


Effects of Different Types of Surfactant Treatments on the Electromechanical Properties of Multiwalled Carbon Nanotubes Decorated Electrospun Nanofibers

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ABSTRACT

Carbon nanotubes (CNTs) have a strong tendency to form agglomeration due to van der Waals interactions, which hinders their practical utilization. Therefore, an effective and stable dispersion of CNTs in a surfactant based solvent is very important for the realization of CNTs based nanocomposites in various applications. In this paper, influence of different types of surfactant on the electromechanical properties of multiwalled carbon nanotubes (MWCNTs) decorated electrospun thermoplastic polyurethane (TPU) nanofibers were investigated by UV-VIS spectroscopy, zeta potential, FT-IR analysis, scanning electron microscopy (SEM) and uniaxial tensile strain sensing. Obtained results suggest that type of surfactant has not only effecting the dispersion level of CNTs but also has a significant influence on the electromechanical properties of CNTs decorated electrospun CNTs/TPU nanofibers. The results of the present study provide new insights into the design and tailoring the electromechanical properties of CNTs decorated electrospun nanofibers.

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1. INTRODUCTION

Among various carbonaceous nanofillers, carbon nanotubes (CNTs) has inspired many scientist owing to their unique physical properties which make them ideal as reinforcing material for high performance nanocomposites. High performance nanocomposites can be synthesized by combining them with CNTs that enables their usage in numerous industrial applications including flexible electronics [1], mechanical sensors [2-7] electromagnetic interference shielding [8] etc. Fabrication of high performance CNTs based nanocomposites still challenging since CNTs have a great tendency of forming bundles due to strong van der Waals interactions [9, 10]. These bundles and agglomerations cause a deterioration in the mechanical and electrical properties of CNTs based composites [11, 12]. Realization of aforementioned applications of CNTs based nanocomposites can be achieved by ensuring a reliable and

effective dispersion of CNTs. To break down the CNTs bundles or agglomerates and disperse them in surfactant containing solution, several approaches including mechanical methods and physical (non-covalent) or chemical (covalent) processes have been adopted to their surface energies [13, 14]. It has been reported that surface modification of CNTs by introducing functional groups, which interacts with surfactant through polar- polar interaction resulting prevention of CNTs to form bundles that helps to better and more stable dispersion [15]. Chemical methods are surface functionalization methods used to improve properties such as wettability, agglomeration, chemical compatibility. With the functionalization process, the dispersibility, reactivity, processability and biocompatibility of CNTs can be increased [16]. Non-covalent surface treatments are a preferred method because of the ability to adsorb various functional groups to the surface without disturbing the structures of π -bonds in

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graphene sheets [17]. However, surfactants and polymers that can be used in this functionalization process are limited and their dispersions are not stable. Functionalized CNTs are difficult to re-modify. Covalent functionalization can greatly improve the solubility, dispersibility and chemical compatibility of CNTs. Covalent functionalization occurs when the π -bonds in the basic structures of CNTs are broken, and the functional group is bonded by forming a covalent bond that shares at least one electron with the functional group. It is this deterioration and restructuring in the structure that changes and improves the properties of the CNTs [18-20]. Mechanical methods such as ultrasonication and high shear mixing on the other hand can indeed separate the CNTs from each other. However, with increasing time and power aspect ratio of CNTs can shorten the by breaking the tubes.

