



Transition from positive to negative electrical resistance response under humidity conditions for PEDOT:PSS-MoS₂ nanocomposite thin films

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Abstract

Here, we report the preparation of PEDOT:PSS-MoS₂ nanocomposite thin films and their electrical resistance response under humidity conditions revealing their promising properties as humidity sensor materials. One of the most interesting features of our samples is the transition from positive to a negative electrical resistance response to humidity conditions with increasing MoS₂ additions. Our confocal Raman imaging studies revealed that the presence of MoS₂ yields a local charge rearrangement in the thiophenyl rings of PEDOT:PSS, in relation to the enhancement of the electrical resistance negative response as observed by impedance spectroscopy analysis. The enhancement on the negative response with increasing MoS₂ additions could be explained through the increment of hole carriers in MoS₂ nanosheets under humidity conditions, thus leading to an enhancement in the electrical transport along the PEDOT:PSS chains.

1 Introduction

Many technological advances that are taking place in these last years, rely on the capacity of materials to have transducing properties that could be used in sensing devices. Through this operation such devices allow the development of automatism for production and services and furthermore the ability to survey information from the surroundings in medical prosthetics or any other application where remote collection of environmental data would be required [1, 2]. Typical properties of interest to be detected and quantitatively assessed are temperature, pressure and ambient humidity [3, 4]. In the case of sensing humidity, data acquisition is

relevant in order to prevent damage in electronic or mechanical devices to avoid injuries in biological tissues, where the ability of sensing was previously damaged. Among the main materials used for this purpose, graphene-polymer junction [5], supramolecular nanofiber [6] and CNT/poly(vinyl alcohol) (PVA) bilayer [7] could be listed. Previous reports about PEDOT:PSS and MoS₂ electrical response to humidity conditions study the potential ability of these materials to sense environmental humidity [8–12]. It has been already observed that pure PEDOT:PSS films showed a positive response of the electrical resistance up to 78% humidity conditions [13]. On the other hand, it has been also observed that bulk MoS₂ showed almost no significant electrical resistance response when exposed to humidity conditions up to 80% [14]. However, in the same study the exfoliation of MoS₂ nanosheets lead to a small but noticeable decrease in the resistance with increasing humidity (negative response) in a broad range from 0% to 80% humidity conditions [14]. It has been also already envisaged by means of DFT calculations that water molecules can act as electron acceptors when adsorbed to MoS₂ surface, thus generating a p-type doping of MoS₂ [15, 16]. It has been already observed that the adsorption of water molecules on both pristine PEDOT:PSS and MoS₂ structures lead to a modification of the electronic properties such as the band gap and HOMO–LUMO energies [17, 18]. Up to now, both PEDOT:PSS and MoS₂ were used separately in humidity sensing applications, both with their own advantages

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and disadvantages. In order to further study the synergistic properties of PEDOT:PSS and MoS₂ nanosheets, we have prepared thin films using PEDOT:PSS as host matrix with different MoS₂ nanosheets loadings to evaluate their electrical response by means of impedance spectroscopy under environmental humidity.

2 Experimental procedure

We undertook the task of preparing PEDOT:PSS-MoS₂-X nanocomposite thin films, where X stands for the weight percentage (% w/w) of MoS₂ in each sample. The samples prepared correspond to X = 5, 10 and 20 and a X = 0 specimen, i.e. pure PEDOT:PSS (purchased from Sigma Aldrich) was used as a reference. MoS₂ was prepared by hydrothermal synthesis using thiourea and MoO₃ as precursors. MoS₂ precursor was obtained from (NH₄)₆Mo₇O₂₄·4H₂O heated at 450 °C for 2 h. Thiourea and MoO₃ were suspended in 80 mL of water and transferred to a 100 mL Teflon lined autoclave with 120 rpm stirring at 185 °C for 24 h. The obtained MoS₂ specimens were then washed several times with water and ethanol and finally the precipitate was dried at 70 °C overnight [19]. Transmission electron microscopy (TEM) images of MoS₂ nanosheets are shown in Fig. 1. The as-obtained MoS₂ under powder form was suspended together with PEDOT:PSS in distilled water, and all the suspension was sonicated at 30 W for half an hour with a pointer sonicator. Each suspension was deposited on glass substrates through a spin coating procedure (1500 rpm, 15 s, twice) to form our PEDOT:PSS-MoS₂-X specimens with X = 0, 5, 10, 15 and 20 thin film samples.

