Effects of MWCNT and Sodium Dodecyl Sulfate (SDS) contents on the electrical conductivity and sensor properties of thermoplastic polyurethane nanosurfaces

RUSEN INAN
ISMAIL USTA

YESIM MUGE SAHIN

ABSTRACT – REZUMAT

Effects of MWCNT and Sodium Dodecyl Sulfate (SDS) contents on the electrical conductivity and sensor properties of thermoplastic polyurethane nanosurfaces

Recently, strain sensors have significant areas of usage with their strain, stretching and wearable features for various applications such as personal health monitoring, joint movement detection, robotics, etc. To achieve this, various studies are conducted to optimize the production of MWCNT-based nanocomposites. In the present study, a total of 12 solutions were formulated by introducing Multi-Walled Carbon Nanotubes (MWCNT) and Sodium Dodecyl Sulfate (SDS) at different concentrations and ratios into the Thermoplastic Polyurethane (TPU) solution, and then nanocomposite surfaces were produced from these solutions through an electrospinning process. These samples were subjected to resistance changes due to elongation, gauge factor changes due to elongation to evaluate the sensor property and cycle tests to evaluate the sustainability of the sensor feature. According to the results, the best sensor properties were obtained in the samples with SDS added at a rate of 1/20 (MWCNT:SDS) for 0.3 MWCNT and 0.5 MWCNT; on the other hand 1/26 weight ratio of MWCNT:SDS for 0.7 MWCNT.

Keywords: smart textiles, wearable sensors, MWCNT, SDS, electrospinning

Influența conținutului de MWCNT și de dodecil sulfat de sodiu (SDS) asupra conductivității electrice și proprietăților senzoriale ale nanosuprafețelor din poliuretan termoplastic

Recent, senzorii de deformare au început să aibă domenii semnificative de utilizare pe baza caracteristicilor lor de deformare, întindere și purtare pentru diverse aplicații, cum ar fi monitorizarea sănătății personale, detectarea mișcării articulațiilor, robotică etc. În scopul de a realiza aceste funcții, sunt efectuate diverse studii pentru a optimiza producția de nanocompozite pe bază de MWCNT. În studiul de față, a fost sintetizat un total de 12 soluții prin introducerea de nanotuburi de carbon cu pereți multipli (MWCNT) și dodecil sulfat de sodiu (SDS) la diferite concentrații și rapoarte în soluția de poliuretan termoplastic (TPU), iar apoi suprafețele nanocompozite au fost produse din aceste soluții printr-un proces de electrofilare. Aceste probe au fost supuse modificărilor de rezistență din cauza alungirii, ale factorului de etalonare datorat alungirii pentru a evalua proprietățile senzoriale și testelor ciclice în vederea evaluării sustenabilității proprietăților senzoriale. Conform rezultatelor, cele mai bune proprietăți senzoriale au fost obținute în probele cu SDS adăugat la un raport de flotă de 1/20 al MWCNT:SDS pentru 0,3 MWCNT și 0,5 MWCNT; pe de altă parte, raportul de flotă de 1/26 al MWCNT:SDS pentru 0,7 MWCNT.

Cuvinte-cheie: textile inteligente, senzori purtabili, MWCNT, SDS, electrofilare

INTRODUCTION

With the evolution of wearable electronics technology, the anticipated characteristics of textile products have transformed; in the contemporary context, textiles are expected to not only offer warmth and comfort but also to provide a sense of ease and additional functionalities, such as the detection of human movement, information processing, and energy storage [1, 2]. Textile materials present advantages over other materials, such as hardness and strength, but they can also be easily integrated into a wide range of end-use requirements and have softness and flexibility that allow for different uses [3, 4]. Products with electrical conductivity, called smart or electronic textiles, can be obtained by combining textile structures and electronic elements. E-textiles are an outstanding technology capturing significant attention for their ability to incorporate attributes such as antennas, mobile memory elements and wearable sensors into established textile products [5]. With their ability to strain, stretch and be wearable, strain sensors are a remarkable sub-branch of the wearable electronics field and can be applied in the fields of health imaging, joint movement detection, robotics and wearable electronics thanks to these properties [6–9]. Interest in smart textile applications, such as stretchable batteries and wearable sensors increases day by day. The high precision, robustness, and expansive operational scope of strain sensors are important in meeting the requisites of real-world applications [10, 11]. However, commercially available strain sensors lack these characteristics. Carbon black, metallic

nanoparticles, carbon nanotubes, nanocables, and graphene stands can be used for fabricating strain sensors with the desired characteristics. Carbon nanotubes stand out among these materials for their ability to provide high conductivity even at very low concentration levels [12–14]. Using their electrical conductivity, high strength and good mechanical properties, carbon nanotubes are known for their high potential for use in the production of flexible strain sensors [9, 15].

