

Highly conductive paper fabricated with multiwalled carbon nanotubes and poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) by unidirectional drying

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Abstract Poly(3, 4-ethylenedioxythiophene)-poly(styrene-sulfonate) (PEDOT:PSS) was used to directly disperse multiwalled carbon nanotubes (MWNTs) into a stable solution without the aid of any other surfactants. The solution was coated onto paper substrates and was subsequently dried via two different methods: conventional multidirectional drying and a special unidirectional drying method with the aid of a ceramic heating board. The samples that were dried using the unidirectional drying method had a lower resistance than when dried with the conventional drying method, which allowed it to have a better conductivity. For solutions containing 1–4 mg/mL MWNTs, which were dispersed with 1 mg/mL PEDOT:PSS, the measured conductivity ranged from 21 ± 0.5 to 46 ± 10 S/cm. These values are one to three orders of magnitude higher than those obtained for MWNT films often reported in the literature as well as for films we fabricated using MWNTs dispersed with SDBS, or PEDOT:PSS solution by itself. AFM and SEM images showed that the surface of MWNTs films made with PEDOT:PSS as a surfactant was very smooth and the layers tended to be thinner than when made with the other surfactants. It was demonstrated using FTIR analysis that the MWNTs become part of the 3D structure of the paper substrate material because chemical bonding between the PEDOT:PSS, the MWNTs and the paper fibers occurred during the drying stage.

Introduction

Compared with plastics, glass, metal, and ceramic materials, paper substrates are lightweight, and can be easily folded. In addition, because of the uniqueness of the 3D hierarchical porous fiber structure of paper [1], it has strong adhesion for inks that are normally added onto paper and can therefore be used to fabricate lightweight, highly conductive paper materials [2, 3]. Such materials have been demonstrated to be useful for novel applications, such as in microfluidic devices and in electronics [4], for the formation of superhydrophobic surfaces [5–7], as well as in gas sensors [8]. Highly conductive paper materials are expected to play an important role in the sustainable development of the economy and the development of an environmentally friendly future, since they will be able to be used in e-paper, smartcards, e-tags, and many other special printing [9] and packaging products.

In the past few years, conductive papers have been manufactured by adding various fillers, including conductive polymers, titanium oxide, luminescent polymers, and ferric oxides [10–12]. Many different methods have been tried including layer-by-layer assembly [13–15], Meyer-rod coating [3], papermaking [16], and spin-coating method [16]. But conductive papers available have some limitations. Among these are that the conductivity is not high enough [13, 17], or they require deposition of thick layers which results in higher costs [16], or the tendency of coated papers to have weak mechanical properties [18]. As can be seen from Table 1, the reported conductivities for materials containing carbon nanotubes range from 10^{-2} to 10 S/cm. In order to be able to use conductive paper materials in a variety of electronic applications, they should have the following attributes: (1) to have the ability to store high amounts of energy, (2) to increase the durability, (3) to be

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Table 1 Electrical properties of some carbon nanotube composite materials

Materials	Method	Electrical property		Filler type	Ref.
		Conductivity (S/cm)	Resistance (Ω/sq)		
PEDOT:PSS/cellulose	Layer-by-layer	1–10		1 g cellulose–550 mg PEDOT: PSS	[2]
PEI/PEDOT:PSS/CNT-PSS/ wood fibers	Layer-by-layer	0.02–2		PEI (3 mg/mL)/PEDOT:PSS (3 mg/mL)/CNT-PSS (35 µg/mL)	[11]
Alumina/CNT/Cladophora cellulose	Spark plasma sintering	5.76		Alumina–5 wt% CNT	[13]
Ag/paper	Meyer-rod-coating	2	500 nm Ag coating layer		[3]
SWNT/paper	Meyer-rod-coating	10	5 mg/mL SWNT in solution		[3]
Gold/paper	Meyer-rod-coating	7	50 nm gold coating layer		[3]
CNT/cellulose	Papermaking	9.12	Cellulose–16.7 wt% CNT		[14]
FMCNT/PEDOT: PSS	Spin-coating	260	25 mg FMCNT–3 g PEDOT:PSS		[15]

Table 2 List of solution compositions prepared and their sample name designations