For the modification of CNTs in the preparation of CNTs based polymer nanocomposites various surfactants are employed [21-24]. Depending on the polarity of the surfactant head group, surfactants can be categorized into four types, which are non-ionic, anionic, cationic, and amphoteric [25]. Among various surfactants, sodium dodecyl sulfate (SDS) and cetyltrimethylammonium bromide (CTAB) have drawn attention. SDS is an organic compound and it is the most widely studied anionic (negatively charged) surface agent. It consists of a head and tail with amphiphilic properties, consisting of a 12-carbon chain attached to a sulfate group [26]. At low concentrations of SDS, which allows the CNTs to be suspended, CNTs agglomeration can be observed even after ultrasonication. By increasing and optimizing the SDS concentration, a homogeneous carbon nanotube solution appearance can be achieved [27, 28]. CTAB on the other hand is an important cationic (positively charged) surfactant consisting of a head with three methyl and an ammonium group and a tail with 16 carbons [29]. It is used as coating, stabilization and passivation agent. Through CTAB modification sensitivity and detection limits of materials can be improved by increasing their detection performance. To date, there are various studies dealing with the role of the surfactants on the dispersion of CNTs and their mechanisms [30-35]. Zou et al. [35] conducted the absorbance of CNTs: polycarboxylate-based cement superplasticizer at different concentrations at different sonication energies. It was seen that absorbance rate increases gradually with the increase of the sonication power that is proportional to the CNTs concentration. Recently, Rajendran et al. [33] has studied a comparative analysis of the dispersion of CNTs in polar solvents. It was suggested that the degree of affinity CNTs with -COOH group depends on the polarity of the solvent type. Chatterjee et al. [36] investigated the effect of the different surfactants i.e. DOC, SDBS, CTAB, BzlkCl, and TX405 on the selectivity and sensitivity of CNTs based volatile organic compounds (VOC) biomarkers of lung cancer. For the assessment of the influence of surfactant type on the properties of MWCNTs decorated electrospun nanofibers, there are still various aspects to be investigated. In this work, the effect of dispersion type on the properties of -COOH functionalized MWCNTs-TPU based nanofibrous structures fabricated by

electrospinning technology was investigated. Samples were fabricated by dip coating electrospun TPU nanofibers into MWCNTs based dispersed solution. Dispersion quality of MWCNTs were assessed by UV-Vis and Zeta potential analysis. Results are finally further discussed by assessing FTIR, SEM, 2P-4P probe electrical resistance and uniaxial tensile strain measurements.

2. MATERIAL AND METHOD

2.1 Materials and Chemicals

From Molchem Technologies (London, United Kingdom) purchased industrial -COOH functionalized MWCNTs has 92% purity and 8– 15 nm outer diameter. Thermoplastic polyurethane (TPU) (Elastollan 1185A10) was obtained from Biesterfeld in granule form with 1.12 kg/dm³ density, N, N-Dimethylformamide (DMF), Sodium Dodecyl Sulfate (SDS) and Cetyltrimethylammonium bromide (CTAB) were bought from Labor Teknik (Istanbul, Turkey), Akbel Kimya (Bursa, Turkey) and Alfa Aesar (Thermo Fisher GmbH, Germany), respectively. All chemicals used in this study were used and received without any further treatment.

2.2 Characterization of electrospun MWCNTs/TPU Nanofibers

To understand the morphology as well as complex piezoresistive behavior of MWCNTs/TPU nanofibers, various characterization methods including SEM, U-Vis, FTIR, Zeta potential, 2-point and 4-point probe resistance measurements and uniaxial loading measurements were conducted. For the electrospinning process, samples were electrospun by an electrospinning unit (Inovenso NS24XP). SEM analysis (Zeiss Supra 55 VP) was used to see the microstructure and dispersion quality of the fibers decorated with MWCNTs. To do this, all samples were coated with gold and all analysis was performed at 10 kV and 13 mm WD (work distance). In order to determine the stability of the mixed solutions containing MWCNTs/surfactant agents, samples were analyzed by UV-Vis Spectroscopy (NanoPlus). For the precipitation of the MWCNTs contained solutions for UV-Vis analysis, the solutions were first centrifuged by a centrifugation device (Hermle Z206A) at 6000 rpm for 30 minutes and then passed through a 0.2 μ m Polytetrafluoroethylene (PTFE) filter syringe. FTIR analysis was performed with Bruker brand Tensor 27 model device in order to see interface interaction between TPU fibers and dispersed MWCNTs structures. To perform electrical measurements (2-point probe resistance measurement), copper tapes were applied on the both side of films with a distance of 3 cm. A digital multimeter (Fluke 179) with a resolution of 0.1 Ω was then used to measure the electrical resistance of the electrospun TPU films coated with MWCNTs. Moreover, a high precision instrument (Keithley 2400) was used to evaluate surface resistivity measurements (4-point probe resistance measurement). The average of the measurements taken from 10 different locations in the area between the copper wires was calculated. Subsequently, to see the relative

