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RESEARCH ARTICLE

High-strength electrically conductive fibers: Functionalization of polyamide, aramid, and polyester fibers with PEDOT polymer

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In this work, high-performance fibers such as aramid (Twaron), polyamide (PA6), polyester (PET), and hybrid Twaron/PA6 fibers were transformed into electroactive fibers by coating them with conjugated polymer, poly(3,4-ethylenedioxythiophene) (PEDOT) through vapor phase polymerization (VPP) method. The VPP is considered as an efficient technique for depositing CPs on different substrates regardless of their lower solubility in various solvents. In this paper, PEDOT-coated high-performance fibers were prepared under already optimized reaction conditions, and then a comparison between electrical, thermal, and mechanical properties of different fibers, before and after coating, was made. The obtained coated fibers were characterized through scanning electron microscope (SEM), thermogravimetric analysis (TGA), 2-probe electrical resistance measurement method, and tensile testing. It was revealed that at particular reaction conditions, all high performance textile substrates were successfully converted into electroactive fibers. The voltage-current (*V-I*) characteristics showed that PEDOT-coated polyester fibers exhibited highest conductivity value among all other substrate fibers. The active PEDOT layers on high performance fibers could behave as an antistatic coating to minimize the risks associated with static charges at work places. Also, the obtained fibers have potential to be used as smart materials for various medical, sports, and military applications.

KEYWORDS

high-performance fibers, high-strength conductive fibers, poly(3,4-ethylenedioxythiophene) (PEDOT), vapor phase polymerization (VPP)

1 | INTRODUCTION

Functional or smart textiles are considered the shape of future textile industry. Fibers with multifunctionalities (with better electrical, mechanical, and thermal properties) have already found a considerable place in various application areas such as signal and power transmitter in strain sensors,[1] ECG measurements,[2] motion capture devices for sports and military applications,[3] pressure sensors,[4] and photovoltaic devices.[5] Functionality such as electrical conductivity in conventional textile fibers can be introduced either by coating them with carbon black, metallic powder, and conductive polymers or mixing them with these active materials during the production process (melt spinning). However, lower mechanical properties, inferior electrical conductivity value, and higher production cost hinder their application areas.

Development of all organic conductive fibers (without incorporation of inorganic and metallic contents) with exceptional mechanical properties is a major interest of researchers throughout the world. They have advantages over other functional fibers such as flexibility, durability, cost-effectiveness, and ease in measurements.[6] The aforementioned objectives can be achieved by treating/coating the conventional textiles with conductive polymers as they have benefits of higher electrical conductivity, light-weight, and potential applications in organic transistor, light emitting diodes (LEDs), coatings for fuel cells and corrosion protection, antistatic coating, organic electrodes, and biosensors.[7-14] The textile fibers coated with conductive polymers such as poly(3,4-ethylenedioxythiophene) (PEDOT), polypyrrol (PPy), and polyaniline (PANI) not only retain their mechanical strength, flexibility, and comfortability but also exhibit high electrical conductivity values. Among other conductive polymers, PEDOT is considered a better option for functionalizing the textile substrates due to its good environmental stability and potential applications in the areas of heat generation,[15] EMI shielding,[16] LEDs,[17] and chemical sensors.[18]

High-performance fibers such aramid (Kevlar, Nomex, and Twaron), polyamide (6 and 6,6), and hybrid yarns of aramid/polyamide have exceptionally good mechanical properties and thermal stability. They are being widely used in aerospace and military applications, bulletproof fabrics, ballistic composites, fire-fighter suits, and automotive industry.[19] They are completely insulating materials and can build up static

charges, which could cause spark and dangerous explosion on working sites.[20] It is, therefore, of utmost important to use some finishing or antistatic agents on these materials. Production of conductive aramid and polyamide fibers by coating with carbon black, nickel-copper composite layer and supercritical carbon dioxide has already been reported.[21,22] However, stiff coating layers and lower comfortability issues limit their applications in apparel. Functionalization of these high-performance fibers with conductive polymers not only eliminates the static electricity issues but also gives smart fibers with superior mechanical, thermal, and electrical properties that could enhance their application areas.