Solution (15 mL)	Dispersed by	Samples names
MWNTs: 1, 2, 3, 4 and 5 mg/mL	SDBS (10 mg/mL) + DI water	SDBS 115#, SDBS 215#, SDBS 315#, SDBS 415# and SDBS 515#
MWNTs: 1, 2, 3, 4 and 5 mg/mL	PEDOT:PSS (1 mg/mL) + DI water	PED 115#, PED 215#, PED 315#, PED 415# and PED 515#
PEDOT:PSS: 1, 2, 3, 4 and 5 mg/mL	DI water	PEDO 115#, PEDO 215#, PEDO 315#, PEDO 415# and PEDO 515#

imparted with superhydrophobic properties, (4) to be light and collapsible, while (5) maintaining strong mechanical properties, and (6) be environmentally friendly at the same time.

In this article, we report on the usage of PEDOT:PSS as a surfactant to disperse multiwalled carbon nanotubes (MWNTs) in the solution, coating the solution onto the paper surface with a special drying method to prepare a wide range of conductivity paper materials. The results are compared to other conductive papers made with PEDOT:PSS only or by dispersing MWNTs in SDBS. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) were used to analyze the structure of the samples and FTIR was used to determine the extent of chemical bonding. The electrical properties of the samples were measured using four-point probe resistivity measurements.

Materials and experimental details

Chemical and materials

The MWNTs with outer diameter: 8–15 nm, length: 0.5–2.0 µm, purity: >95 wt%, ash: <1.5 wt%, were obtained from cheaptubes.com. Poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate): 1.3 wt%, and sodium

dodecylbenzenesulfonate (SDBS), were purchased from Sigma-Aldrich. Filter paper qualitative circles 411, obtained from VWR.com, of size 125 mm in diameter, were used as the paper base material. All materials were used as-received without further modification.

The solution preparation

The MWNTs were added into distilled water, ultrasonicated for about 20 min, and then the surfactants (either PEDOT:PSS or SDBS) were added into the solution. The solution was ultrasonicated for an additional 30 min. After this process, the mixtures were magnetically stirred for about 12 h. The content of MWNTs in the solution varied from 1 to 5 mg/mL. When SDBS was used as a surfactant, 10 mg/mL was added to the solution. When PEDOT:PSS was used as a surfactant, only 1 mg/mL was added. All solutions were made with 15 mL distilled water. Table 2 lists the different MWNT solutions prepared and their sample designations.

Meyer-rod coating method with and without a ceramic heating board

Using a ceramic heated board, the temperature was maintained at about 90 °C. A piece of filter paper

(size:length × width = $7.7 \times 4.7 \text{ cm}^2$) was put on top of the heating board. The solution was coated onto the surface of the paper using the Meyer-rod coating method [19], then the paper was allowed to dry at 65 °C for 30 min. Because of the range of temperature used and the 3D structure of the fibers, the water was able to be easily absorbed into the direction of higher temperature, which induced the unidirectional drying of water vapor. The process was repeated 15 times to have homogeneous conducting films. For the last step, the films were dried at 70 °C for about 24 h. Processed this way, MWNTs were found to more easily penetrate into the 3D structure of the paper fibers as compared to when the material was prepared by standard oven drying, where water vapor flows in all directions, as shown in Fig. 1.

Analysis of the samples

The surface and structure characterization of the samples, and the film thicknesses were analyzed by SEM and AFM. SEM imaging was carried out using a Hitachi S800 SEM at an accelerating voltage of 5 or 6 kV. Surface elemental analysis was performed with energy dispersive spectroscopy (EDS). AFM images were acquired using an XE-100E, Park Systems scanning probe microscope in non-contact mode. ACTA cantilevers, which have tip diameter of <10 nm, were used for the imaging. The electrical properties of all films were measured using a Keithley 6221 AC/DC current source and a Keithley 2182A nanovoltmeter on a four-point probe configuration. The current was swept from 0 to 1 mA.

Results and discussion

Surface and structure characterization of the samples

When the papers were coated with the MWNT solutions, the appearance of the paper quickly turned dark, becoming darker with increasing content of MWNT added, irrespective of which surfactant was used, SDBS or PEDOT:PSS. In contrast, for the papers coated with only PEDOT:PSS

solution, the color was found to turn blue. This is characteristic of the appearance of conducting polymers such as PEDOT:PSS.