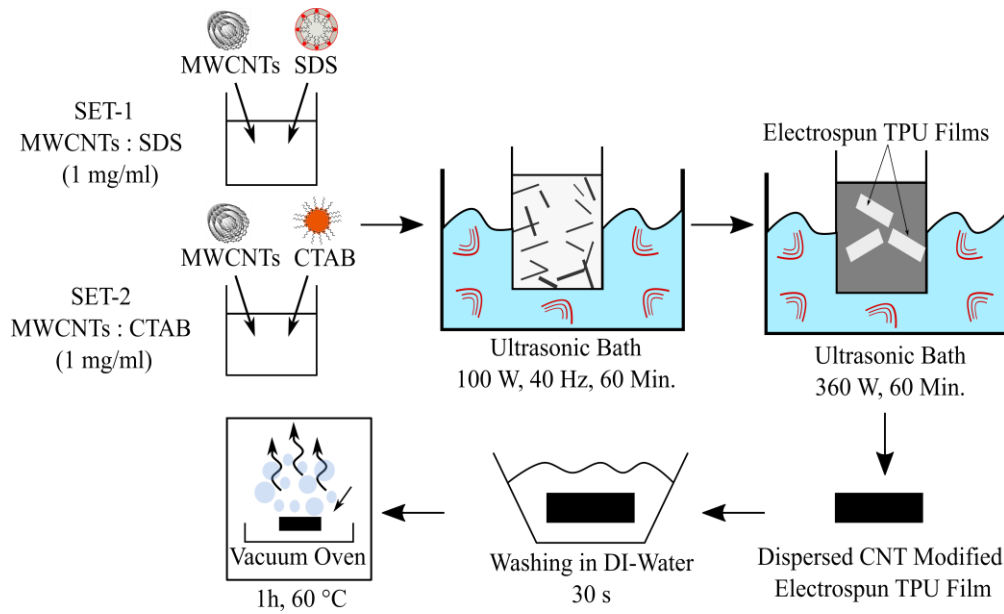


Figure 2. Schematic representation of dispersion of MWCNTs at different surfactants and decoration of electrospun TPU nanofibers by dipping them in dispersed MWCNTs-surfactant solutions

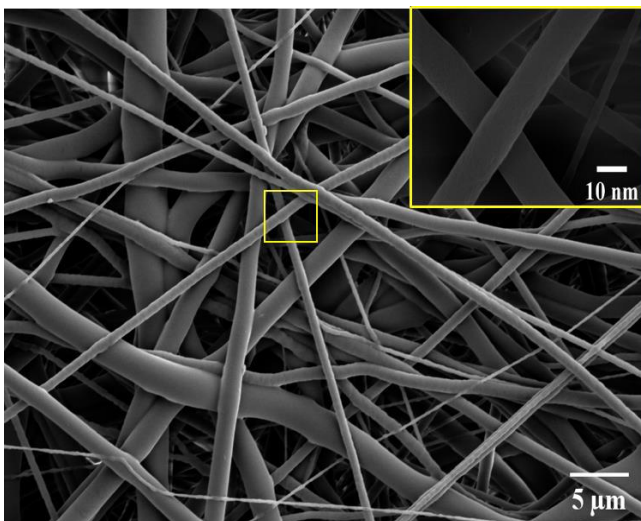


Figure 3. Morphology and structure of electrospun TPU fibers. Inset shows closer image of morphology of TPU fibers