However, implementation of conductive polymers on different substrates is quite difficult due to their aromatic rigid backbone structures, which makes them insoluble in organic and inorganic solvents. It has been reported that conductive polymers can be applied on polyamide and silk by in situ polymerization method but coated fibers exhibited lower conductivity values.[23,24] In order to get higher electrical properties and uniform thin layers of conductive polymers on different substrates, an efficient technique, called vapor phase polymerization (VPP), was utilized in this field. In VPP process, conductive polymers polymerize directly on the surface of substrate material in the presence of suitable oxidizing agent. A lot of efforts have already been made to enhance the conductivity values of CPs by utilizing different chemicals during the polymerization process.[25-27]

Previously, VPP method had been utilized to coat aramid, PET, PA6, lycra, and viscose fibers by PPy and PEDOT polymers, and coated-conductive fibers could have been used as humidity, temperature, and strain sensors.[15,28-31] Similarly, a comparison between PPy-coated viscose fibers prepared with solution phase and vapor phase process was made, and it was revealed that VPP method produced better results.[29] Other reports indicate that PEDOT coated textile fabrics can be used as flexible heat generating and EMI shielding textile elements.[16]

The current paper is the continuation of our previous research work in which functionalization of viscose and polyester yarns with PEDOT polymer through VPP process was studied.[30-32] It was revealed that both viscose and polyester were successfully coated with PEDOT and exhibited quite good conductivity values. However, viscose fibers lost mechanical properties significantly after PEDOT coating whereas polyester retained its virgin properties. This motivated us to select different high-performance fibers with excellent mechanical and thermal properties and see the possibility to transform them in electroactive fibers by utilizing the same VPP method. In this paper, high-performance fibers such as aramid, polyamide, polyester, and hybrid yarns of aramid/polyamide, having different yarn structure (twisted and untwisted), were selected. The PEDOT polymer was deposited on these substrate fibers by VPP process under same reaction conditions which were optimized in our previous work.[31] Finally, a comparison between electrical, mechanical, and thermal properties of all newly coated and previously coated fibers was made to investigate the quality of the fibers. The scanning electron microscopy (SEM) was used to study the surface morphology of coated fibers. Electrical properties were measured by using 2-probe method that was studied in our previous publication.[33] Thermal and mechanical properties were investigated by thermogravimetric analysis (TGA) and tensile testing, respectively.

TABLE 1 Specifications of substrates fibers used in experimentation

Fiber type	No. of filaments	Linear density (dtex)	Twist/m
Viscose	720	1220	Z100
PET (twisted)	210	1400	Z115
PET (untwisted)	210	2200	.....
Polyamide (PA6)	210	1400	S150
Aramid	210	1680	.....
Aramid/PA6 hybrid	210/210	3080	S81

By this comparison, it would be possible to investigate that PEDOT could also be applied on various high-performance textile substrates by VPP technique without affecting their virgin properties. The PEDOT-coated fibers could not only be used for smart textile applications but also have minimal static electricity problems in technical applications. In our future work, other characterizations including abrasion and bending resistance of coated fibers, aging properties, process cost, and continuous production of coated fiber by VPP process will be highlighted.

2 | EXPERIMENTAL

2.1 | Materials

In this study, polyester fibers (twisted and untwisted), aramid fibers (Twaron), polyamide 6 (PA6), and hybrid yarns aramid/PA6 purchased from Performance Fibers and viscose fibers provided by CORDENKA® were selected as substrate materials. The specifications of all substrate fibers are given in Table 1. In VPP process, 3,4-ethylenedioxythiophene (EDOT) monomer (CLEVIOUS® M V2) was polymerized on the surface of substrate fibers in the presence of ferric (III) chloride ($FeCl_3$) (Sigma-Aldrich, 98%) oxidant. The oxidant solution was prepared in 1-butanol (C_4H_9OH) (Aldrich, 99%) solvent with 15 wt.% concentration. All materials were used without any further modification.

