Electrical Resistance Measurement Methods and Electrical Characterization of Poly(3,4-ethylenedioxythiophene)-Coated Conductive Fibers

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ABSTRACT: Textile fibers and yarns of high conductivity, and their integration into wearable textiles for different electronic applications, have become an important research field for many research groups throughout the world. We have produced novel electrically conductive textile yarns by vapor-phase polymerization (VPP) of a conjugated polymer, poly(3,4-ethylenedioxythiophene) (PEDOT), on the surface of commercially available textile yarns (viscose). In this article, we have presented a novel setup for electrical resistance measurements, which can be used not only for fibrous structures but also for woven structures of specific dimensions. We have reported a two-point resistance-measuring method using an already manufactured setup and also a comparison with the conventionally used method (so-called crocodile clip method). We found that the electrical properties of PEDOT-coated viscose fibers strongly depend on the concentration of oxidant ($FeCl_3$) and the doping (oxidation) process of PEDOT. To evaluate the results, we used mass specific resistance values of PEDOT-coated viscose yarns instead of normal surface resistance values. The voltage–current (*V–I*) characteristics support the ohmic behavior of coated fibers to some extent. Monitoring of the charging effect of the flow of current through conductive fibers for prolonged periods of time showed that conductivity remains constant. The change in electrical resistance values with increase in the length of coated fibers was also reported. The resistance-measuring setup employed could also be used for continuous measurement of resistance in the production of conductive fibers, as well as for four-point resistance measurement. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 2954–2961, 2012

Key words: electrical resistance measurement; conductive yarn; PEDOT coating; electrical characterization; two-point resistance measurement

INTRODUCTION

Electrically conductive fibers will be a key component of the smart and interactive textiles that will be used in the future, and they feature widely as power and signal transmitters in many prospective applications such as strain sensors,[1] ECG measurement,[2] sports and military garments, motion capture devices,[3] electrotherapy,[4] pressure sensors,[5] and photovoltaic devices.[6] During the last decade, many research groups have been trying to develop highly conductive fibers without incorporating any metal. For this purpose, intrinsic conductive polymers (ICPs) are a good choice because of their high conductivity, their light weight, and their possible use in developing light-emitting diodes (LEDs), organic transistors, coating for fuel cells, corrosion protection, functional textiles, organic electrodes, and biosensors.[7–14] Polymer-based electrically conductive fibers could be produced either directly from conjugated polymers by melt spinning, wet spinning,[15,16] or by coating conventional nonconducting materials with ICPs. To obtain improved electrical properties, we have to compromise on the mechanical properties of conductive fibers. A very interesting approach to obtain highly conductive textile fibers and yarns would be to coat them with conjugated polymers using a vapor-phase polymerization (VPP) process, which has already been developed.[17,18] Thus, textile fibers coated with conjugated polymers should give conductive fibers, which will retain their mechanical properties and at the same time exhibiting good electrical properties. Conductive textile fibers based on viscose, nylon, or PET should have optimal properties for use in yarns and fabrics. They are also available in large quantities at very low prices.

Among the large number of conjugated polymers, PEDOT has received considerable attention by scientists because of its good environmental stability and its application in different fields such as EMI shielding,[19] heat generation,[20] LEDs,[21] and chemical sensors.[22]

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The electrical resistance of conductive fibers and small-diameter wires can be measured by using two-probe and four-probe methods. Usually, the four-probe method is used to minimize the contact resistance, if fibers are highly conductive. There have been very few publications regarding electrical resistance measurements on fiber structure. However, few research groups have developed specific electrical resistance measurement devices for use with woven and nonwoven textile fibers under specified climatic conditions.[23–31] For the first time, Berberi et al.[32] reported the measurement of resistivity of fibers during the spinning process.

In this article, we have described electrical resistance measurements on and electrical characterization of conductive textile yarns, which we produced in our previous work by VPP of poly(3,4-ethylenedioxythiophene) (PEDOT) on the surface of oxidant ($FeCl_3$)-enriched viscose yarn fibers. We have devised a setup for electrical resistance measurements, using the Keithly 6000 picoammeter. We measured resistance values for PEDOT-coated yarn fibers by the two-probe resistance measurement method, both with a novel already manufactured setup and with the conventional method (crocodile clips method), and we then compared the results of the two methods. Other electrical characterizations were also done using the same setup. The effects of oxidant concentration, doping process, length of fibers, and continuous flow of current through the fibers on the electrical properties of PEDOT-coated viscose fibers were also determined.

The proposed setup could also be used for continuous evaluation of the electrical properties of conductive fibers in a continuous production line, possibly without affecting the surface morphology of coated fibers.