During the literature review, many investigations on strain sensor production have been documented.

In the study conducted by Şanli, a structure is produced by placing the KNT/TPU mat with an electrospun structure between two PDMS mats. KNTs within KNT (-COOH functionalized)/TPU samples are prepared by utilizing three distinct dispersion methods: magnetic stirring, ultrasonic bathing, and sonication. By detailed microscopic examination of the samples, electromechanical properties, piezoresistive properties and the KNT/TPU strain sensor mechanism in the electrospun sandwich structure are examined [14].

In their study, He et al. obtained the MWCNT/TPU structure with high flexibility and good efficiency by the process of wet spinning. In this structure, TPU serves as the flexible matrix material while MWCNT provides the sensor properties. Then, an assessment encompassing structural, mechanical, electrical, and strain detection features of the material is conducted. The optimal conductivity value at 28 MPa and 565% elongation is achieved as 6.77 Siemens/cm [7].

In their study, Nankali et al. obtained PDMS/MWCNT-based strain sensors by utilising the vacuum filtration technique and investigating the electrical characteristics of the structure. The electrical characteristics of CNT samples at varying rates of concentration are examined. As revealed by the measurements, the electrical resistance is observed to be within the range of 12.5 KΩ to 22.8 MΩ. Piezoresistive films within the CNT concentration range of 1.4–2.9 mg/mm^2 exhibit pronounced resistance drops, establishing this interval as the filtration threshold region. According to SEM images, the nanocomposite layer thicknesses of the strain sensors in this region are observed to be between 790 nanometers and 1210 nanometers. Then, the percolation curve is determined by using the curve fitting method on the experimental data. The percolation threshold is detected to be 1.992 mg/mm^2. Finally, to determine the minimum gauge factor of the sensors in the percolation zone, a flexible strain sensor is selected at the upper limit of this zone and the piezoresistive properties of the sample obtained are examined [16].

In their study, Wang et al. prepare a fibre-shaped strain sensor using TPU MWCNT and the cost-effective wet-spun method. The prepared sensor achieves 320% strain in uniaxial (---sided) strain tests. In addition, high precision (22.2 gauge factor at 160% strain, 97.1 gauge factor at 160% – 320% strain) and fast response time (<200 ms) are achieved [8].

In their study, Kumar et al. prepare a robust, stretchable and high-sensitivity MWCNT-reinforced TPU nanocomposite for piezoresistive strain/detection. The distribution of MWCNT within the TPU matrix provides a low leaching threshold (0.1% by weight) and superior electrical conductivity. MWCNT/TPU nanocomposites have shown different sensitivity and strain ranges depending on MWCNT concentrations. For TPU nanocomposites loaded with MWCNT of 0.3%, 0.5% and 1% by weight, a near-linear piezoresistive response is achieved at 15%, 35% and 45% strain, as well as 22, 8.3 and 7.0 gauge factor values [17].

In their study, Wang et al. obtain nanofiber films using the electrospinning method with 10%, 12.5% and 15% MWCNT TPU. TPU functions as the matrix framework while MWCNT operates as the conductive framework. An analogous trend is observed wherein elevating the quantity of MWCNT results in a reduction of thermal weight loss within the fibre film. Electrical conductivity improves, and thermal and sensing properties suggest a change. When all performance values are taken into consideration, it is observed that MWCNT/TPU nanofiber film containing 12.5% MWCNT obtains the best values for human movement and pressure sensing [15].

In the study conducted by Şanli et al., the effect of electrospinning parameters on the morphological and electromechanical properties of MWCNT/TPU nanofibers is examined. They observe that electrospinning parameters as well as MWCNT concentrations influence the morphological, electromechanical and sensor properties of MWCNT/TPU nanofiber membranes. They suggest that when the collector speed decreases and the collector distance increases, particularly during extended electrospinning periods, the resulting fibre structures exhibit a higher degree of uniform distribution, and this phenomenon has a beneficial impact on conductivity and strain sensitivity [18].