2.2 | VPP polymerization of PEDOT

The detailed VPP process for depositing PEDOT on different textile substrates has already been explained in our previous publications.[30-32] Previously optimized reaction parameters at which better electrical properties were achieved, shown in Table 2, were selected for this work. The VPP of PEDOT on textile substrates is carried out in 5 different steps, shown in Figure 1 with specific numbers. At first, 150-mm-long substrate fibers were pretreated in $FeCl_3$ oxidant solution

TABLE 2 Optimized reaction conditions for better electrical conductivity of PEDOT

Parameters	Values
Length of fibers	150 mm
Oxidant concentration	15 wt.% $FeCl_3$
Dipping time	10 min
Drying time	30 min
Drying temperature	Room temp.
Monomer heating temp.	70°C
Reaction temp.	50°C
Polymerization time	15 min

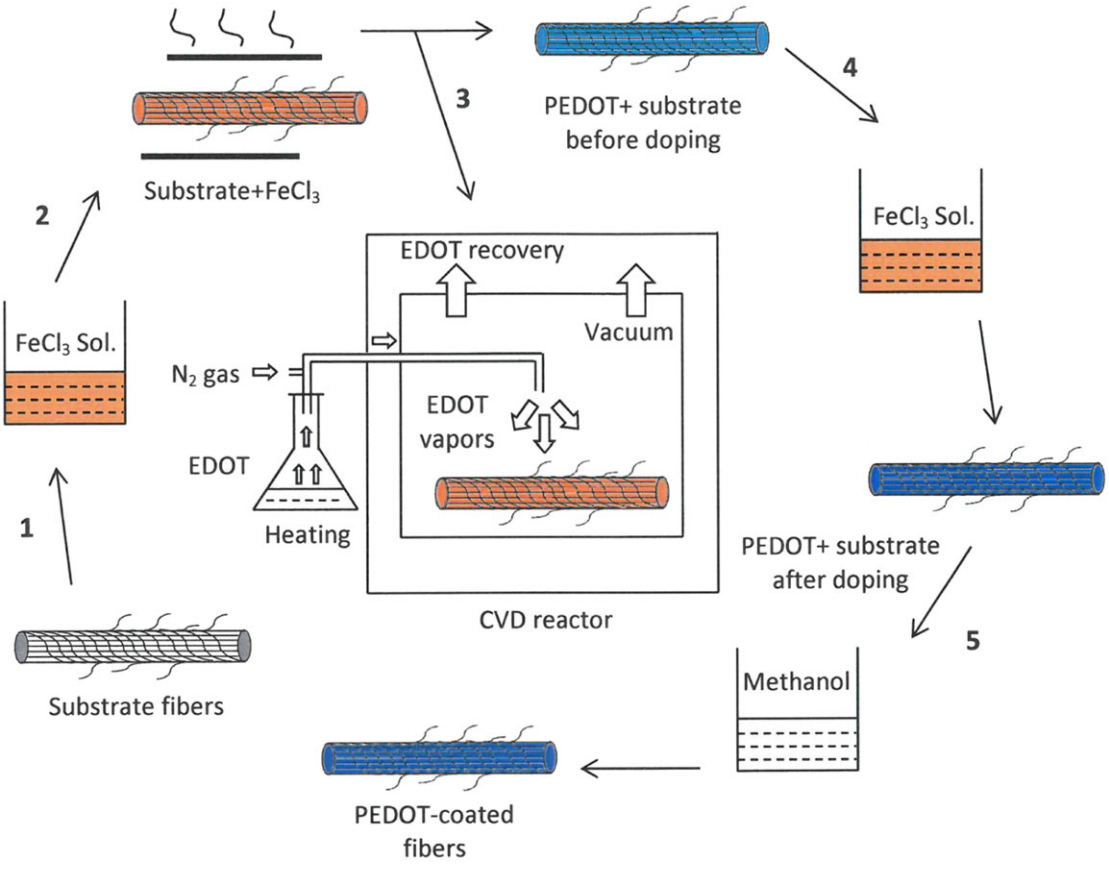

FIGURE 1 Schematic diagram for VPP of PEDOT on textile fibers [Colour figure can be viewed at wileyonlinelibrary.com]

for specific time (1), dried at room temperature (2), and exposed to the reaction chamber flushed with EDOT monomer vapors along with nitrogen gas (3). After some time, the polymerization reaction of EDOT to PEDOT was started on the surface of oxidant containing substrates and a thin, uniform dark-blue layer of PEDOT was formed which showed the successful deposition of PEDOT on substrate surface. In order to improve the electrical conductivity, PEDOT-coated fibers were doped with 3 wt.% $FeCl_3$ solution (4), and finally the coated fibers were washed with methanol (5) for removing by-products, residual oxidant and unreacted monomer and dried at room temperature.