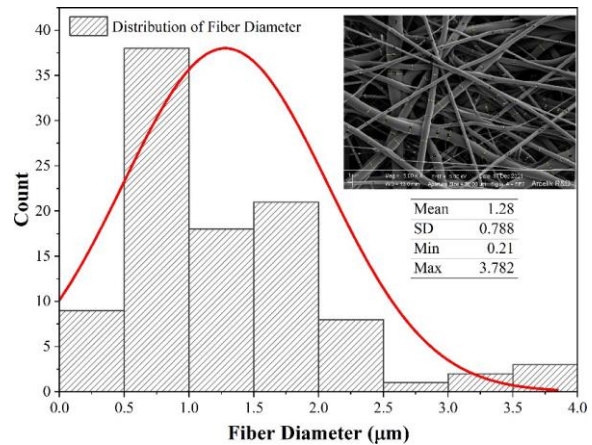


Figure 4. Distribution of fiber diameter of electrospun TPU fibers calculated by ImageJ software

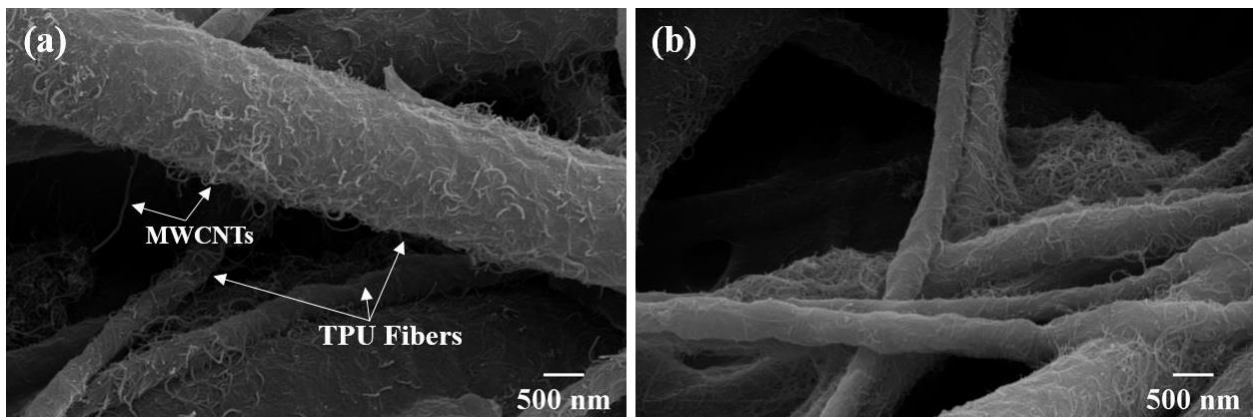


Figure 5. SEM image of electrospun (a) MWCNTs-SDS/TPU and (b) MWCNTs-CTAB/TPU sample. Here, MWCNTs concentration is set for both sample as 1 wt.%/v

UV-Vis analysis was performed to determine the dispersion efficiency of MWCNTs in MWCNTs-surfactant solutions. Owing to 1D van Hove singularities, individual CNTs exhibit characteristic bands and they are active in the UV-vis region. Whereas, agglomerated carbon nanotubes do not exhibit active behavior at wavelengths between 200-1200 nm [38-40]. Therefore, it is possible to relate the amount of individual CNTs dispersed in solution to the absorption intensity [41]. Figure 6 shows the UV-Vis spectra of bundled MWCNTs, MWCNTs-SDS and MWCNTs-CTAB solutions. It is clear to see that non-sonicated MWCNTs show almost no absorption in the UV-spectrum (see inset Figure 6a) due to existence of big agglomerates due to strong van der Waals attractions between the CNTs [42]. Introduction of SDS and CTAB surfactants with provided mechanical energy with sonication overcame the strong van der Waals interactions that lead to disentanglement of MWCNTs. Through UV-Vis spectra, it is seen that absorbance for both MWCNTs-SDS and MWCNTs-CTAB solutions give maximum peak between 200 nm and 250 nm and this absorbance gradually decreases [43]. It is important to note that, compared to the CTAB, dispersions with SDS surfactant give higher absorbance indicating better dispersion which lead to higher electrical conductivity.