From the SEM images in Fig. 2, we can see that the MWNTs easily penetrated into the microstructure of blank paper when dispersed with SDBS (Fig. 2a), but the surface was rougher when compared to the uncoated paper sample (Fig. 2b). In contrast, the sample coated with PEDOT:PSS solution only (Fig. 2c) showed a smoother surface. However, it was found that when the contents of PEDOT:PSS was more than 5 mg/mL, the samples fractured easily. When the paper fibers were coated with MWNT solution dispersed with PEDOT:PSS (Fig. 2d), the surface was smoother than when the MWNTs were dispersed in SDBS, and the mechanical properties did not seem to be affected since we only used 1 mg/mL PEDOT:PSS in this case. PEDOT:PSS not only dispersed MWNTs well, but it also helped to connect the MWNTs with the paper fibers better and enhanced the mechanical properties of these films.

The thicknesses of the paper samples, which varied from 160 to 200 μm, were determined by geometric measurements and from SEM images of cross sections of the samples. The thicknesses of the coated layers were determined by comparing the thicknesses of uncoated paper with the thickness of coated paper samples, as depicted in Fig. 3a, b. The film thicknesses were determined by subtracting the average thickness of the uncoated paper from the total thickness obtained for the coated papers. Figure 4a summarizes the comparison of the thickness of paper samples that were dried with or without the special unidirectional drying method. It can be seen that drying with the ceramic heating board results in specimens with thinner thicknesses. Figure 4b depicts the average measured thickness of the coated layers that were deposited using solutions with different contents of MWNTs for the three different series of samples investigated, utilizing the unidirectional method.

Analysis from AFM images and surface roughness of the samples displayed in Fig. 5 suggest that the degree of MWNT penetration into the microstructure of the paper fibers is substantial. It can be seen in Fig. 5a that the microstructure of the MWNT/PEDOT:PSS film that was

Fig. 1 Drying procedure for the MWNTs coated paper:
a evaporation in a standard drying oven and **b** drying in modified oven with a ceramic heating board

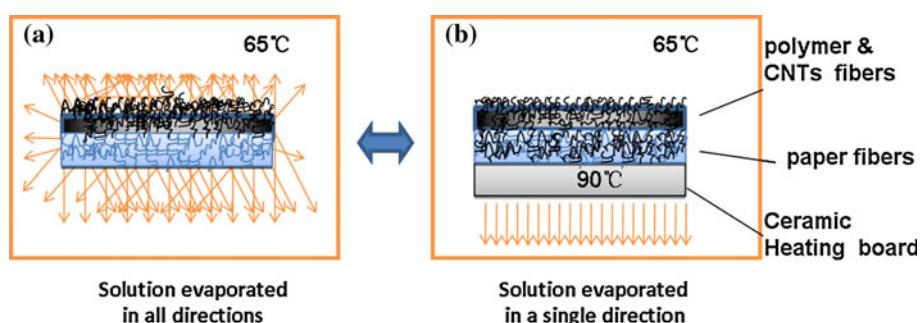


Fig. 2 SEM images of blank paper and coated paper samples with different solutions **a** SDBS 115# 2KX, **b** uncoated paper# 2KX, **c** PEDO 115# 2KX and **d** PED 115# 2KX