MATERIAL AND METHODS

Materials and chemicals

MWCNTs with COOH Function with more than 92% purity and outer diameter of 8–15 nm were purchased from Nanografi Nano Technology. Thermoplastic polyurethane (TPU) (Ellastolan 1185A10) was supplied from Biesterfeld GmbH (Istanbul, Türkiye) in granular form, N, N-Dimethylformamide (DMF) and Tetrahydrofuran (THF) from Progen Kimya and Sodium Dodecyl Sulfate (SDS) from Merck.

Nanocomposite production by electrospinning method

In the sample preparation stage which is schematized in figure 1, it was first dried in the oven at 60°C for 1 h to blow the moisture off the TPU granules. Then, a TPU solution with a concentration of 10% by weight was prepared. When this solution was prepared, TPU granules weighing 1 g were dissolved in 10 ml of DMF: THF solvent system. During the

preparation of this solvent system, the mass proportion of DMF to THF was established as 2:3. The mixture underwent homogenization via magnetic stirring for 2 hours under ambient conditions. Secondly, a solution was created by introducing MWCNT in weights of 0.3%, 0.5%, and 0.7% into 30 ml of DMF, along with (MWCNT:SDS) weight ratios of 1/0, 1/20, 1/23, and 1/26 about the weight of MWCNTs. A sonication process of 90 minutes was applied to the solution, ensuring the harmonious dispersion of MWCNTs and SDS. The mixture of these two solutions in a 1:1 ratio was followed by a 30-minute sonication to attain a stable composite mixture. Then, 10 ml of this solution was drawn into a syringe and nanocomposites were produced via the electrospinning technique. In the electrospinning method in nanocomposite production, the voltage applied was 32 kV, the feed rate was 1.6 ml/h, the ambient temperature was 21±2°C, and the collector rotation speed was 280 rpm.
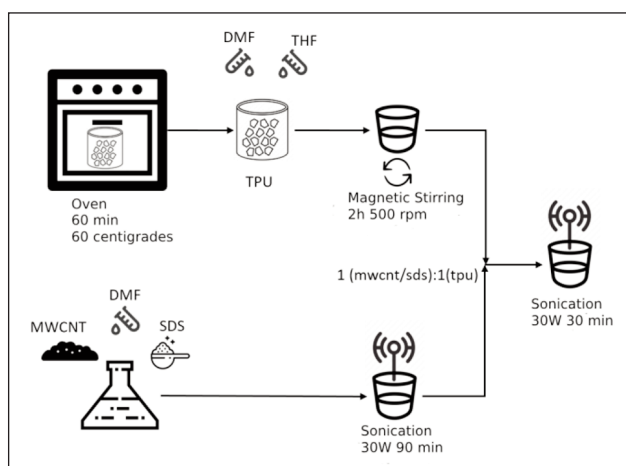

Fig. 1. Nanocomposite production

Characterization techniques

SEM analyses of nanocomposite structures derived from solutions, featuring distinct MWCNT and SDS ratios, were conducted using the Fei Quanta 450 model FEG SEM ESEM instrument. Viscosity measurements of the solution systems were performed by utilizing the Lamy Rheology RM 100 Plus Viscometer. The solution's conductivity was measured by using the WTW Profile Cond 3110 Conductivity Meter equipment. Alterations in resistance within the nanocomposite structures were measured employing the Keithley 2450 Sourcemeter and the Instron 4411 instruments. During measurements, nanocomposite surfaces measuring 3 cm × 1 cm were sectioned and evaluated using the Instron instrument. During this measurement, probes of the Keithley resistance measurement instrument were attached to the sample from the top and bottom, and resistance changes under voltage were measured for 30 minutes. Additionally, regarding the endurance of the sensor behaviour exhibited by these nanocomposite surfaces, resistance variations were identified through 100 repetitions under identical test conditions.

RESULTS AND DISCUSSION

The results of microscopic tests, electromechanical analyses of nanocomposite structures obtained from MWCNT at different concentrations and SDS used at different rates are shown and interpreted.

Morphological properties

The morphological properties of samples were examined by scanning electron microscope (SEM) and the results showed excessive bead formation and agglomeration on the samples' surface which were prepared with a 1/0 (MWCNT:SDS) (figure 2, *a, e, i*). Figure 2, *b, f, j* indicates that SDS addition is eliminated agglomeration. Also, bead formation is strongly inhibited in the presence of SDS (figure 2, *b, c, d, f, g, h, j, k, l*). Therefore, these results show that SDS addition improved the distribution uniformity of MWCNTs in TPU and prevented agglomeration and bead formation. According to resistivity measurements, it was found that there was no resistivity change in the samples with agglomeration (figure 2, *a, e, i*).
The SEM images of the samples with the best sensor characteristics shown in the measurements are given in figure 2.