Moreover, in order to determine the stability of the dispersions, zeta potential measurements for MWCNTs-SDS and MWCNTs-CTAB were conducted shown in Figure 7b. Basically, as shown in inset Figure 7b, zeta potential is an electrical potential at slipping plane or at boundary of the double layer which is a technique for the evaluation of the surface charge of nanoparticles including CNTs in a colloidal solution. Here, nanotubes have a charge on the surface that attracts a thin layer of ions to the nanotube surface which is called stern layer [44]. To evaluate this, the surface of MWCNTs was modified by cationic (CTAB) and anionic (SDS) surfactant at a fixed MWCNTs concentration (1 wt.%/v) using sonication technique. From the conducted studies, it is indicated that the magnitude of zeta potential is predictive for the colloidal stability of the dispersion and a zeta potential value greater than ± 60 mV indicates that the dispersions have excellent stability [45]. Obtained results show that MWCNTs-CTAB dispersions have peaks at around 71 mV and 72 mV, whereas MWCNTs-SDS dispersions have -101 mV and -96 mV zeta potential values, indicating that the MWCNT have better and more stable dispersion with SDS rather than CTAB due to higher level of zeta potential at same concentration and dispersion parameters. Positive values of MWCNTs-CTAB and negative values for MWCNTs-SDS based dispersions are attributed to the absorption of cationic and anionic charge on the MWCNTs surface [46]. Depending on the nature of the surfactants, cation and anion are forming. Here, surfactants are absorbed in the MWCNTs surface where either positive or

negative change result in electrostatic repulsion between the molecules that leads to stabilization of the nanotubes colloids [47].

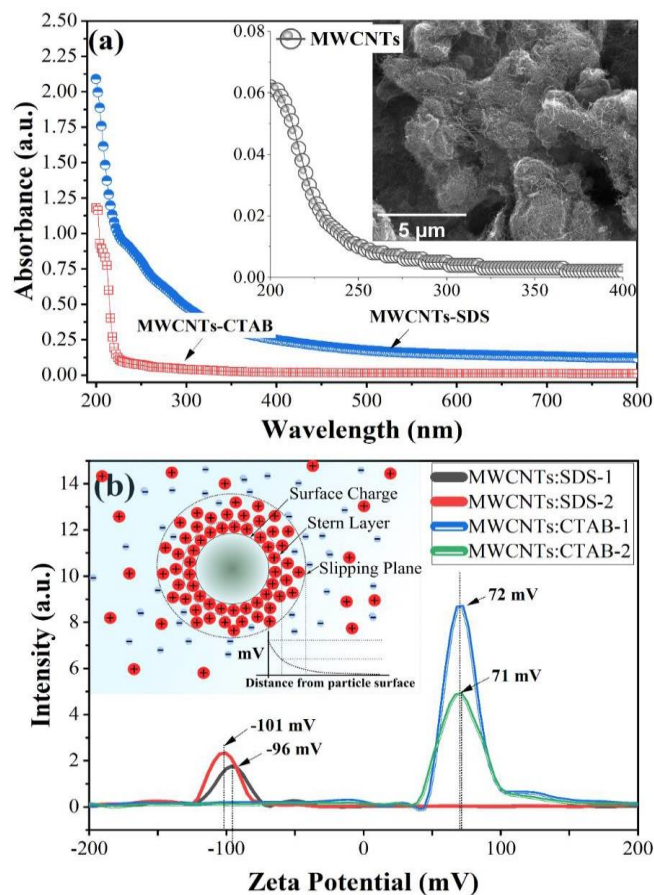


Figure 6. (a) UV-Vis spectra and (b) Zeta potential of MWCNTs-SDS and MWCNTs-CTAB dispersions. Inset figure in (a) shows UV-Vis and SEM image of non-dispersed MWCNTs and (b) shows the basic working principle of zeta potential