The reaction conditions were kept constant for all types of substrate yarns.

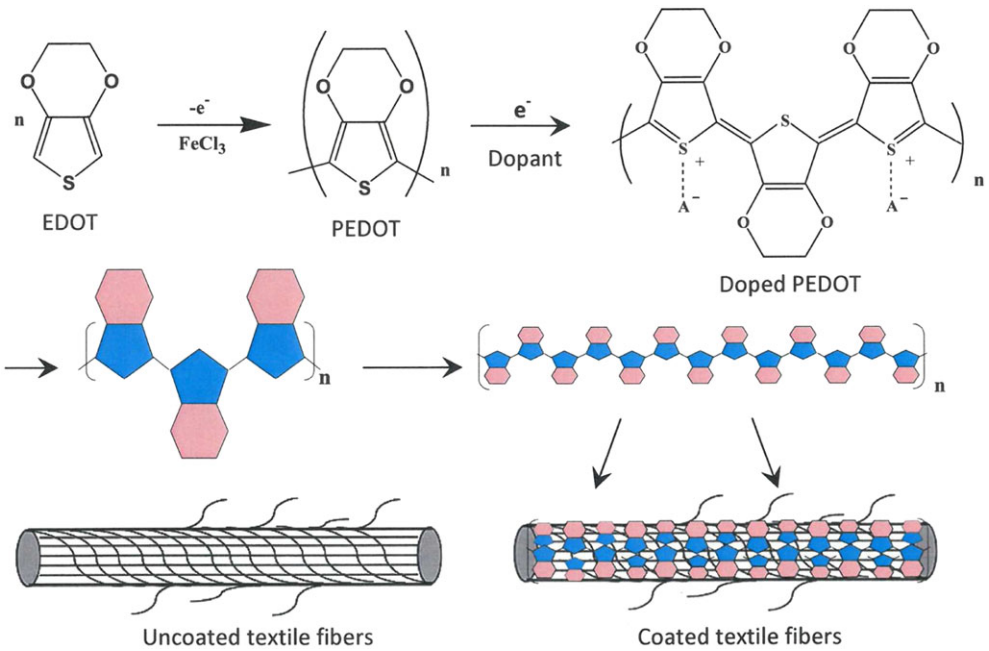

FIGURE 2 Polymerization and doping mechanism of PEDOT along with schematic of substrate fibers before and after PEDOT coating [Colour figure can be viewed at wileyonlinelibrary.com]