Electromechanical properties

Electromechanical examination of the samples obtained by the addition of different MWCNT and varying rates of SDS is performed.
The resistance change is observed when the load is applied to the strain sensor. To make sense of this change, a gauge factor calculation is made. The gauge factor is calculated by the following formulas 1 and 2.

$$GF = \frac{\Delta R/R_0}{\varepsilon} \qquad (1)$$

$$\left(\frac{\Delta R}{R_0}\right) = \frac{R_t - R_0}{R_0} \qquad (2)$$

$\Delta R/R_0$ is calculated by the formula above. Here, ΔR represents the change in resistance, R_0 represents the sensor resistance when no load is applied, and R_t represents the resistance shown by the sensor under load. ε represents the amount of strain applied. Electromechanical examinations of the samples obtained by adding different MWCNTs and varying amounts of SDS were carried out (figure 3). Correlation and regression analyses of the values obtained in these examinations were performed and the statistical validity of the results was analysed.
Figure 3, *a* shows the elongation-gauge factor graph of the sample with 0, 1/20, 1/23 and 1/26 MWCNT/SDS ratios with a concentration of 0.3 MWCNT, The resistance changes under a tensile stress of the samples obtained by adding 0.3 MWCNT and varying amounts of SDS are given in figure 3, *a*. When figure 3, *a* is examined, the sample with 