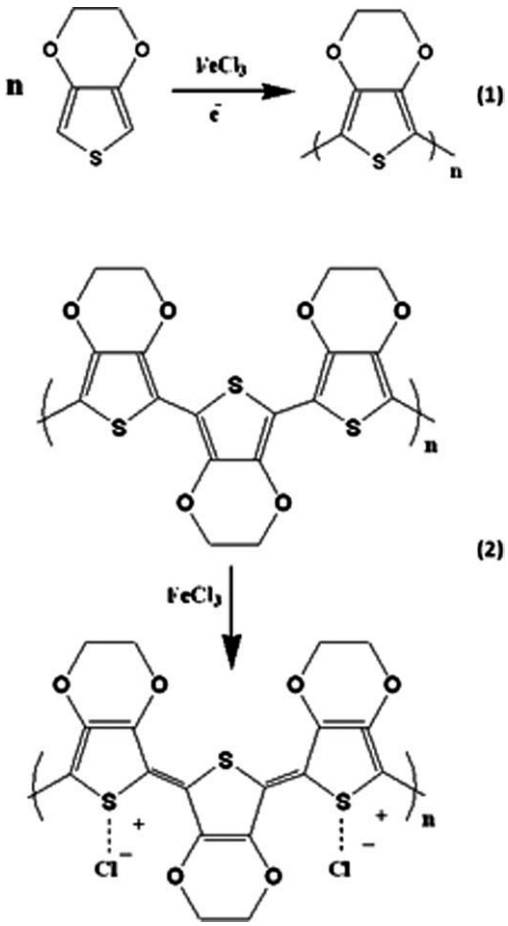

Figure 1 Mechanisms for vapor-phase polymerization (VPP) of PEDOT: (1) oxidation of EDOT monomer to PEDOT and (2) doping (oxidation) mechanism of PEDOT.

EXPERIMENTAL

Materials and sample preparation

Sample preparation of conductive yarns

We used viscose yarn fibers (1220 dtex, number of filaments 720, Z100 twist/m) purchased from CORDENKA® (Obernburg, Germany), 3,4-ethylenedioxythiophene (EDOT, CLEVIOUS M V2, H.C. Starck, Germany), monomer (CLEVIOUS® M V2), ferric (III) chloride ($FeCl_3$) (Sigma-Aldrich, Germany, 98%), and C_4H_9OH (Aldrich, 99%) for sample preparation of conductive yarns. All of these materials were used without any further modification.

We have already explained in detail the VPP of PEDOT on the surface of viscose yarn fibers, and this has been accepted for publication elsewhere.[33] Briefly, we prepared solutions of oxidant ($FeCl_3$) in butanol at various concentrations (3–15 wt %). The viscose yarn fibers (cut to 150-mm length) were pretreated with oxidant solution by dipping at room temperature for 5 min to 24 h, and then they were cooled at ambient conditions (23°C ± 2°C and 12% ± 5% RH) for 5–45 min. The oxidant-enriched viscose fibers were then inserted in a tubular reactor and flushed with EDOT monomer vapor and nitrogen gas for 5–60 min. The inside temperature of the reactor was fixed at 50°C by circulating hot water from the outer jacket of the reactor.

The EDOT monomers were immediately polymerized to form a darkish-blue layer of PEDOT on the surface of the viscose fibers. After polymerization, the PEDOT-coated viscose fibers were treated again (for doping purposes) with ferric (III) chloride ($FeCl_3$) solution to introduce negative charge along the backbone of the polymer chains to increase the conductivity of PEDOT. The mechanisms of oxidation of EDOT monomer and PEDOT polymer are shown in eq. (1) and eq. (2) of Figure 1.

In this study, we used PEDOT-coated viscose fibers prepared with various oxidant concentrations (3–15 wt %), but other reaction conditions such as dipping time of the fibers in oxidant solutions

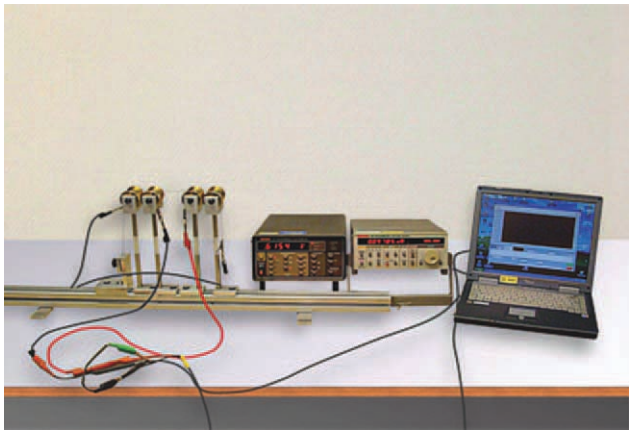

Figure 2 Electrical resistance-measuring setup used for electrical characterizations of PEDOT-coated viscose fibers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

(10 min), drying time (30 min), reaction time (15 min), and reaction temperature (50°C) were kept constant.