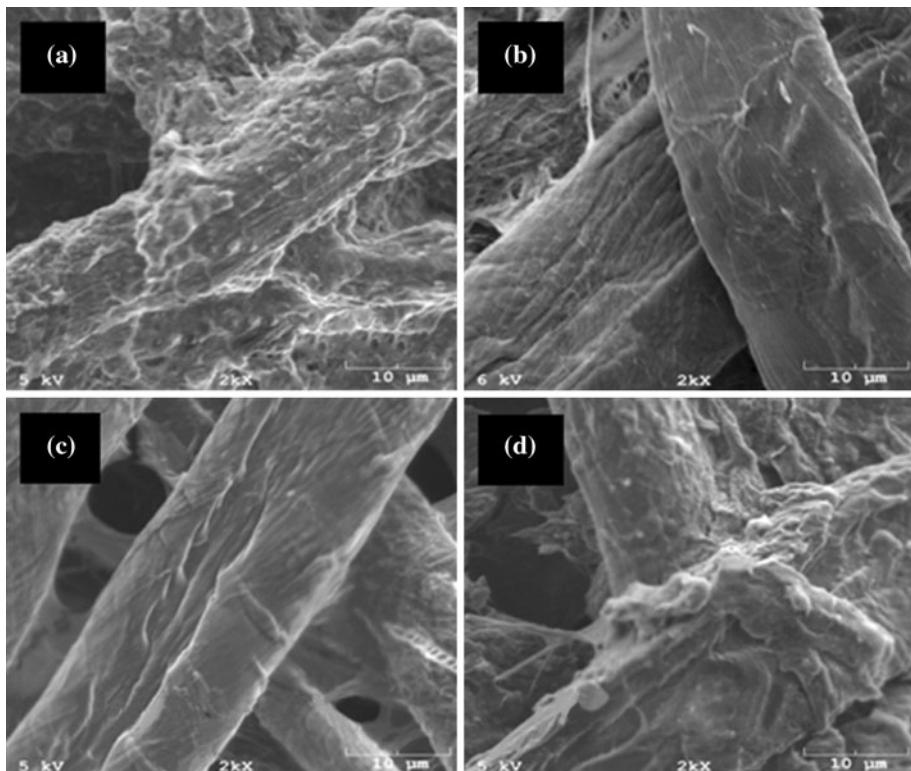


Fig. 3 SEM images of cross sections **a** uncoated sample cross section and **b** coated samples cross section. The thickness was determined by subtracting the uncoated thickness from the coated thickness

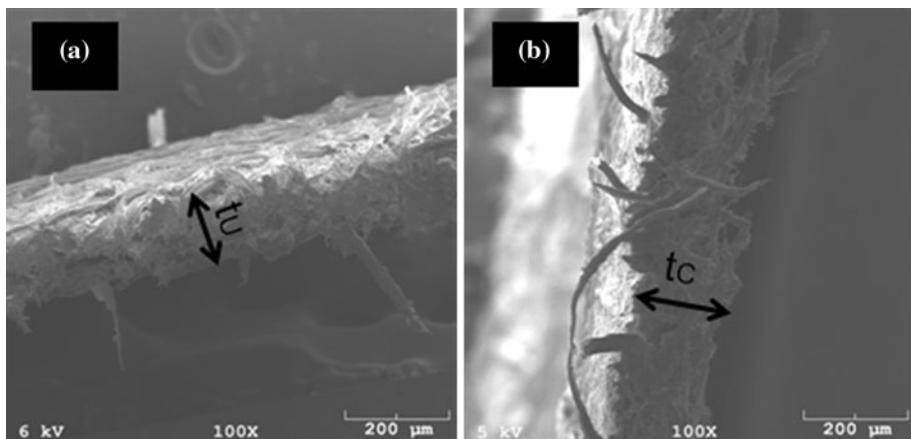


Fig. 4 Thickness of MWNT layers measured from SEM cross sections. **a** Coating layers with or without special drying method and **b** coating layers by different solutions with special drying method

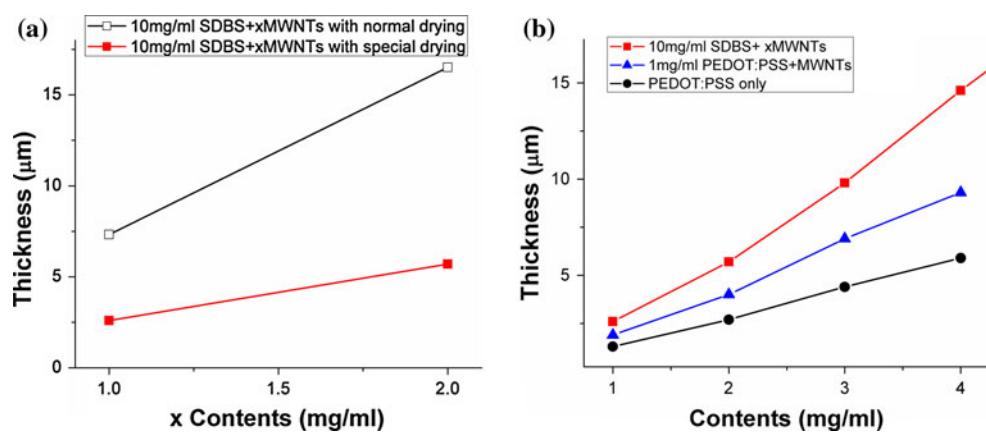


Fig. 5 AFM images of the samples deposited using the PED 115# solution (MWNTs dispersed in PEDOT:PSS). **a** Film on glass plate (surface roughness: 22.400 nm) and **b** film on paper (surface roughness: 87.589 nm)