1/20 ratio of MWCNT:SDS showed up to 2% elongation, this change in resistance against elongation was evident that this nanocomposite can be applicable as a strain sensor. Therefore, it can be seen that the sample
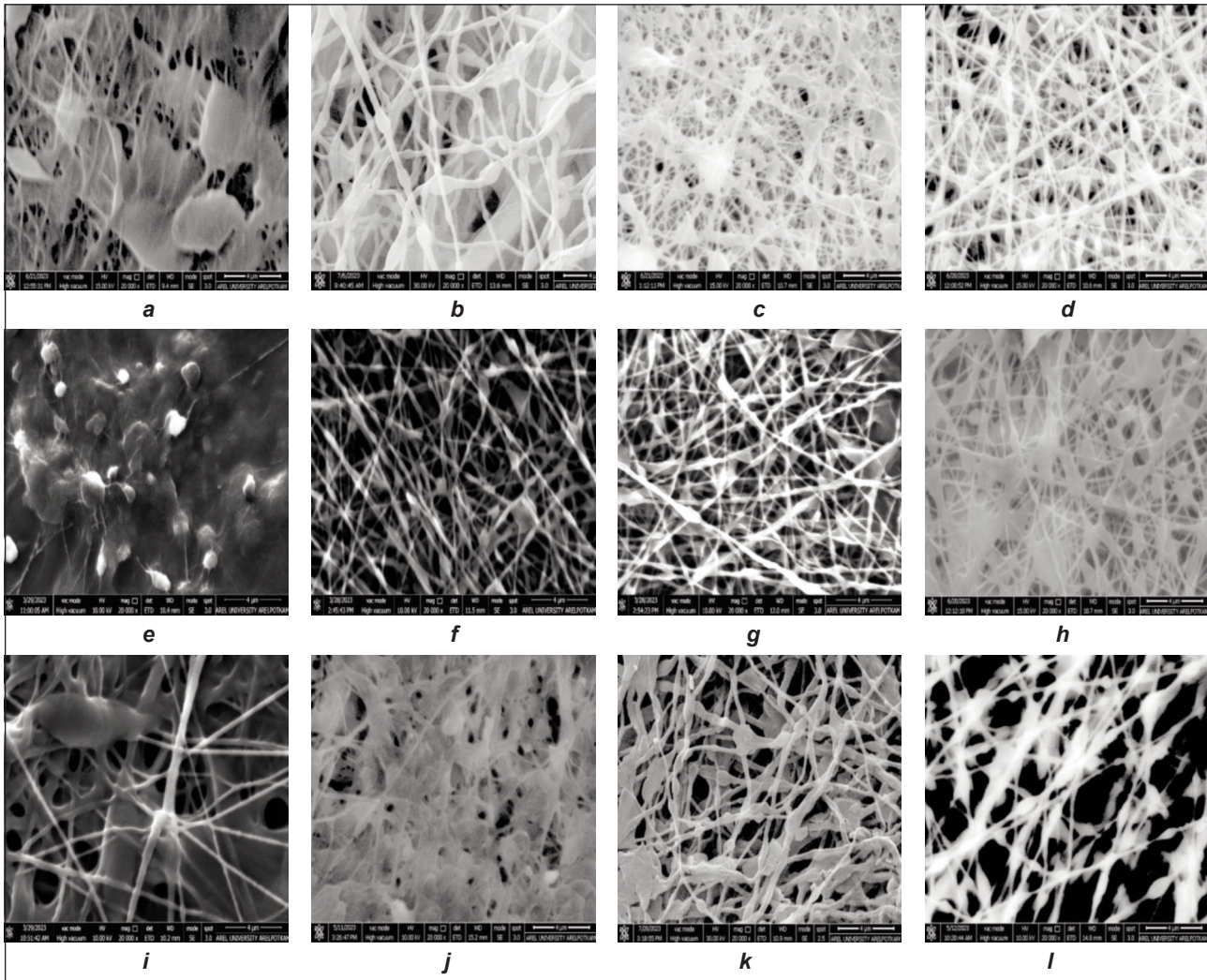
Fig. 2. SEM images: *a* – MWCNT 0.3 SDS 0; *b* – MWCNT 0.3 SDS 20; *c* – MWCNT 0.3 SDS 23; *d* – MWCNT 0.3 SDS 26; *e* – MWCNT 0.5 SDS 0; *f* – MWCNT 0.5 SDS 20; *g* – MWCNT 0.5 SDS 23; *h* – MWCNT 0.5 SDS 26; *i* – MWCNT 0.7 SDS 0; *j* – MWCNT 0.7 SDS 20; *k* – MWCNT 0.7 SDS 23; *l* – MWCNT 0.7 SDS 26
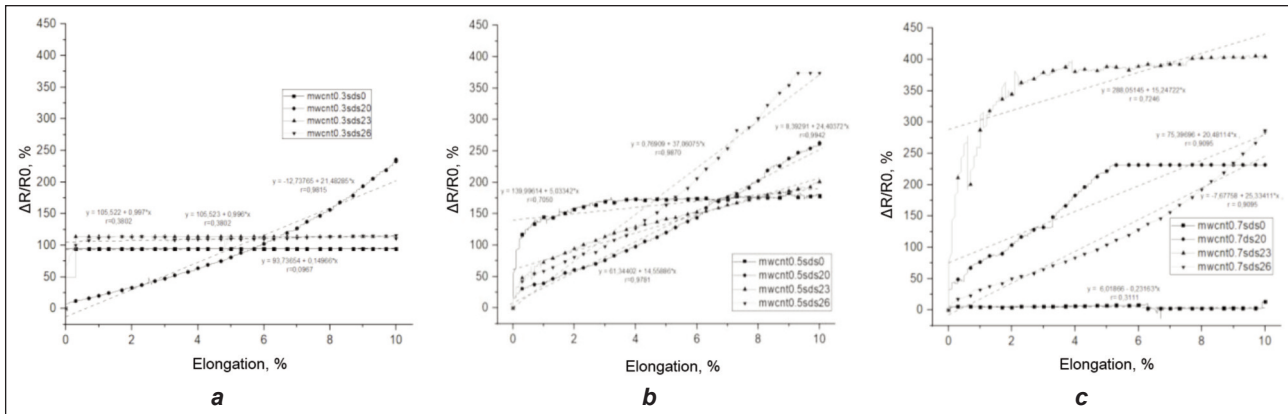

Fig. 3. Graphical representation of: *a* – ΔR/R0 change at MWCNT 0.3 constant value at 1/20, 1/23 and 1/26 MWCNT:SDS ratios; *b* – ΔR/R0 change at MWCNT 0.5 constant value at 1/20, 1/23 and 1/26 MWCNT:SDS ratios; *c* – ΔR/R0 change at MWCNT 0.7 constant value at 1/20, 1/23 and 1/26 MWCNT:SDS ratios

created by adding 1/20 SDS showed the best resistance change among the samples with 0.3 MWCNT added. It is considered that the increase in resistance of this sample largely depends on the increase of elongation, as can be seen from the correlation coefficient ($r = 0.98$), so it can be used as a tension sensor. It was statistically observed that the increase in resistance in response to the increase in stress did not occur in other samples. Therefore, these samples do not have sensor features.