Resistance-measuring setup

The complete resistance-measuring setup is shown in Figure 2. Basically, it had two parts: (a) a Keithly picoammeter 6000 with a voltage source and a high-impedance voltmeter included; these two instruments were connected to a computer (right-hand side of the figure); and (b) a sample holder (at the left of the figure). We manufactured the sample holder in our workshop [see Fig. 3(A)]. It had four essentially identical units to support the test object, which could be a single thread or a piece of fabric. The wheels (1) were made of brass, with dimensions $R_1 = 30$ mm, $R_2 = 40$ mm, and $L = 50$ mm, as shown in Figure 3(B). The wheel holders (2) were made of aluminum, and the rods (3) that held them were made of steel. The intermediate rods (4) were made of Teflon to assure electrical insulation from the common holder below. The Teflon holder was attached to a steel rod that sat on an aluminum plate (5) that could be loosened and moved in the slot of the aluminum profile (6). The test object, of a certain specific length, was fastened at one end into a holder (7) made of aluminum, which sat on the aluminum profile (6) (also adjustable). The other end of the test object was pulled down by a weight, (8) for example 50 g, to ensure that the test object was stretched and also in contact with two or four wheels, as shown in Figure 3(A).

For resistance measurements of conductive fibers during production, the inner two wheels were placed 20–30 mm higher than the other two side-wheels. The sample holder was placed just before the final winding of fiber on the bobbin. With the movement of the object, the wheels would also rotate; thus, the surface morphology of the object would not be affected because of its friction with the surface of the wheels.

Resistance measurement methods

The resistance across the samples can be measured by using a manufactured setup with the two-point and four-point methods. Conventionally, the two-point method with crocodile clips is used, but if the resistance value is very small (in the ohm range or lower), then the four-point method gives more precise results by eliminating the effect of contact resistance.

A schematic diagram of the two-point method is shown in Figure 4, where U is the voltage source, V

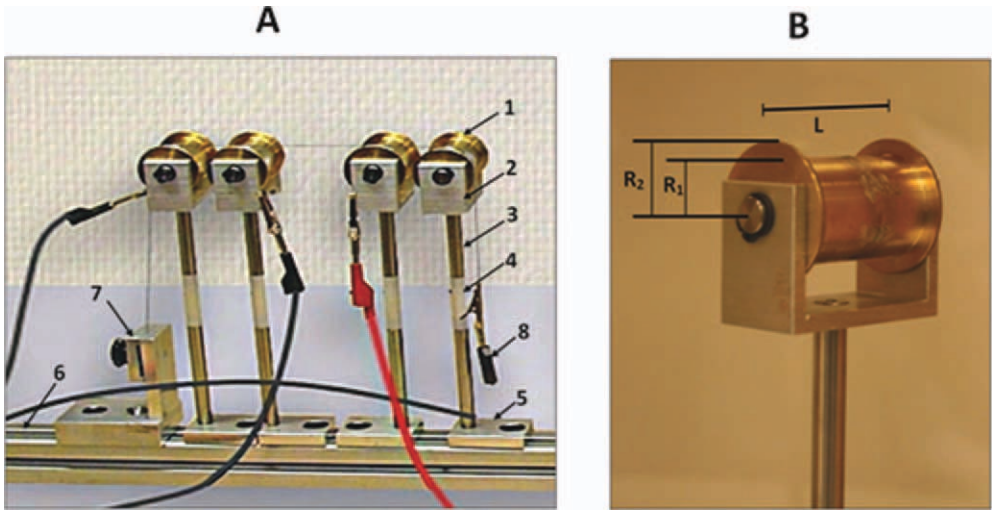

Figure 3 Constructed sample holding unit. (A) Different parts of setup and (B) dimensions of a single brass wheel. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