Grazing incidence small angle X-ray scattering (GISAXS) was performed using a Rigaku Ultima IV diffraction system working in parallel beam configuration with CuK α radiation in the q range 0.007–0.10 Å⁻¹ and applying an offset angle of 0.2° to the main specular reflection determined from a rocking curve. Confocal Raman images and Raman spectra were obtained using WITec Alpha 300-RA. Raman spectra for PEDOT:PSS-MoS₂-X nanocomposites were collected using a 532 nm laser excitation wavelength, with power adjusted at ~ 3 mW to avoid polymer decomposition. For each case, a set of 20 spectra with 0.2 s integration time were averaged. *In-depth* confocal Raman images were obtained for an 8 × 8 μm² cross-section with a resolution of ~ 265 × 265 nm² per pixel, totalizing 900 Raman spectra per image. *In-plane* confocal Raman images were obtained for a 20 × 20 μm² total area with a resolution of ~ 265 × 265 nm² per pixel, totalizing 5625 Raman spectra per image. AC impedance spectroscopy was collected for PEDOT:PSS-MoS₂-X thin films specimens using a two-probe in-plane configuration with a 5 mm separation between silver paste contacts. AC impedance spectroscopy was collected at T = 300 K using a 100 mV A.C. voltage amplitude in the f = 0.1 Hz–1 MHz frequency range. AC impedance spectroscopy data was collected at 75% humidity conditions and also at typical environmental humidity condition (50%) which was set as reference and the chamber humidity was measured with a standard commercial humidity sensor with a 0.1% resolution. We chose 50% humidity as a reference for typical environmental humidity conditions and 75% humidity as the threshold humidity condition above which the films are damaged due to the high humidity, as similarly observed for other PEDOT:PSS films [13, 14].

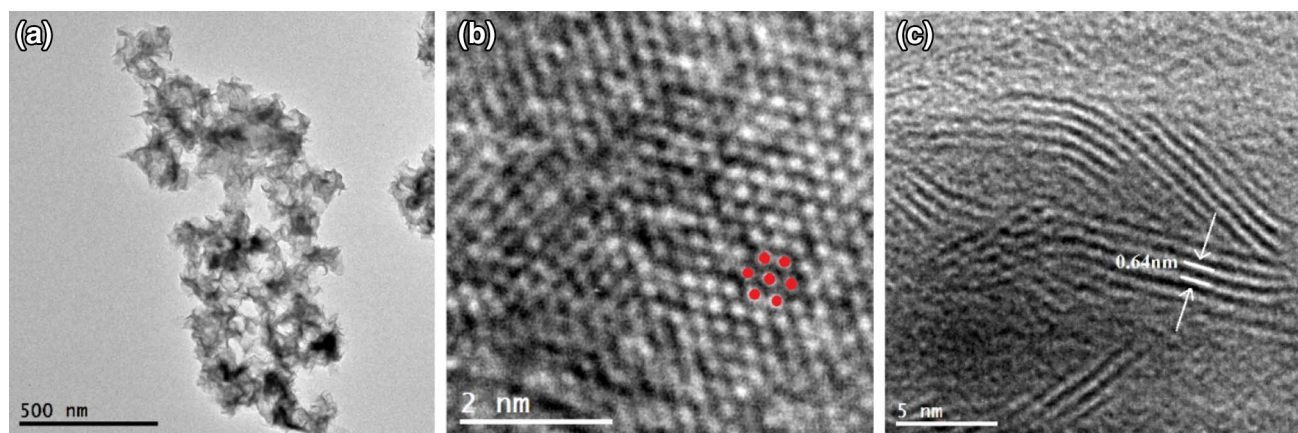


Fig. 1 **a** Transmission electron microscopy (TEM) images for MoS₂ nanosheets and high resolution transmission electron microscopy (HR-TEM) images for **b** the hexagonal crystalline array of atoms

(marked with red spots) and **c** the interlayer distance between layers (marked with white arrows). (Color figure online)

3 Results and discussion

Transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HR-TEM) images of MoS₂ nanosheets are shown in Fig. 1. The morphology of MoS₂ nanosheets showed a flower-like microstructure as shown in Fig. 1a. The hexagonal crystalline array in the plane of MoS₂ nanosheets and the interlayer distance between MoS₂ nanosheets is clearly evidenced in Fig. 1b, c, respectively. Figure 2b shows the evolution of GI-SAXS patterns for PEDOT:PSS films with different MoS₂ additions. Unloaded PEDOT:PSS profile shows a well-defined peak at $q_{\text{max}} = 0.033 \text{ \AA}^{-1}$ in relation to a $d = 2\pi/q_{\text{max}} = 20 \text{ nm}$ mean coherence length associated to the self-organization among the polymer chain fibers [20]. The addition of MoS₂ sheets lead to a shift in the maximum peak to 0.04–0.05 $\text{\AA}^{-1}$ which is suggesting a decrease on the mean coherence length among polymer fibers to $d \sim 13\text{--}16 \text{ nm}$, as similarly observed