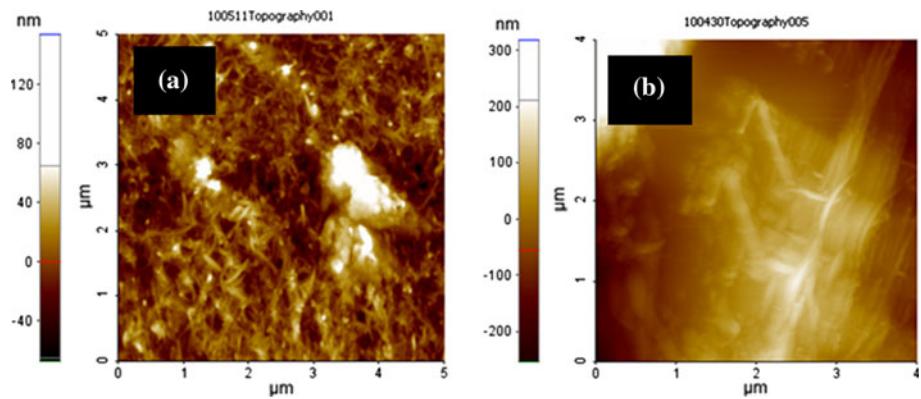
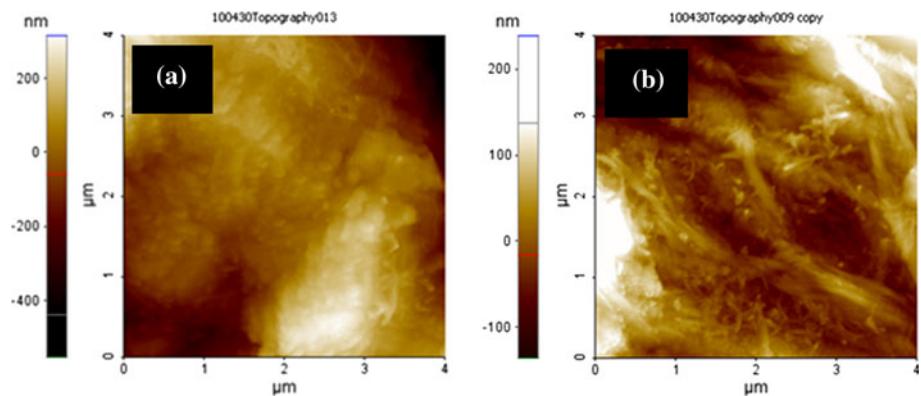


Fig. 6 AFM images of films made using SDBS 115# deposited on paper. Topography obtained **a** with heating board (surface roughness: 134.306 nm) and **b** without heating board (surface roughness: 62.219 nm)



coated onto a glass plate appears rougher but its surface roughness is only 22 nm, while the surface roughness for the film deposited onto the paper substrate has four times the surface roughness of the film on the glass slide but has an overall smoother appearance (see Fig. 5b). This can be understood by looking at the structure of the uncoated paper on a larger scale (see Fig. 2b) as compared to the structure of the composite MWNT/PEDOT:PSS film shown in Fig. 2d.

Furthermore, Fig. 6a demonstrates that when using the special drying method, the MWNT particles were more easily absorbed into the 3D structure of the paper fibers, whether PEDOT:PSS or SDBS were used (SDBS shown in this case). This resulted in films with higher overall surface roughness than for those films that were dried without a heating board (Fig. 6b). It is also clear that the surface of the sample coated with MWNT dispersed with PEDOT:PSS (Fig. 5b) was smoother than that with any of the samples made with SDBS surfactant (Fig. 6a, b).