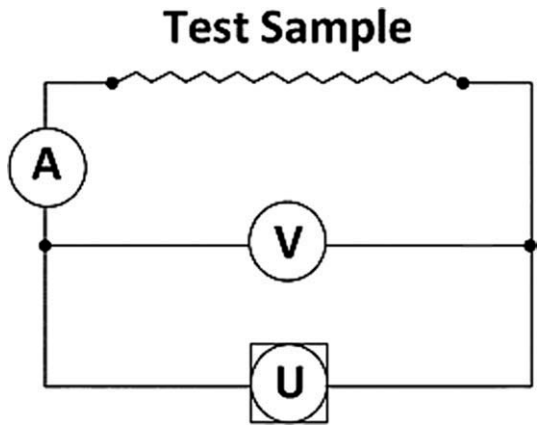

Figure 4 Principle coupling scheme of two-point resistance measurement setup.

is the voltmeter, and A is the current meter. The current that is applied across the sample is the correct current, but the measured voltage includes the voltage drop over the ampere meter, which limits the accuracy of measurement. Another scheme equivalent to the two-point method, which we have used for our setup, is shown in Figure 5. In this figure, R_c^1 and R_c^4 indicate the contact resistances between the outer two wheels and the sample, and R_m is the bulk resistance of the PEDOT-coated conductive yarn sample, which we want to measure. The Keithly 6000 picoammeter was connected to the outer two wheels with suitable connectors.

Figure 6 illustrates the principal coupling scheme for the four-point resistance-measuring method, and Figure 7 shows the corresponding model used in our setup. The bulk resistance of sample R_m is then

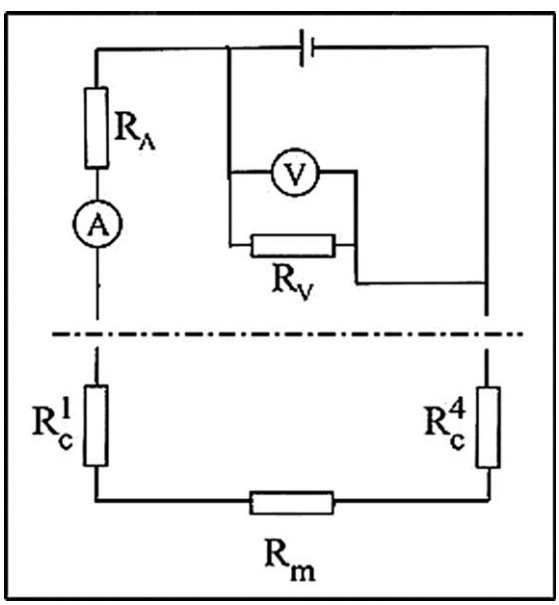

Figure 5 Two-point resistance-measuring model applied on constructed setup.

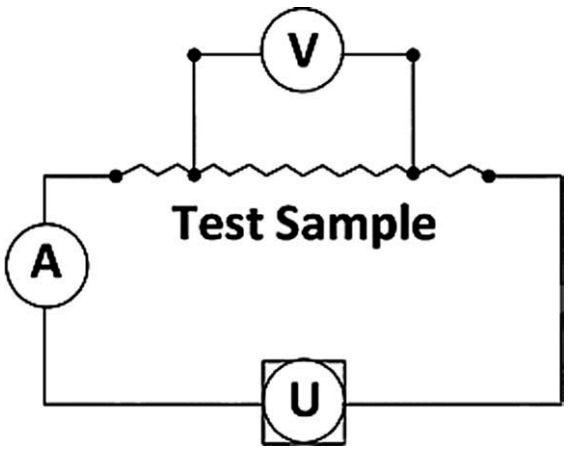

Figure 6 Principle coupling scheme for four-point resistance measurement setup.

divided into R_m^1, R_m^2, and R_m^3. For the four-point method, an external voltmeter is connected across the inner two wheels, and the outer two wheels are connected to the Keithly 6000 picoammeter as shown in Figure 7.

Although we are not using four-point resistance-measuring method yet due to limited length of conductive fibers produced, we will use it in our future work, and the description could help someone to use same setup for four-point measurements.

For standardization of manufactured setup, we used a conductive yarn of carbon fibers with known resistance value (885 Ω/m) and then measured the

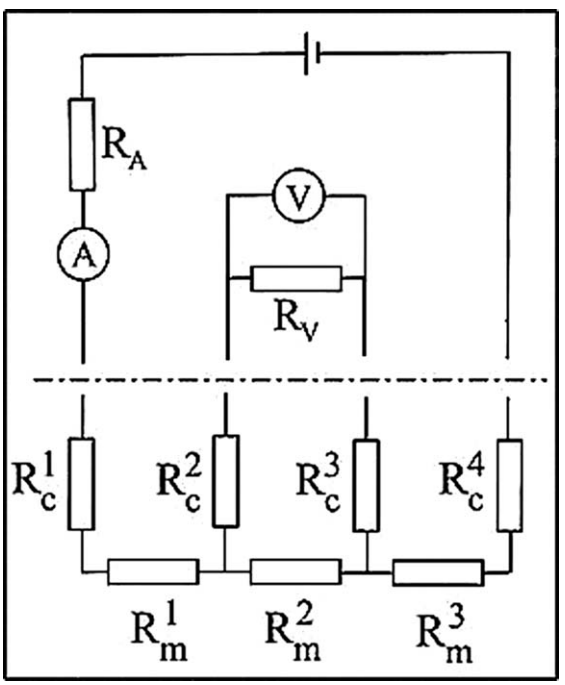

Figure 7 Four-point resistance-measuring model applied on constructed setup.