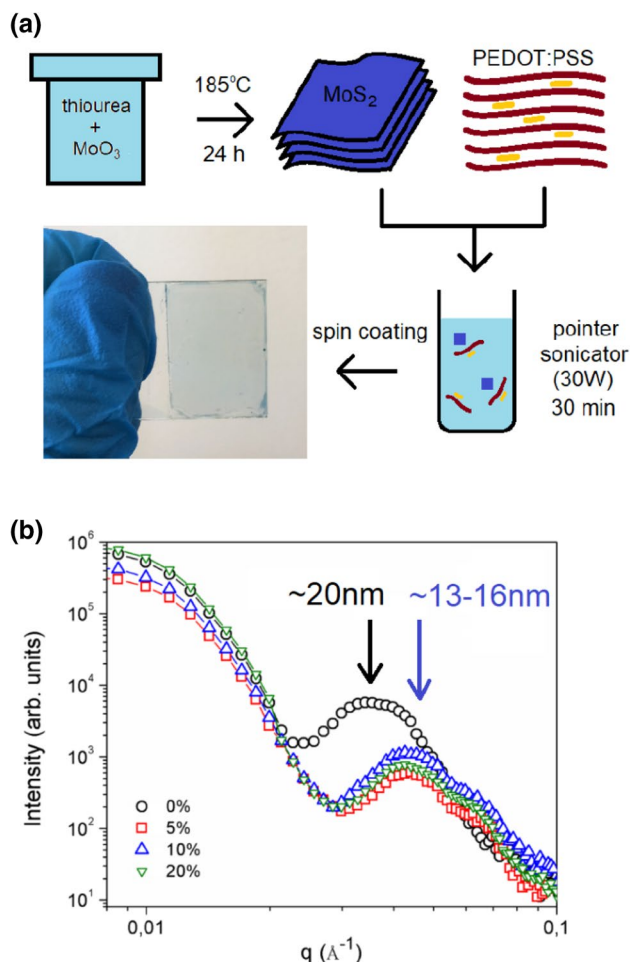


Fig. 2 **a** Pictorial scheme of the synthesis procedure and picture of our PEDOT:PSS-MoS₂-X thin film devices. **b** Grazing incidence small-angle X-ray scattering (GI-SAXS) for X=0, 5, 10 and 20%

with the addition of carbon nanotubes fillers [20]. In the present case, MoS₂ nanosheets could be interacting with PEDOT:PSS chains leading to an increase in the agglomeration of the polymer chains that could be explaining the decrease in the mean coherence lengths.

In-depth and in-plane confocal Raman images for a selected PEDOT:PSS-MoS₂-X with X=20% thin film specimen are shown in Fig. 3a,b. The images were obtained by filtering the Raman using the relative intensity maximization of the peak associated to the C=C stretching mode (at $\sim 1440 \text{ cm}^{-1}$) of PEDOT:PSS and the peak associated to the A_{1g} mode (at $\sim 400 \text{ cm}^{-1}$) of MoS₂ to obtain PEDOT:PSS-rich and MoS₂-rich zones. In-depth confocal Raman microscopy images (Fig. 3a) showed similar homogeneous thicknesses in the 1–2 μm range for all thin film specimens, as can be observed in Fig. S1 (Supporting Information). In-plane confocal Raman microscopy images (Fig. 3b) showed the presence of MoS₂ nanosheets clusters embedded in the PEDOT:PSS host structure. Additional in-plane confocal Raman microscopy images discussing the MoS₂ nanosheets distribution in the PEDOT:PSS matrix is shown in Fig. S2 (Supporting Information). Confocal Raman spectra for selected PEDOT:PSS-rich and MoS₂-rich regions of the sample are shown in Fig. 3c. The presence of typical PEDOT:PSS Raman modes was clearly evidenced, especially in PEDOT:PSS-rich region. The most relevant Raman peaks ascribed to PEDOT:PSS are those