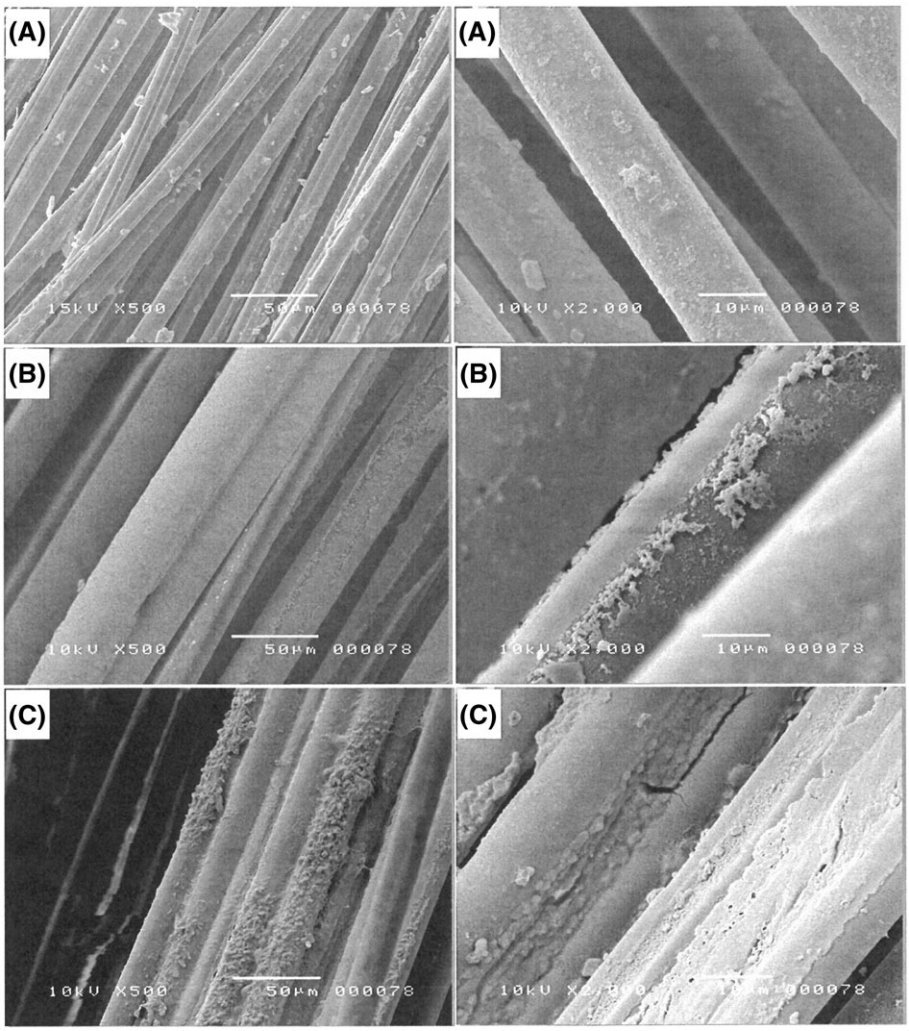

FIGURE 3 SEM images; *A* (viscose), *B* (PET-twisted), and *C* (PET-untwisted) at 500 and 2000 magnification

2.3 | Scanning electron microscopy (SEM)

The surface morphology of PEODT-coated fibers was investigated through scanning electron microscopy (SEM). For this purpose, experiments were performed on gold sputtered PEDOT-coated samples in JEOL JSM-840A scanning electron microscope at 10-kV accelerated voltage.

2.4 | Surface resistance measurement

Surface resistance of PEDOT-coated conductive fiber was measured using Keithley 6000 picoammeter and 2-probe method. A self-prepared fiber holding setup, studied in our previous publication,[33] was used for this purpose. Electrical resistance was measured at different voltage values between 1 and 10 V at room temperature (23 ± 2°C) and RH (12 ± 5%) along 150-mm-long fibers, holding between 2 crocodile clips. One fiber was tested 10 times, and then the mean values were used.

2.5 | Effect of charging on conductivity of coated fibers

The effect of charging on conductivity of PEDOT-coated fibers during the flow of current for longer period of time was determined by abovementioned Keithley 6000 picoammeter and in same environmental conditions. A 20-mA current was allowed to pass through the PEDOT-coated fibers (cut into 150-mm length) fixed between 2 crocodile clips for 400 seconds. Five fibers of each type were tested, and then average values were evaluated.

2.6 | Tensile testing

The tensile properties of PEDOT-coated fibers were investigated by using a Tinius Olsen 10-kN universal testing machine (manufactured by Tinius Olsen Testing Machine Company, UK) on 100-mm-long fibers under a crosshead speed of 20 mm/min at room temperature. We calculated maximum force at break and percent elongation. Mechanical testing was performed on at least 5 samples for each type of fibers, and then average values were reported.

2.7 | Thermogravimetric analysis (TGA)

The thermal stability of the PEDOT-coated fibers and the amount of PEDOT in coated samples were determined by TGA. Experiments were performed by using a TA Instrument Q500 TGA apparatus at a heating rate of 10°C/min from 25 to 600°C in nitrogen atmosphere with a purge rate of 20 mL/minute.