surface resistance values by crocodile clips and the manufactured setup. For both methods, same length of yarn was used. It was investigated that the surface resistance values measured by manufactured setup were $\sim$ 1.5 ($\pm$0.3) times higher than the resistance values measured with crocodile clips.

Electrical characterization

For electrical characterization, we cut PEDOT-coated viscose fibers to a length of 150 mm. These had been prepared at variable oxidant ($FeCl_3$) concentrations (3–15 wt %),[33] and we made all measurements under ambient conditions (23°C $\pm$ 2°C and 12% $\pm$ 5% RH). We measured electrical resistance values across all fibers by using both the manufactured setup and the two-point resistance-measuring method with crocodile clips, and then we compared the methods. Each fiber was tested 12 times, and then average values were used. We could not use the four-point resistance-measuring method because of the limited length of the fibers and somewhat higher resistance values, for which, two-point method gives acceptable results. We are working on continuous production of PEDOT-coated conductive fibers and will explain the four-point method in future articles.

For textile yarns and fibers, it has been reported in literature that mass specific resistance, R_s, is used more effectively instead of conventional electrical resistance.[34] The mass-specific resistance, R_s in Ω g/cm^2, can be represented by the following mathematical relation:

$$R_s = \rho d.$$

In this expression, ρ is the specific resistance of conductive fibers in Ω cm, and d is the density of material in g/cm^3. For textile fibers or yarns, mass-specific resistance, R_s, can be expressed in terms of fiber or yarn linear density. So, mass-specific resistance of a random specimen is given as:

$$R_s = RTN/L \times 10^5. \quad (1)$$

In this mathematical relation, R is the resistance in Ω, L is the length of specimen (cm), N represents the number of ends of yarn or fiber, and T is the linear density of yarn or fiber (g/1000 m).

We investigated the electrical properties of PEDOT-coated viscose fibers as a function of the oxidant concentration, the doping process (oxidation of PEDOT), and the length of fibers. The voltage–current (V–I) characteristics and the effect of charging on the flow of current through PEDOT-coated fibers for a prolonged time have also been reported in this article. We used our manufactured setup for all of these electrical characterizations.

RESULTS AND DISCUSSION

The electrical resistance values across the fibers were measured when the voltage varied between 1 and 10 V with a current of 2.5 mA. A comparison between the electrical resistance values of PEDOT-coated viscose fibers prepared at different concentrations of oxidant (3–9 wt %), measured both with the manufactured setup and with crocodile clips, is shown in Figure 8. In all graphs [(A), (B), and (C)], it is apparent that the electrical resistance values measured with the manufactured setup were higher than the values measured with crocodile clips.