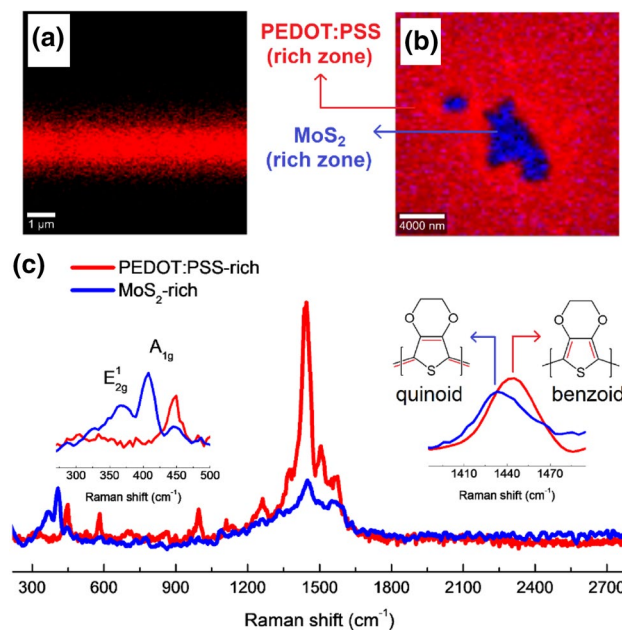


Fig. 3 **a** In-depth and **b** in-plane confocal Raman images for PEDOT:PSS-MoS₂-X with X=20%. **c** Confocal Raman spectra for PEDOT:PSS-rich and MoS₂-rich zones of PEDOT:PSS-MoS₂-X with X=20% showing an inset of Raman peaks ascribed to MoS₂ and Raman peak shifting for benzoid-to-quinoid transition in the MoS₂ rich zone

located at 700 cm^{-1} , that could be attributed to C–S–C bending, at 990 cm^{-1} related to C–C antisymmetric stretching, at $1100\text{--}1135\text{ cm}^{-1}$ associated to C–O–C stretching overlapped with PSS sulfonate stretching, at 1258 cm^{-1} ascribed to C–C stretching, at 1366 cm^{-1} in relation to C–C inter-ring stretching, at $1434\text{--}1444\text{ cm}^{-1}$ associated to C=C symmetric stretching of the aromatic modes and two at 1509 and 1567 cm^{-1} attributed to C=C asymmetrical stretching modes, respectively [21, 22]. On the other hand, MoS₂ most relevant Raman modes are located at 370 and 408 cm^{-1} , ascribed to E_{2g} and A_{1g} vibrational modes, respectively, as it can be seen in the left inset of the Fig. 3c [23]. One of the most relevant features of MoS₂-rich region Raman spectra is a clear redshift of the peak associated to the C=C symmetric stretching of the aromatic mode from 1444 to 1430 cm^{-1} , which is typically attributed to a transition from a benzoid to a quinoid PEDOT configuration [20], as shown in the right inset of Fig. 3c. This reveals that the presence of the dichalcogenide yields a charge rearrangement in the thiophenyl ring, that could allow conduction along these rings chains by means of the π cloud.

A brief scheme on the set up of our impedance spectroscopy measurements under humidity conditions is shown in Fig. 4. The total impedance response is described by the following equation:

$$Z = Z' - jZ''$$

where Z' and Z'' are the real and imaginary components of the impedance, respectively. Nyquist plots ($-Z''$ vs. Z') of the impedance response for $X=0, 5, 10$ and 20 , under humidity conditions (75%) in addition to reference humidity conditions (Ref=50%) are presented in Fig. 5a. The difference in the magnitude for both impedance data sets is so remarkable that an inset graph was necessary for impedance data under 75% humidity conditions for loaded MoS₂ films, in order that both Nyquist plots could be seen in detail. The impedance data of our films were fitted with a circuit model based in the parallel combination of a resistor (R) and a constant phase element (CPE) connected in serie with a Warburg (W) diffusion element, as schematized in Fig. 4. The CPE impedance can be described by the following equation:

$$1/Z_{\text{CPE}} = Y_0(j\omega)^\alpha$$

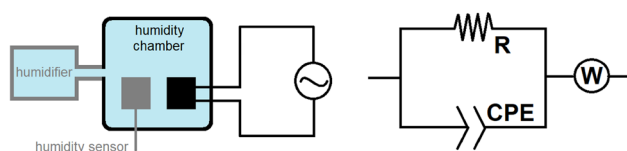


Fig. 4 Scheme of the AC impedance measurements setup and equivalent circuit model used for fitting impedance data for our PEDOT:PSS–MoS₂–X