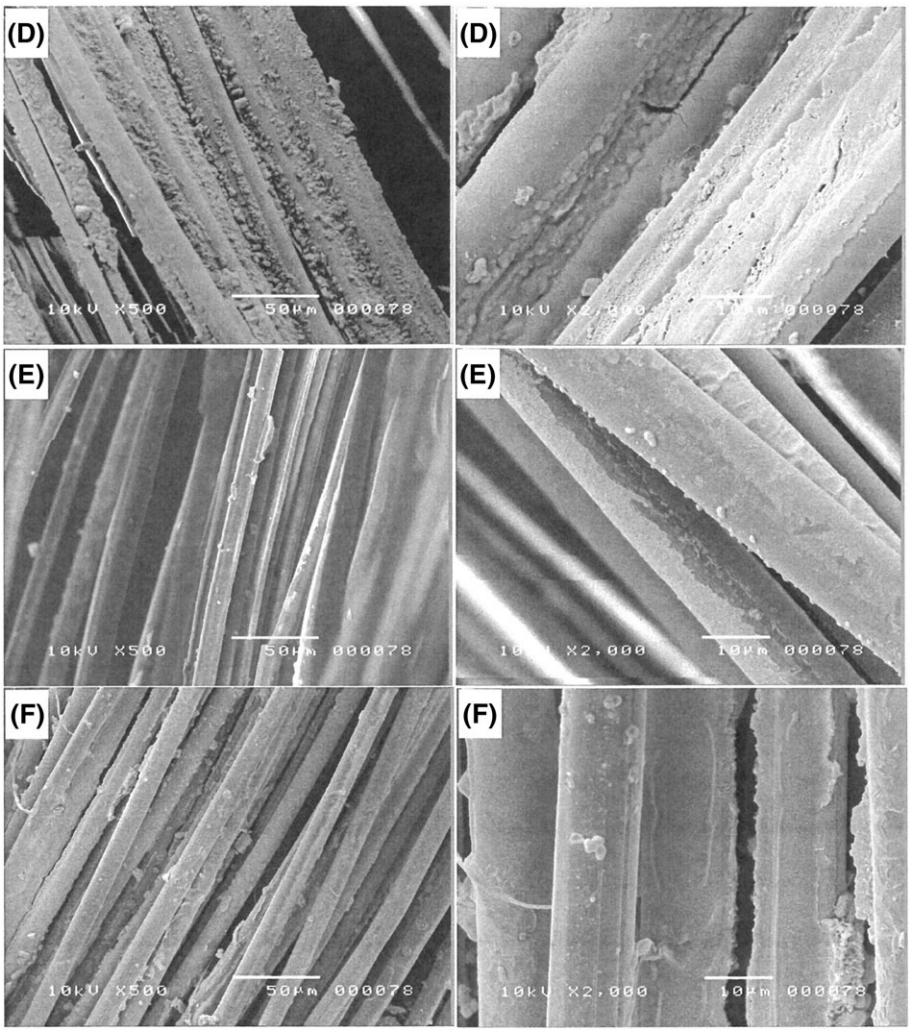

FIGURE 4 SEM images; *D* (aramid), *E* (polyamide), and *F* (aramid/polyamide) at 500 and 2000 magnification

3 | RESULTS AND DISCUSSION

3.1 | Polymerization of EDOT to PEDOT on substrate surface

Conductive polymers (CPs), also known as synthetic metals, can be produced with wide range of conductivities (10^{-8}-10^{5} S/cm) by various processing methods such as, in situ polymerization, electrochemical polymerization, wet spinning, and melt spinning. However, their processing is still a challenge due to inferior solubility in various solvents.

Among other processing techniques, direct deposition of PEDOT on the surface of substrates by VPP or chemical vapor deposition (CVD) is considered the most efficient one which minimizes the solubility issues of conductive polymers. It gives not only the higher conductivity values but also thin and uniformly distributed PEDOT coating throughout the substrate.

In our previous publications, it was investigated that various substrates such as viscose, polyester, and PTFE, in yarns and membrane forms, were successfully be functionalized by VPP of PEDOT.[30,31,34] It was revealed that different processing conditions can have diversified effects on the quality of PEDOT coating and electrical conductivity of obtained functional substrates. It was found that viscose and polyester fibers could be transformed into electrically conductive fibers at controlled reaction conditions, shown in Table 1.[31] In this research work, we focused on another high-performance textile fiber such as aramid, polyamide, polyester, and their hybrid structures to see the possibility of functionalizing them with PEDOT. The motivation behind this work is to minimize the static electricity problems associated with these high-performance fibers and to produce the high-strength electrically conductive fibers for various smart textile applications.