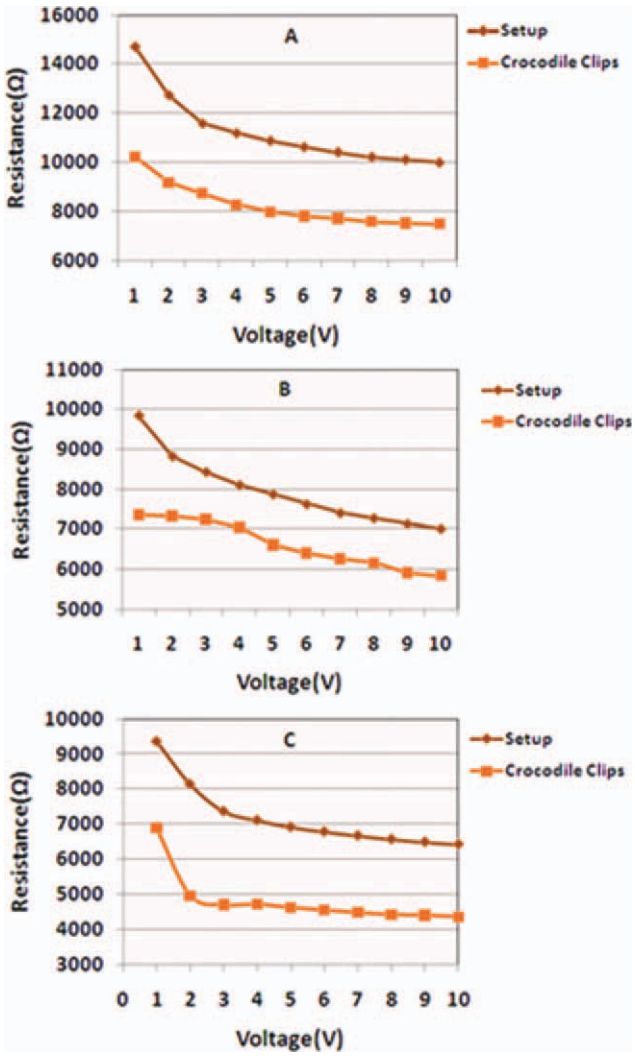

Figure 8 Comparison between resistance values obtained with conventionally used crocodile clips and constructed setup for different PEDOT-coated viscose fibers prepared with variable ($FeCl_3$) concentration (A) 3 wt %, (B) 5 wt %, and (C) 9 wt %. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

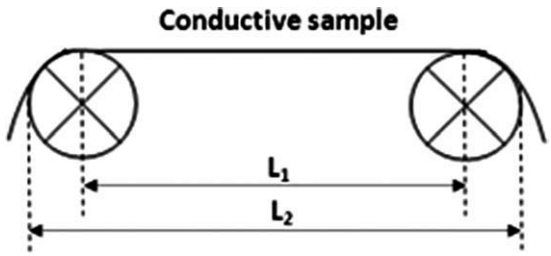

Figure 9 Schematic diagram of brass wheels holding conductive yarn sample.

As it has been mentioned earlier that the resistance values measured with manufactured setup are about 1.5 (±0.3) times higher than the values measured with crocodile clips, and Figure 8 shows that the difference in measured resistance values with both methods is of approximately same magnitude as noticed during standardization of the manufactured setup. It might be possible due to the much higher contact force and much smaller contact area of the crocodile clips as compared with the wheels of setup as mentioned in schematic diagram of brass wheels with conductive yarns and shown in Figure 9.

In Figure 9, L_1 is the length of conductive yarn that has been measured, and we used the same length for crocodile clip method. However, the actual length of conductive yarn having contact with brass wheels is L_2, which has higher contact area than the crocodile clips, and hence, manufactured setup shows higher resistance values as compared to crocodile clip method. Also, conductive yarns or fibers with higher lengths show higher resistance values, as mentioned in the next section.

The conventional method to measure conductivity of metal wires, here two-point method with crocodile clips, is straightforward and easy. However, when we use soft fibers/textiles, crocodile clips can cause problem, such as breaking of the fiber and uneven contacting. First, the developed method should be better for the sensitive fibers, as the contact is physical and not mechanical, and second, the distance between contact points is kept easily constant.

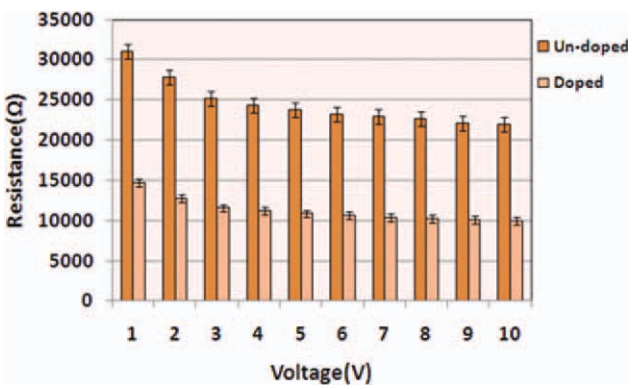

Figure 10 Effect of doping process on resistance values of PEDOT-coated viscose fibers prepared with 3 wt % oxidant concentration. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

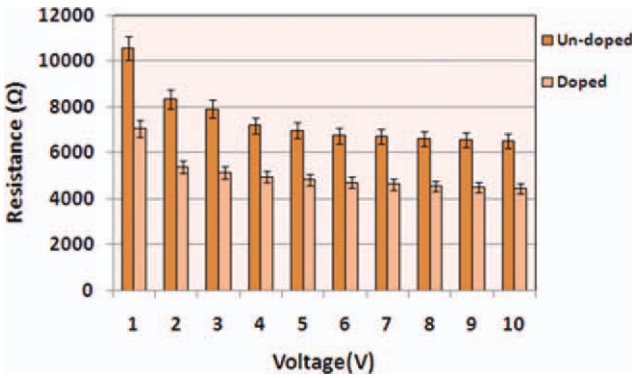

Figure 11 Effect of doping process on resistance values of PEDOT-coated viscose fibers prepared with 15 wt % oxidant concentration. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