Furthermore, MWCNTs-surfactant/TPU nanofibrous structures were examined by FT-IR device to analyze the interface interaction between TPU fibers and dispersed MWCNTs structures. The FT-IR spectrum of the electrospun TPU film, MWCNTs-SDS/TPU and MWCNTs-CTAB/TPU are given in Figure 7a. Here, it is seen that for the electrospun neat TPU fibers, the peak at 3302 cm^{-1} is the characteristic N-H stretching band of the urethane structure, and the peak at 1728 cm^{-1} and at 1699 cm^{-1} are indicative of the free carbonyl group in the urethane bonds ($-\text{H}-\text{N}-\text{COO}$) and free H bonded C-O bonds respectively. The peaks at 2920 cm^{-1} and 2820 cm^{-1} are characteristics of medium alkenes-CH stretching vibrations. Also, the peak at the wavelength of 1525 cm^{-1} correlates with C-H. In addition, the C-O and C-O-C bands are available at wavelengths of 1219 cm^{-1} and 1074 cm^{-1} , respectively. Peaks at similar wavelengths were also observed in the FT-IR spectrum in the literature [48, 49]. Moreover, the FT-IR spectra of the MWCNTs decorated films match the spectra of the TPU film. It is seen that bathochromic shift (red shift) occurs as the TPU peaks shift

to the right in the coated films. Related studies indicating that the red shift can be attributed to the effects of increasing electron conjugation of conductive and semiconductor materials and the effect of increasing electron conjugation of CNTs [50-52]. Namely, abovementioned shifts indicate strong interactions between TPU chains and MWCNTs that is critical for the enhancement of electromechanical properties of electrospun MWCNTs decorated TPU fibrous structures. Subsequently, to study the effect of different type of surfactants on the electrical properties of electrospun MWCNTs-surfactant/TPU nanofibrous structures were measured by 2-point and 4-point probe resistance measurements given in Figure 7b. From the 2-points probe resistance measurements, it is calculated that electrospun samples based on MWCNTs-SDS/TPU and MWCNTs-CTAB/TPU have a resistance value around 25 k Ω and 31.3 k Ω , respectively. From the 4-point surface resistance measurements these resistance values decreased to 19 k Ω \pm 3.52% and 27 k Ω \pm 4.79% for MWCNTs-SDS/TPU and MWCNTs-CTAB/TPU samples, respectively. These findings suggest that MWCNTs are better dispersed in SDS surfactant that leads lower resistance values.

Finally, to see the effect of surfactant on the strain sensing characteristics of electrospun nanofibers, samples were undergone to uniaxial loading with the tensile speed of 1 mm/h and relative resistance changes at each strain value were recorded. It is seen that both samples show strain sensitivity with two linear regions. As previously reported [6], the piezoresistive working mechanism of MWCNTs decorated electrospun nanofibers can be explained as follows (see inset Figure 7c). At low strain range (region I) up to 36%, randomly oriented TPU fibers starts to move away from each other that lead to increase in resistance value. With the further increase of uniaxial strain (Region II), the distance between dispersed MWCNTs on the TPU fibers increase that results in a rapid increase in resistance [53]. Up to 50% of tensile strain, the relative resistance change for the MWCNTs-CTAB sample is nearly 264%, this value gets to 292% for the MWCNTs-SDS/TPU sample, which is attributed again a better dispersion of MWCNTs that contributes the piezoresistivity of nanofibrous structure with enhanced sensitivity. Namely, formation of more homogeneously dispersed MWCNTs networks enhance the overall relative resistance change under uniaxial applied strain.