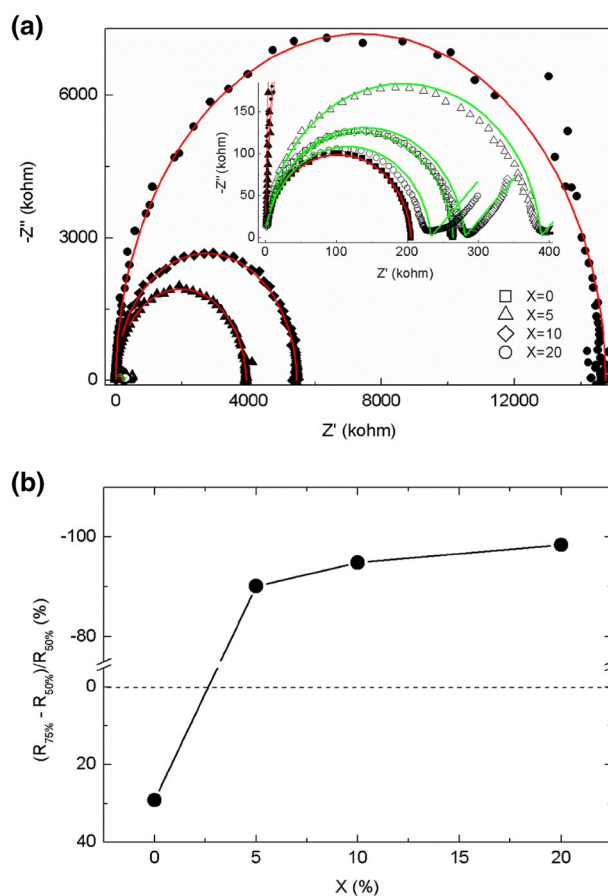


Fig. 5 **a** Nyquist plots and circuit model fittings for $X=0, 5, 10$ and 20% films, under humidity environment condition (75% humidity, hollow shapes) and the standard humidity conditions (50% humidity, full shapes). **b** Resistance response expressed as $100 \cdot (R_{75\%} - R_{\text{ref}})/R_{\text{ref}}$ as a function of MoS₂ content

where ω is the frequency, Y_0 is the numerical value of admittance at $\omega = 1\text{ rad s}^{-1}$ and α is an exponential factor. In the case of $\alpha = 1$, the CPE resembles an ideal capacitor and $Y_0 = C$ [24]. The fitting parameters for all samples at standard and 75% humidity conditions are summarized in Table 1. No relevant variations were observed for the CPE fitted parameters such as α values very close to unity ($\alpha \sim 0.96\text{--}0.99$) suggesting no drastic deviations from an ideal capacitor and a slight increase in Y_0 values (from ~ 10 to $20 \times 10^{-12}\text{ S s}^\alpha$) when exposed to 75% humidity conditions respect to standard humidity conditions. The estimation of the frequency of the Nyquist plots maximum (f_{max}) as an approximation to the relaxation frequency showed a clear decrease with increasing MoS₂ loadings from ~ 100 to 2 kHz for standard humidity conditions. This is probably due to the increase of the weight fraction of MoS₂ nanosheets, which presents lower conductivity, as similarly observed for PEDOT films heavy PSS doped [25]. However, a slight decrease for $X=0$ (from 100 to 80 kHz) and

Table 1 AC impedance most relevant parameters for PEDOT:PSS-MoS₂-X with X=0, 5, 10 and 20 compositions at 50% (left) and 75% humidity conditions (right) separated with an arrow (→)

	R (k ohm)	Y_0 (10^{-12} S s ^a)	α	$f_{\max}$ (kHz)
X=0	204.8 ± 0.5 → 264.5 ± 0.6	10.5 ± 0.5 → 18.3 ± 0.7	0.976 ± 0.003 → 0.967 ± 0.003	~ 100 → 80
X=5	3929 ± 10 → 387 ± 1	9.6 ± 0.2 → 15.3 ± 0.6	0.988 ± 0.002 → 0.966 ± 0.003	~ 5 → 40
X=10	5451 ± 14 → 280 ± 1	9.6 ± 0.2 → 22.0 ± 0.9	0.988 ± 0.001 → 0.958 ± 0.003	~ 3 → 45
X=20	14.750 ± 40 → 232 ± 1	6.0 ± 0.1 → 20.5 ± 0.9	0.993 ± 0.001 → 0.955 ± 0.003	~ 2 → 50