PEDOT in undoped form has low or no conductivity,[35] as shown in Figure 1. The electrical properties of PEDOT-coated viscose fibers (prepared at 3 and 15 wt %) are illustrated in Figures 10 and 11 as a function of doping process. It is worth noting that without doping, PEDOT-coated viscose fibers showed higher resistance values. After doping with dopant ($FeCl_3$) solution, the conductivity of coated fibers increased, and hence their resistance values decreased. The difference between the resistance values, before doping and after doping, was higher for the fibers produced with 3-wt % oxidant solution than with 15-wt % solution. This may have been due to the higher concentration of oxidant, which was consumed in less quantity during the polymerization process, and excess amount acted as dopant, which has partially oxidized PEDOT. This is why PEDOT-coated viscose fibers are still very good conductors of electricity without any doping process. Further treatment with $FeCl_3$ solution completely oxidized PEDOT; hence, the conductivity increased. This enhancement in conductivity was greater in fibers with 3 wt % oxidant solution than with 15 wt % solution, because less amounts of undoped PEDOT chains were present in 15 wt % samples, which required further oxidation.

The variation in resistance values of PEDOT-coated viscose fibers prepared at different oxidant concentrations with increasing length of fibers (from 2 to 12 cm) is shown in Figure 12. The experiments were repeated 12 times for each fiber, and then average values were computed. The resistance value increased with increasing length of fibers. For all types of fibers, the trends were not exactly linear but

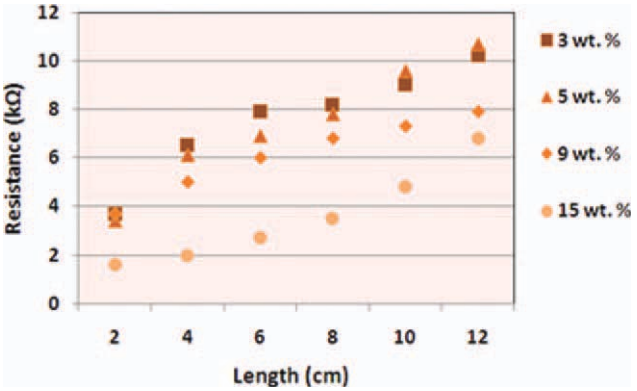

Figure 12 Variation in electrical resistance values with increasing length of PEDOT-coated viscose fibers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

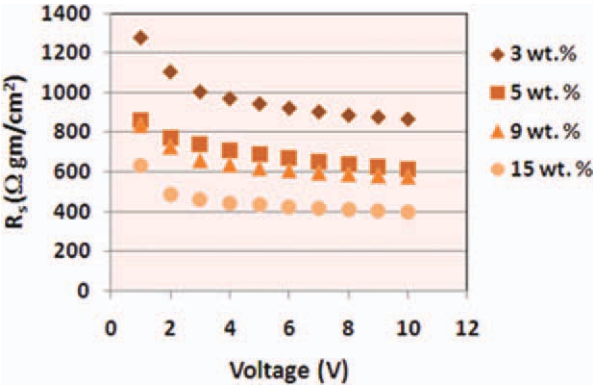

Figure 14 Mass-specific resistance values of different samples prepared with variable oxidant concentrations. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

approximated straight lines. This may have been because of the nonuniform distribution of the PEDOT layer across the whole fiber. The guide for the eye is include to indicate the measurement errors due to contact resistance for the samples prepared with 3, 5, and 9 wt % oxidant solutions. For 15 wt %, the upturn of the curve is most likely to originate from nonlinear effects of both fiber and contacts, since all resistance values are measured with the same voltage. The contacts between conjugated polymer chains are less important on yarns because of the circular form of the yarns.[36] However, the overall results obtained corresponded exactly to those predicted: that the resistance of the conductive fibers would be a function of their length.

The conductivity of PEDOT-coated fibers strongly depends on the oxidant ($FeCl_3$) concentration. The voltage versus current (*V–I*) characteristic curves for PEDOT-coated viscose fibers prepared at different oxidant concentrations are shown in Figure 13, and all the curves of *V-I* characteristics are linear. The conductivity of PEDOT-coated fibers at an oxidant concentration of 3 wt % is much less than the conductivity of fibers prepared with oxidant at 15 wt %. The current (*I*) values across all fibers were measured when the voltage varied from 1 to 10 V. We used fibers of uniform length (150 mm), and each fiber was tested 12 times and then average values were taken.