FTIR analysis

The FTIR analysis was carried out to provide some evidence for chemical bonding between the MWNTs, PEDOT:PSS, and the paper fibers. The FTIR spectra of the three individual materials are shown in Fig. 7, which are compared to the spectrum for a composite MWNT/

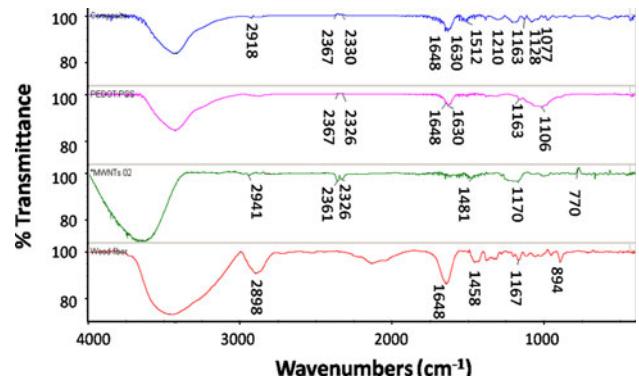


Fig. 7 FTIR spectra of a composite film made from MWNTs and PEDOT:PSS and deposited onto filter paper and the spectra for individual materials: PEDOT:PSS, MWNTs and filter paper fibers (from top to bottom)

PEDOT:PSS/paper fiber film. It can be seen from Fig. 7 that the composite film shows bands at 1077, 1128, 1163, 1210, 1512, 1648, 2330, 2367, and 2918 cm⁻¹. The C–H bending band at 770 cm⁻¹ [20] in the spectrum of MWNTs, the CH₂=CH strong band at 894 cm⁻¹ [21] in the spectrum of filter paper fiber, and the C–C vibration at 1016 cm⁻¹ [22] in the spectrum of PEDOT:PSS all disappeared when the MWNTs solution that was dispersed by PEDOT:PSS was coated on the surface of filter paper. It can also be seen that the asymmetric S=O at 1163 cm⁻¹,

C–O–C ester stretching band at 1167 cm^{-1} [23], and C–O stretching band at 1170 cm^{-1} [24] from PEDOT:PSS, paper fiber, and MWNTs, respectively, are also not present in the composite sample. However, the C–S stretching band in the composite sample is present at 1077 cm^{-1} [25], the C–C stretching vibration at 1128 cm^{-1} , and the C–O stretching vibration at 1210 cm^{-1} [26] are observed in the spectrum of the composite. The C=C aromatic ring at 1481 cm^{-1} [27] in the MWNTs and at 1458 cm^{-1} [28] in the paper fiber are not present, but the C=C lower stretching band exists at 1512 cm^{-1} [29] in the spectrum of the composite. The C=C stretching vibration at 1630 cm^{-1} [30] and the C=O band at 1648 cm^{-1} [31] present in the composite sample are characteristic peaks of PEDOT:PSS. The O–C–O absorption band at 2326 cm^{-1} from PEDOT:PSS disappeared, and the O–C–O band observed in the MWNTs at 2361 cm^{-1} is shifted to 2330 cm^{-1} in the composite [32]. The absorption band at 2367 cm^{-1} can be attributed to the C≡N nitrile bond [33]. The C–H on the neighbor group of C-6 at 2898 cm^{-1} is present in the spectrum of paper fiber. In contrast, the C–H stretching vibration in the composite film appears at around 2918 cm^{-1} [34]. The FTIR analysis shows that when the MWNTs solution was dispersed by PEDOT:PSS, and then coated onto the filter paper fibers, the C–H band at (770 cm^{-1}) and the aromatic ring (1481 cm^{-1}) of MWNTs are not present, and that the asymmetric S=O (1163 cm^{-1})

and –O–C–O– absorption band (at 2326 cm^{-1}) of PEDOT:PSS were broken, and the C=C and CH on the neighbor group of C-6 in the structure of paper fibers disappeared, while C–S stretch (at 1077 cm^{-1}), and C–O stretching vibration (at 1210 cm^{-1}), and C=C lower stretching band (at 1512 cm^{-1}) exist in the structure of the composite sample. This demonstrates that PEDOT:PSS was not only excellent as a surfactant, but that it formed chemical bonds with the MWNTs and paper fibers. Table 3 summarizes the FTIR bands present.