Fitting parameters with corresponding errors were obtained from the circuit model shown in Fig. 3 and $f_{\max}$ was estimated from the frequency at the maximum of the semicircle arc of the Nyquist plots

a notorious increase for $X > 0$ (from 5 to 2 to 40–50 kHz) from standard to 75% humidity conditions, respectively. In one hand, the decrease of the relaxation frequency for pristine PEDOT:PSS could be due to the decreasing interaction between PEDOT chains by the water uptake under high relative humidity [13]. On the other hand, the increase of the relaxation frequency for those PEDOT:PSS films with MoS₂ loadings is suggesting that its presence promotes faster charge transfer with increasing humidity. In agreement with the previous observation, the fitted resistance at standard humidity conditions showed a drastic increment from ~ 205 to 14,750 k ohm when increasing the MoS₂ additions. Although our Raman analysis discussed earlier evidenced a benzoid to quinoid conformation transition of thiophenyl rings when increasing the MoS₂ weight fraction. It has been already observed that PEDOT:PSS films presents a decrease in the conductivity with the addition of MoS₂ nanosheets, when increasing from 0 to 20% weight fractions [26]. This decrease in the electrical conductivity at standard humidity conditions is mainly caused by the separation of the conductive PEDOT chains due to the intercalation of MoS₂ nanosheets [26]. However, the most relevant changes in the fitted parameters is that $X=0$ showed a small increment in the resistance (R) when exposed to 75% humidity conditions while $X=20$ showed an almost three-orders-of-magnitude decrease in the resistance (R) from ~ 14.75 M ohm down to ~ 232 k ohm for standard and 75% humidity conditions, respectively. Figure 5b shows the response of the electrical resistance under 75% humidity respect to standard humidity ($R_{\text{ref}} = 50\%$) conditions expressed as $\text{Response (\%)} = 100 \cdot (R_{75\%} - R_{\text{ref}}) / R_{\text{ref}}$ as a function of the MoS₂ content in the composites. It is important to remark that measurements in both conditions for each sample were obtained with the same electrical contacts configuration to make reliable the comparison between measurements for the same specimen. One of the most interesting features of our samples is a clear transition from positive to a negative response with increasing MoS₂ additions respect to MoS₂ unloaded PEDOT:PSS samples.

It has been already observed that pure PEDOT:PSS films showed a positive response (response ~ + 100%) of

the electrical resistance with increasing humidity from 50 to 75% [13] and exfoliated MoS₂ nanosheets showed a small negative electrical resistance response from 50 to 75% humidity [14]. It has been already observed that the adsorption of water molecules on both pristine PEDOT:PSS and MoS₂ structures lead to a modification of the electronic properties such as the band gap and HOMO–LUMO energies [17, 18]. In particular, the adsorption of water molecules can act as electron acceptors when adsorbed to MoS₂ surface, thus generating a p-type doping of MoS₂ [15, 16]. In our case, we observed a drastic increase on the negative electrical resistance response of PEDOT:PSS with increasing MoS₂ additions up to response ~ – 100% for $X=20$ from 50 to 75% humidity. This drastic enhancement on the electrical resistance negative response could be in possible agreement with MoS₂ p-type doping when exposed to humidity conditions. In this scenario, the hole carriers generated in MoS₂ nanosheets, due to the water molecules adsorption, are plausible to be injected into the PEDOT:PSS chains, which is also well known as a hole conducting polymer [27, 28].

4 Conclusions

In the present report, we show the preparation of PEDOT:PSS-MoS₂-X thin film nanocomposites studying its structure, microstructure and electrical response to humidity conditions. Grazing incidence X-ray scattering showed a slight decrease of the mean correlation length between polymer fibers and confocal Raman images revealed electronic interactions between MoS₂ nanosheets and PEDOT:PSS polymer yielding to modifications of the benzoid-to-quinoid structure. At high humidity conditions (75%) an interesting transition from positive to negative electrical resistance response was observed with increasing MoS₂ loadings. We have concluded that the increasing environmental humidity enhance the conductivity of PEDOT:PSS-MoS₂-X thin film nanocomposites and that the dichalcogenide addition plays a role by promoting a charge rearrangement in the thiophenyl ring of PEDOT. We think that the transition from positive to negative electrical resistance response in these

PEDOT:PSS-MoS₂ nanocomposites could have a special significance to humidity sensor materials due to the fact that the electrical resistance response changes drastically (almost three-orders of magnitude) for relatively low amounts of MoS₂ nanosheets loadings.

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