The dependence of electrical properties of PEDOT-coated viscose fibers on oxidant concentration is also indicated in Figure 14. For textile yarns or fibers, the mass-specific resistance (Ω g/cm^2) gives better results instead of normal surface resistance values. Mass-specific resistance values of all samples prepared with different oxidant concentrations were calculated according to eq. (1). In Figure 14, mass-specific resistance values of different samples prepared at different oxidant concentrations (3–15 wt %) are drawn against applied voltage (1 to 10 V). It is clear that with increasing oxidant concentration,

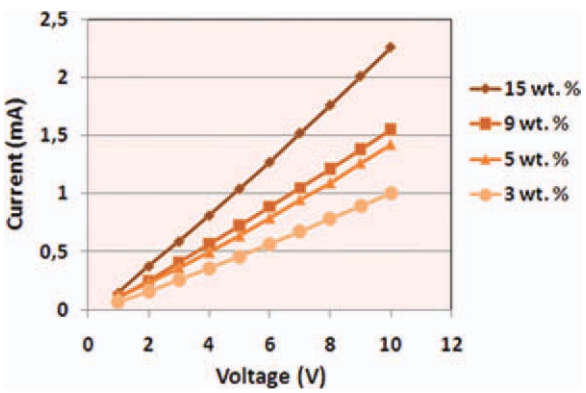

Figure 13 Voltage versus current (*V–I*) characteristics of different fibers prepared with variable oxidant concentrations. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

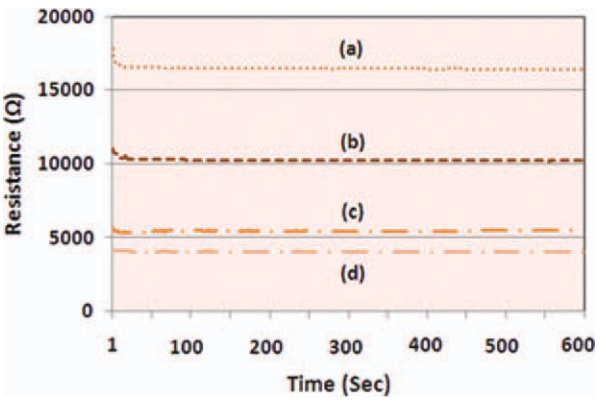

Figure 15 Effect of charging for longer period of time on resistance values of different fibers obtained with varying oxidant concentrations: (a) 3 wt %, (b) 5 wt %, (c) 9 wt %, and (d) 15 wt %. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the mass-specific resistance values decreases, and hence, the electrical properties increases.

The effect of charging on conductivity during current flow through PEDOT-coated viscose fibers is shown in Figure 15. For medical applications, three types of current—direct current, induction current, and alternating current—are used, ranging from 0.1 to 10 mA.[4] It is to be expected that if current flows through conductive fibers for long periods of time, they will heat up, and the resistance will increase and the conductivity will decrease. We selected a current of 20 mA and a voltage of 10 V, and experiments were performed for about 600 s on PEDOT-coated viscose fibers prepared at different oxidant ($FeCl_3$) concentrations. From Figure 15, it is clear that the resistance of all fiber samples remained constant throughout the 10-min period, so that there was no change in conductivity. This kind of property is required for medical applications such as electrotherapy, where current is applied across the sensors for prolonged periods of time. It could be possible that if we apply higher current value, i.e., 50 mA at same voltage value for longer period of time, then heat will produce, and resistance value will change.

CONCLUSIONS

Two-point resistance measurements both using crocodile clips and the manufactured setup showed that resistance measured with the manufactured setup is $\sim$ 1.5 times greater than the resistance measured with crocodile clips; thus, the latter could effectively be used for resistance measurements across single fibers. The surface properties of samples were also less affected by the manufactured setup compared with crocodile clips, which caused internal break-up of conjugated polymer chains and reduced conductivity of fibers. The manufactured setup could also be used for the four-point resistance measurement method. The resistance measurements on the fibers, before doping and after doping, revealed that the conductivity increased more on fibers that had been prepared with the lower oxidant concentrations. From results, we can thus also conclude that the conductivity of PEDOT-coated viscose fibers is a function of oxidant concentration and length of fibers. The voltage versus current (V–I) characteristics of different fibers showed that all fibers obey Ohm's law. Flow of current through fibers for longer periods of time revealed that there is no charging effect on the conductivity of fibers. These fibers can be used for different electronic applications where constant conductivity is required for a prolonged period of time.

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