In VPP process, the obtained PEDOT-coated fibers exhibit lower or no electrical conductivity values. In order to increase the electrical conductivity of PEDOT, it was doped with $FeCl_3$ doping agent that introduces positive charges along the backbone structure of PEDOT which are balanced by the anions {A^- = $[FeCl_4]^-$ or Cl^-} provided by the doping agent. The polymerization mechanism of EDOT to PEDOT and un-doped PEDOT to doped-PEDOT is shown in Figure 2. The distribution of PEDOT on fiber surface and impregnation insight the fibers is shown in schematic diagram of Figure 2 and analyzed by SEM.

3.2 | Surface morphology of coated fibers

The distribution of PEDOT on substrate fibers was analyzed by SEM images. It was investigated earlier that quite thick PEDOT layer can be formed on viscose and polyester (untwisted) fibers with 15 wt.% oxidant solution.[31] The same concentration of oxidant solution was utilized for current high-performance fibers, and results were compared with previous findings as well. In Figures 3 and 4, SEM images of all coated fibers at 2 different magnifications, 500 and 2000, are shown. It can be seen in Figure 3 that viscose, polyester (twisted) and polyester (untwisted) have been coated with PEDOT. However, some parts of polyester (twisted) fibers are not coated completely. It is because of the relatively packed structure which hinders the penetration of $FeCl_3$ oxidant and, hence, responsible for less or no PEDOT coating. Similarly, in Figure 4, the SEM micrographs of PEDOT-coated aramid, PA6, and hybrid fibers of aramid/PA6 are shown. It can be observed that quite thick PEDOT layer was formed on aramid fibers as compared to PA6. In case of hybrid substrate, relatively less quantity of PEDOT was deposited, and it might be due to the twisting pattern of the fibers. The deposition and quality of PEDOT on these fibers can also be verified by electrical properties obtained. The coating thickness of PEDOT layers on substrate fibers was difficult to measure due to the multifilaments involved in each sample.

3.3 | Electrical resistance measurements

Electrical properties of PEDOT-coated fibers were determined by Keithley 6000 picoammeter. For this purpose, 2-probe method was used along with self-made fiber holding setup, which has been discussed in our previous work.[33] The electrical resistance was measured along 150-mm-long PEDOT-coated fibers when voltage was varied from 1 to 10 V and then plotted the voltage-current *(V-I)* curve to see their electrical behavior. It can be seen in Figure 5 that the maximum current density is observed for polyester (untwisted) fibers. It was worth noting that the same polyester with some twists showed lower current density which might be due to the packed structure and lower amount of PEDOT coating that has also been verified by SEM analysis. On the other hand side, aramid fibers showed lower current density than viscose which means that lower conductivity can be achieved with aramid fibers than viscose fibers. The lowest current density was observed for aramid/PA6 hybrid fibers which also fit the results obtained with SEM images.

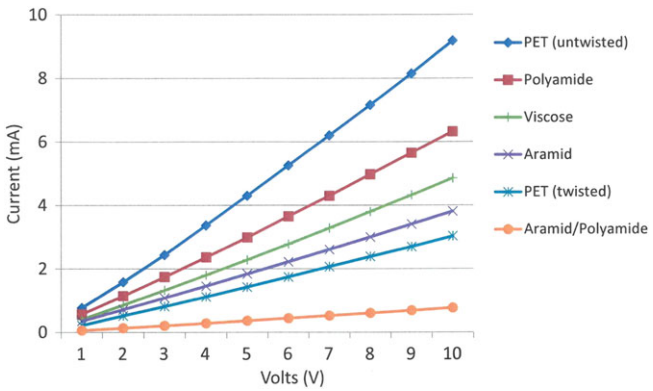

FIGURE 5 Voltage–current *(V-I)* characteristics of PEDOT-coated fibers [Colour figure can be viewed at wileyonlinelibrary.com]

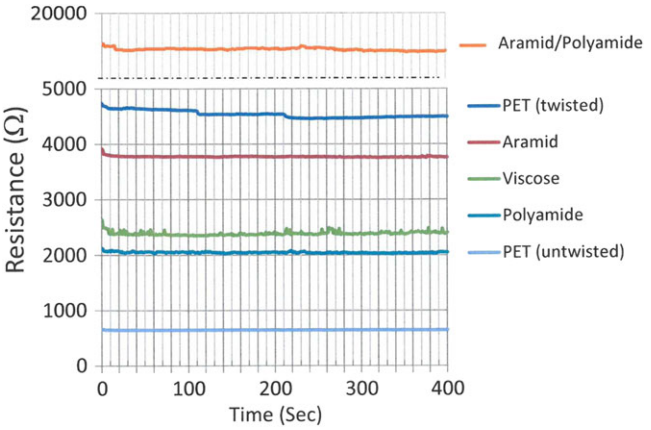

FIGURE 6 Effect of charging on electrical properties of PEDOT-coated fibers [Colour figure can be viewed at wileyonlinelibrary.com]