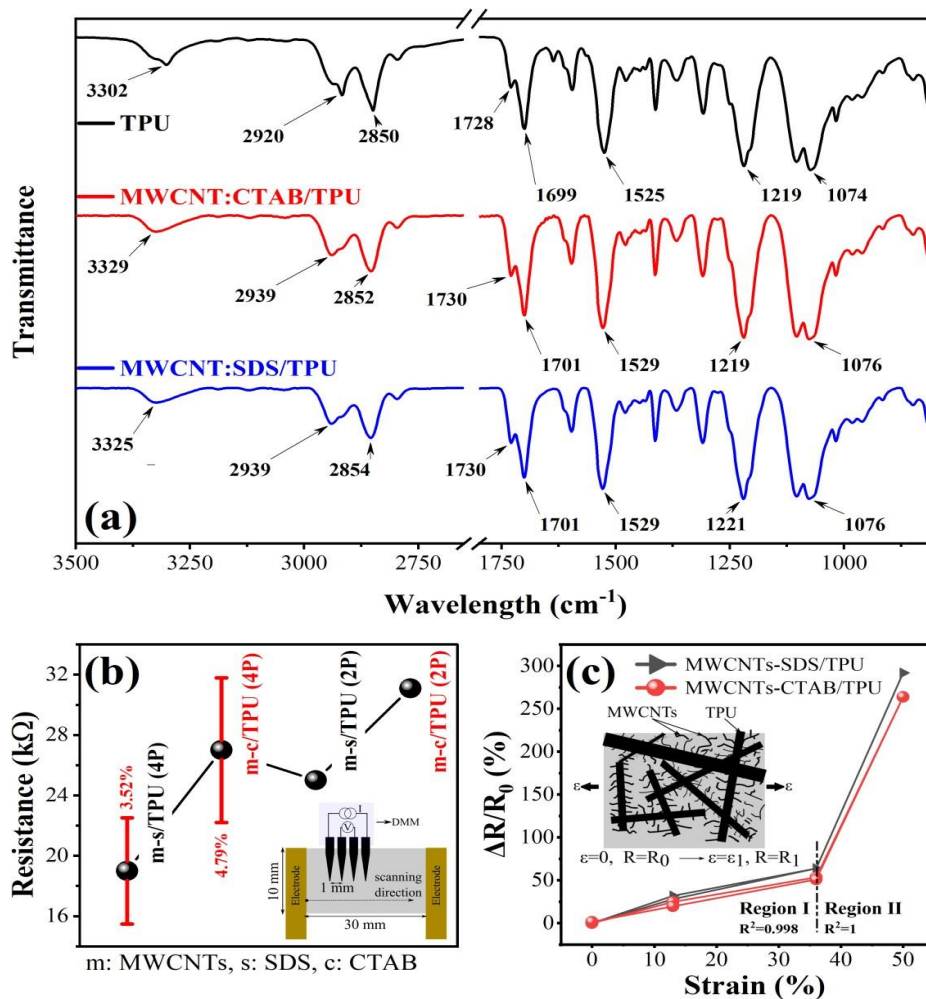


Figure 7. (a) FT-IR Spectra, (b) 2-point and 4-point probe resistance measurements and (c) uniaxial tensile strain measurement of electrospun MWCNTs-SDS/TPU and MWCNTs-CTAB/TPU samples. Inset figure in (b) and (c) shows the measurement setup for 4-point probe resistance measurement and piezoresistive working mechanism of electrospun sample under uniaxial tensile strain

4 CONCLUSION

This study investigates the effect of surfactant type (SDS and CTAB) on the electromechanical properties of MWCNTs decorated flexible electrospun TPU nanofibers. From the morphological investigations of electrospun nanofibers, it is seen that TPU fibers are homogeneously distributed and for both surfactant type and slightly better dispersion for MWCNTs-SDS solution based nanofibers were formed. Dispersion characterizations based on UV-Vis and zeta potential analysis also suggest that MWCNTs-SDS solutions have higher absorbance. Zeta potential results showing that MWCNTs dispersion with SDS have higher level of zeta potential than CTAB solutions which leads again better and more stable dispersion. FTIR analysis of the MWCNTs decorated TPU nanofibers shows a shift at peaks that is attributed to increasing electron conjugation of carbon nanotubes. From both 2P and 4P electrical

resistance measurement MWCNTs-SDS/TPU samples has lower resistance owing to better dispersion of MWCNTs. Subsequently, from the uniaxial tensile strain measurement, MWCNTs-SDS dispersions decorated electrospun TPU nanofibers give higher sensitivity which is as a consequence of better dispersion of MWCNTs that are responsible for piezoresistivity of nanofibers. Obtained results of this study provide new insights into the tailoring the electromechanical properties of MWCNTs decorated electrospun nanofibers by adjusting the type of the surfactant.

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