The resistance changes under the tension test of the samples obtained by adding 0.5 MWCNT and varying
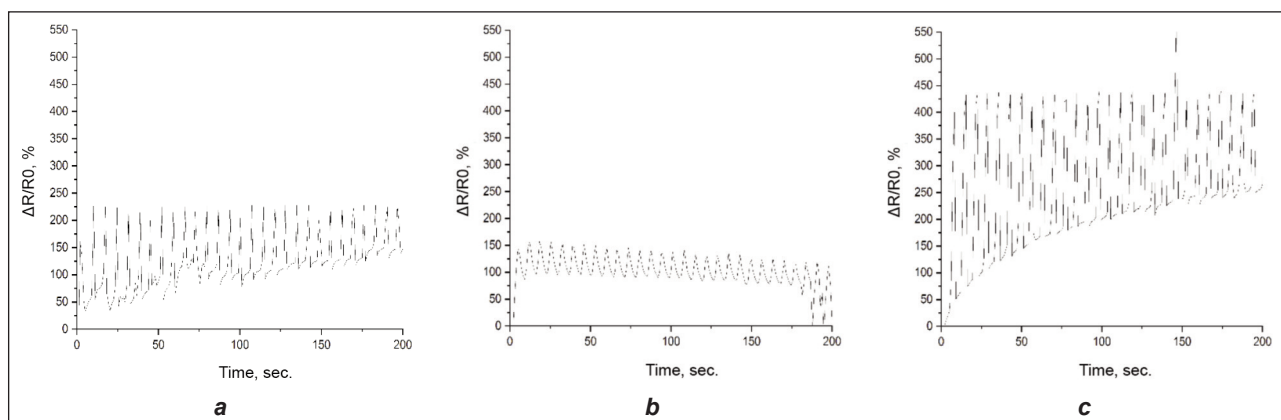
Fig. 4. Strain Cycle Tests performed to samples: *a* – MWCNT 0.3 and MWCNT:SDS 1/20; *b* – MWCNT 0.5 with MWCNT:SDS 1/2; *c* – MWCNT 0.7 with MWCNT:SDS 1/26

amounts of SDS are given in figure 3, *b*. When figure 3, *b* is examined, the sample obtained by adding 1/0 MWCNT:SDS shows a resistance change behaviour up to 2% elongation, but at an elongation value above 2%, it does not show sensor behaviour due to no resistance change. Sensor behaviour was observed in the samples obtained by adding MWCNT:SDS at the rate of 1/20, 1/23 and 1/26, the increase in resistance in the samples was due to the increase in elongation and this increase was statistically significant for all three samples ($r = 0.99$ for 1/20, $r = 0.98$ for 1/23, $r = 0.99$ for 1/26) was found to be significant.

The resistance changes under the tension test of the samples obtained by adding 0.7 MWCNT and varying amounts of SDS are given in figure 3, *c*. When figure 3, *c* is examined, it is seen that there is a change in resistance to extension stress in all of the samples, except for the sample obtained by adding 1/0 MWCNT:SDS. An increase in resistance was observed in response to a 5% increase in elongation in samples created by adding 1/20 and 1/23 MWCNT:SDS. Although sensor behaviour was observed up to 5% elongation value, no resistance change occurred at higher elongation values. Although the correlation coefficients for these samples were significant ($r = 0.91$ for 1/20, $r = 0.72$ for 1/23), no sensor behaviour was observed. As can be seen from the correlation coefficient ($r = 0.91$), the sample created by adding 1/26 MWCNT:SDS shows sensor properties as it is observed that there is a stable resistance change against the increase in elongation. To reveal the continuity of the sensor behaviour of the resulting nanocomposites, these samples are tested with 100 cycles for 400 seconds to understand their characteristics of resistance against strain. The resistance changes that occurred as a result of the applied cycle tests are observed and presented graphically. In these graphs, MWCNT 0.3 MWCNT:SDS 1/20, MWCNT 0.5 MWCNT:SDS 1/20, and MWCNT 0.7 MWCNT:SDS 1/26 examples that best express sensor behaviour are evaluated.