Electrical characterization

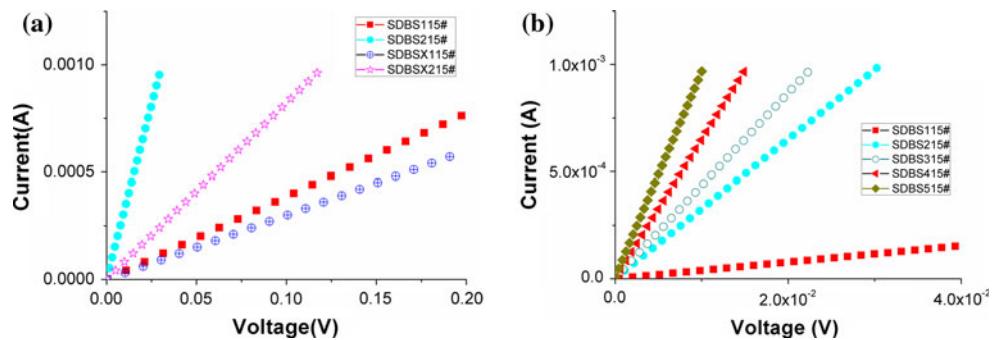
The four-point probe method was used to test the resistance of the samples. Because of the special structure of the paper materials and the drying methods used, the thickness of the coating layers ranged from 1.3 to $19.1\text{ }\mu\text{m}$. To ensure repeatability, measurements were made at 12 different locations for each composite sample. These values were then averaged and the standard deviations were determined to calculate the resistances for each sample measured for each sample set.

At first the resistance (R) of the samples that were fabricated with the special drying method was compared to those dried in a conventional oven. The I – V curves displayed in Fig. 8a, where the slopes represent $1/R$, show that the resistances are lower for the films dried with a ceramic

Table 3 FTIR vibration peaks present in the materials when dried

Composite peaks (cm^{-1})	PEDOT:PSS peaks (cm^{-1})	MWNTs peaks (cm^{-1})	Paper fiber peaks (cm^{-1})	Band group
		770		=C–H bending
			894	$\text{CH}_2=\text{CH}$ strong band
1016				C–C vibration
1077				C–S stretch
1128				C–C
1163	1163			Asymmetric S=O
		1167		C–O–C ester stretching band
		1170		C–O stretching bending
1210				C–O stretching vibration
		1458		C=C aromatic ring
		1481		C=C aromatic ring
1512				C=C lower stretching band
1630	1630			C=C stretching vibration
1648	1648			C=O
		2326		–O–C–O– absorption band
2330		2326		–O–C–O–
		2361		–O–C–O–
2367	2367			C≡N
			2898	CH (neighbor group of C-6)
2918				CH stretching vibration
		2941		CH

Fig. 8 Comparison of $I-V$ curves for the samples that were coated with the solutions. **a** Dried with special drying method (filled symbols) versus conventional drying method (open symbols) and **b** effect of MWNT content, 1–5 mg/mL with special drying method



heating board (shown in solid symbols) than those dried using conventional multidirectional drying. Their smaller thickness (as Fig. 4a showed), indicates that the special unidirectional drying method presented here is better for preparing more conductive paper materials than normal drying methods.

The effect of the MWNT content dispersed with SDBS is shown in Fig. 8b, where the resistance is seen to decrease, as a function of increasing MWNT content. When the paper materials were coated with different contents of PEDOT:PSS solution only, the resistance also decreased with the increase of the contents of PEDOT:PSS (not shown), but the resistance of those films was much higher than those coated with MWNTs solution made with SDBS presented in Fig. 8b. Furthermore, when the content of MWNT was 5 mg/mL, the paper materials became brittle and could be easily broken. When the paper materials were coated with MWNTs solutions that were dispersed with 1 mg/mL PEDOT:PSS, the resistance was lower than for samples coated with MWNT solutions that were made with SDBS. In addition, these paper materials showed some polymer properties as the surface was very smooth (see Figs. 5b, 6b) and the paper fibers and the MWNTs had good connections with one another due to the presence of the PEDOT:PSS conducting polymer.

Figure 9 displays the measured resistance for the three series of samples investigated. The resistances of the samples coated with MWNTs solution, which were dispersed with SDBS, were much lower than the resistance of the samples that were coated with PEDOT:PSS solution only. But when the MWNTs were dispersed with PEDOT:PSS, the resistances of the samples were even lower. This means that although PEDOT:PSS solution has a lower conductivity as compared to MWNTs alone, PEDOT:PSS provides a good connection between MWNTs because of its own conductivity. In addition, the presence of the PEDOT:PSS in the solution helps to fabricate a very smooth surface, especially when it is used with the special drying method after coating. Thus, such materials also display the conformal property of polymers.

