohmic behavior was identified in both cases, even though a four-order of magnitude decrease in the resistivity (from 50 kΩcm to 3.3 Ωcm) is observed to follow the acid doping of the fibers.

The "doping" of a conducting polymer consists in its treatment with either electron acceptor molecules (oxidation) or electron donor molecules (reduction)

that introduce charged conformational defects in the polymeric backbone: the resulting polymer is then a p-type or a n-type one, respectively.[45] [Note that, as usual, the term "doping" is used here in analogy with the lexicon of inorganic semiconductors, where, for example, silicon is treated with (relatively much smaller amounts) of "impurities" such as arsenic, boron, or phosphorus]. PANI, in special, can become more conductive after protonation (acidic "doping"); a process that consists in the treatment of its emeraldine base variety with a strong acid (ex: HCl) that induces the protonation of the imine sites to form the emeraldine salt. In fact, when ZnO_NPs/PANI fibers were exposed to HCl, a rapid drop in resistance is observed within a short period of time. Although the charge introduced along the backbone by this protonation must be counter balanced by the resulting negatively charged chloride counterions, a drastic change in conductivity is brought about by the formation of polarons (radical cations) that can travel along the polymer backbone.[46] We also noticed that the charge transport along the fibers is slightly affected by UV illumination, with a current increase in both forward and reverse bias conditions, a result similar to what was previously found for single ZnO/PANI hybrid layers.[47] We note that Lin et al., in their study of this density–voltage characteristics of Au/n-type ZnO and Au/PANI/n-type ZnO devices,[48] have reported that PANI films deposited by the technique or spin coating on ZnO films prepared by the sol–gel method exhibit a rectifying behavior.

CONCLUSIONS

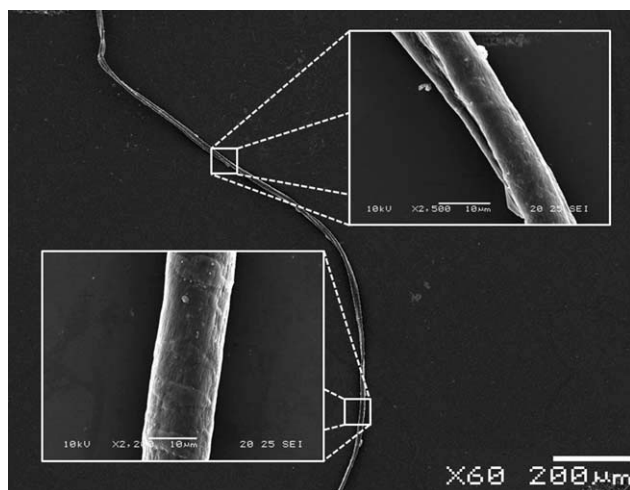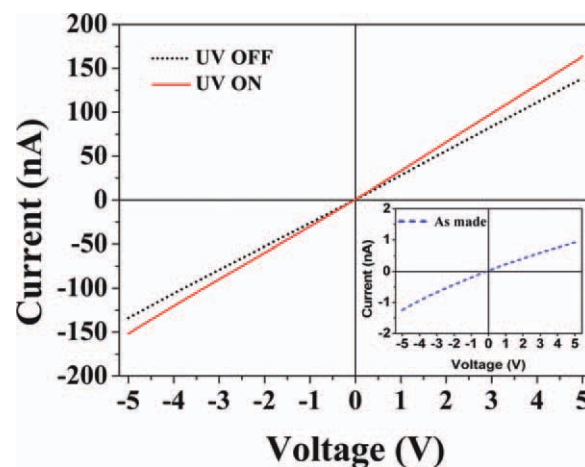We have described an easy and effective route of synthesis of water-soluble ZnO nanostructures dis-

Figure 7 Typical current–voltage profile curve of one HCl doped ZnO_NPs/PANI fiber under UV illumination and in the dark. Inset: current–voltage profile curve of one pristine ZnO_NPs/PANI fiber. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

persed as highly colloidal solutions that allow for the future development of systems with similar functionality. We discuss the preparation of pure ZnO_NPs, pure PANI_NPs, and ZnO_NPs/PANI HION that exhibit well-defined crystalline order and, as an important characteristic, present a high solubility in aqueous media. Both light scattering and zeta-potential measurements confirm that the nanostructures obtained by the emulsion polymerization of aniline in presence of the ZnO_NPs and SDS form stable and monodisperse colloidal solutions. SEM images indicate that the ZnO_NPs are rod-like in shape, whereas the ZnO_NPs/PANI composite samples possess a more fibrous structure. FTIR results suggest that H-bonds were established between the ZnO_NPs and the PANI chains in the HIONs. We were successful in fabricating of ZnO_NPs/PANI fibers by use of the electrospinning technique, and we have shown that the conductivity of the resulting fibers was strongly affected by the doping to HCl vapor, when a four orders of magnitude decrease in the resistivity was observed. Electrical measurements indicate that the ZnO_NPs/PANI structures exhibit a light-sensitive ohmic behavior that could be explored into new applications in the area of UV photodetection, a hypothesis that we are at present investigating in a more detailed manner. Alternatively, these nanocomposites could serve as promising active materials for the development of economical water-based light-sensitive paints and surface coatings. Furthermore, these results also indicate clear avenues for future development of engineered fibers for use in electronic device and sensors.

Figure 6 SEM image of one ZnO_NPs/PANI fiber prepared by electrospinning.

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