Figure 4, *a* shows the graph of the strain-cycle test performed at a concentration of 0.3 MWCNT, figure 4, *b* 0.5 MWCNT and a ratio of 1/20 SDS, and

figure 4, *c* 0.7 MWCNT and a ratio of 1/26 SDS as well. For all 3 measurements, it is determined that the resistance change continues at the end of each cycle. The fact that resistance values obtained in the cycle tests are same at the peak of the curves, is an evidence of a stable sensor behavior under strain for the samples. At the same time, it was determined that the resistance change decreased over time at the lower peak of the durational cycle test performed.

CONCLUSION

In this study, nanocomposites with different MWCNT concentrations and different SDS ratios were produced. Morphological and electromechanical examinations of these samples were performed and the following results were obtained.

Electrospinning could not be performed healthily for samples without SDS participation. Particularly, with the increase in the MWCNT ratio, MWCNTs could not be distributed homogeneously, causing blockages and disruptions in the electrospinning process. Furthermore, it has been detected that in response to the increase in MWCNT concentration, difficulties occur in the electrospinning process.

To avoid aggregation, the introduction of SDS additives was implemented and this enabled sample production. When the scanning electron microscope (SEM) images of the samples without any SDS addition are examined; it is observed that there is excessive pilling and agglomeration (clumps) on the obtained surfaces and it is observed that these clumps decrease as the SDS rate increases. Thus, it is understood that the SDS ratio ensures the uniformity of the distribution of MWCNTs within the TPU and prevents agglomeration. Accordingly, it is seen that there is no resistance change for the samples where agglomeration is present when the voltage is applied to the samples.

When the resistance changes of the samples to elongation are examined; best sensor properties were obtained for 0.3, 0.5 MWCNT at 1/20 MWCNT:SDS and for 0.7 MWCNT at 1/26 MWCNT:SDS ratios.

When the gauge factor behaviour indicating the sensor characteristic of the samples is examined, similar

results are obtained. It is understood that the samples prepared at MWCNT:SDS ratio of 1/20 for MWCNT 0.3 and MWCNT 0.5 concentration as well as MWCNT:SDS 1/26 for MWCNT 0.7 concentration presents the best sensor behavior. For other samples, it is evaluated that the gauge factor value varies in a narrow range and this change will adversely affect the sensor characteristics. In this content, the evaluation emphasizes the significance of the alteration in the linear direction between resistance values resulting from voltage increments, which present more significance for the manifestation of sensor behaviour. On the other hand, challenges encountered during sample production processes and the prevailing production conditions are believed to exert an influence on the sensor attributes exhibited by the samples.

Under the strain applied for the sustainability of the sensor characteristics, the following samples are found to display the best sensor characteristics in the resistance change loop test: the ones obtained with SDS 1/20 ratio at 0.3 concentration, with SDS 1/20 ratio at MWCNT 0.5 concentration and with SDS 1/26 ratio at MWCNT 0.7 concentration. Under an applied strain to investigate the sustainability of the sensor characteristics, the following samples are found to display the best sensor properties in the resistance change loop test: the ones obtained with 1/20 MWCNT:SDS ratio for MWCNT 0.3 and MWCNT 0.5 as well as 1/26 MWCNT:SDS ratio for MWCNT 0.7. It is seen that the divergence in resistance values among the chosen samples exhibits a partial decline as a response to the increase in time during the cycle test. Thus, it is thought that the MWCNTs in the TPU cannot be distributed completely homogeneously and this affects the resistance values of the samples. As a result, it has been demonstrated that samples with voltage sensor characteristics are produced in the study. Among these samples, samples with very good voltage sensor characteristics as well as samples with little or no voltage sensor characteristics are obtained. Here, it is evaluated that the MWCNT ratio and SDS ratio play a critical role together and electrospinning parameters are also effective.

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Authors:

RUSEN INAN[1], ISMAIL USTA[2], YESIM MUGE SAHIN[3,4]

[1]Istanbul Arel University, Vocational School, Fashion Design,
34537, Istanbul, Türkiye

[2]Marmara University, Faculty of Technology, Department of Textile Engineering,
34854, Istanbul, Türkiye
e-mail: iusta@marmara.edu.tr

[3]Istanbul Arel University, Faculty of Engineering and Architecture, Department of Biomedical Engineering,
34537, Istanbul, Türkiye
e-mail: ymugesahin@arel.edu.tr

[4]Polymer Technologies and Composite Application and Research Center (ArelPOTKAM)
Istanbul Arel University, Istanbul, Türkiye

Corresponding author:

RUSEN INAN

e-mail: rusenparlak@arel.edu.tr