Because the thickness of the paper was about 200 μm , while the thickness of the coating layer was no more than

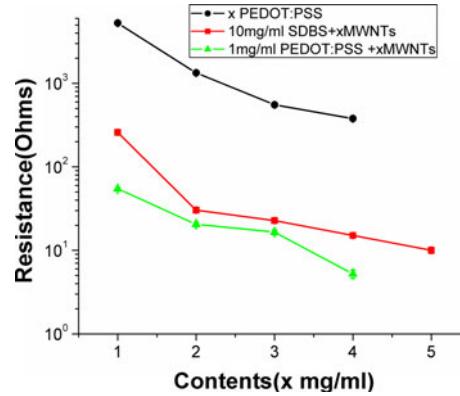


Fig. 9 Comparison of the resistance of the three series of conductive paper samples, where x varied from 1 to 5 mg/mL

20 μm , the conductive paper material can be considered to be thin sheet material (thickness $t \ll s$ where s is the spacing between the probes, 1.68 mm in this case). Therefore, the resistivity of the samples was calculated using Eq. 1[35]:

$$\rho = \frac{\pi t}{\ln 2} \left(\frac{V}{I} \right) = 4.532 \frac{V}{I} t, \quad (1)$$

where ρ is the resistivity of the samples, t is thickness, V is the voltage, and I is the current.

Figure 10 depicts the conductivity ($\sigma = 1/\rho$) of the three series of samples investigated in this study. When the content of MWNTs (or PEDOT:PSS) increases, the conductivity of the samples also increases. The conductivity of the paper material coated with 4 mg/mL PEDOT:PSS solution only was 1.0 S/cm. The sample coated with 4 mg/mL MWNTs solution dispersed by 10 mg/mL SDBS was 11 ± 1 S/cm, while the sample coated with 4 mg/mL MWNTs solution dispersed by 1 mg/mL PEDOT:PSS was 46 ± 10 S/cm. As demonstrated in Figs. 2d, 5b, when the MWNTs that were dispersed with PEDOT:PSS were coated on the surface of paper, the MWNTs were seen to more easily penetrate into the structure of the paper with the evaporating water during the unidirectional drying step. It is important to mention that PEDOT:PSS did not act just as a surfactant, but since it is a type of conductive polymer [2], it helped to create good connections between MWNTs

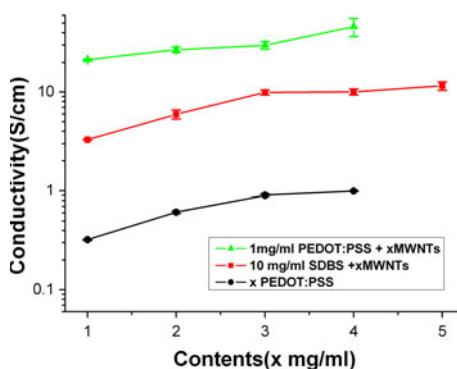


Fig. 10 Comparison of the conductivity of paper samples coated with SDBS + MWNT solution, PEDOT:PSS + MWNT solution, and just PEDOT:PSS solution. x indicates the filler content which varied from 1 to 5 mg/mL

with paper fibers, as demonstrated by the shared bands presented in the FTIR analysis, thus highly increasing the conductivity of samples.

Conclusions

Highly conductive paper materials were fabricated by combining MWNTs and PEDOT:PSS conductive polymer and drying the films with a special unidirectional drying method. The resistance of the samples and the thickness of coating layers were smaller than those samples that had been prepared without the special drying method. When the content of the MWNTs in the solution increased, the resistance decreased. When the samples were coated with 4 mg/mL MWNTs solution that was dispersed with 1 mg/mL PEDOT:PSS, the conductivity reached 46 ± 10 S/cm, which was one and two orders of magnitude larger the conductivity for films made with MWNTs dispersed with SDBS or PEDOT:PSS solution only. When PEDOT:PSS was used as a surfactant for MWNTs, it promoted good connection of MWNTs with the paper fibers, which caused the film surface to become very smooth and highly increased the conductivity of the samples.

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