3.4 | Effect of long-term charging on conductivity

A constant applied voltage on conductive fibers for longer period of time might change their electrical properties due to overcharging, and heat might be generated. The time range characteristics of PEDOT-coated high-performance fibers were determined by applying a DC voltage of 10 V at relatively higher current value (20 mA) for 400 seconds along 150-mm-long fibers. At applied voltage values, the electrical resistance of all coated fibers is in the order of PET(untwisted) < PA6 < viscose < aramid < PET(twisted) < aramid/PA6 hybrid fibers. It is illustrated in Figure 6 that the resistance values of PEDOT-coated polyester (twisted) fibers are decreasing with the passage of time which shows minor charging effects. The other fibers showed almost straight lines which indicate no impact of charging on electrical resistance values. This is also the indication of no heat generation due to the constant flow of current for longer period of time.

3.5 | Mechanical testing

In our previous work, the mechanical characteristics of PEDOT-coated viscose and polyester (untwisted) fibers were determined. It was

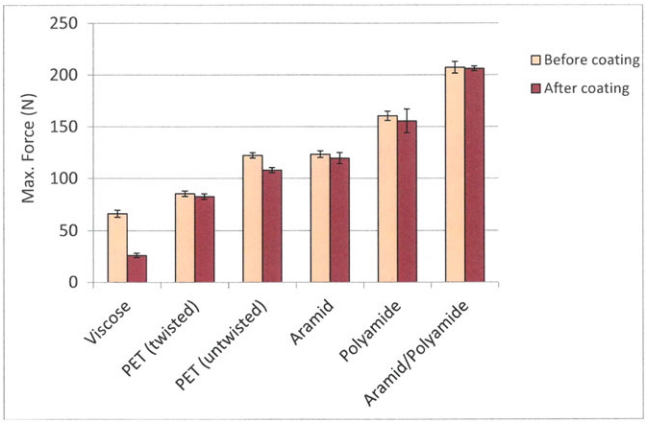

FIGURE 7 Strength of substrate fibers before and after PEDOT coating [Colour figure can be viewed at wileyonlinelibrary.com]

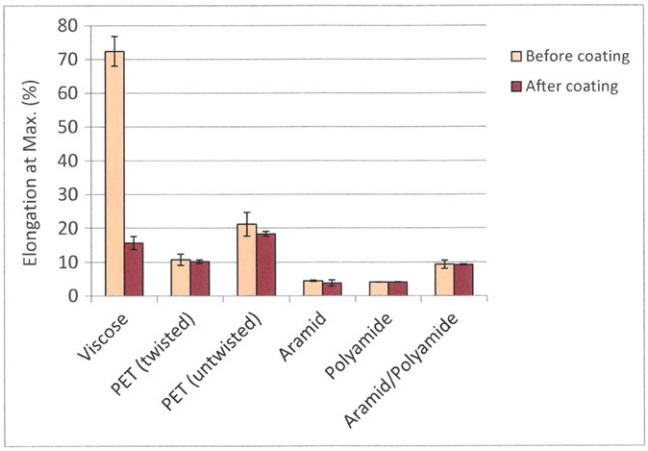

FIGURE 8 Max. percent elongation of fibers before and after PEDOT coating [Colour figure can be viewed at wileyonlinelibrary.com]

revealed that the strength and elongation % of viscose yarn were reduced tremendously after PEDOT coating due to the acid hydrolysis of viscose in acidic solution of $FeCl_3$. However, polyester showed better electrical and retained mechanical properties after treatment with PEDOT.[31] In Figures 7 and 8, strength at break and maximum elongation % of previously studied viscose and polyester fibers are compared with PEDOT-coated high performance fibers. It is observed that the strength of aramid, PA6, and polyester fibers were slightly reduced after PEDOT coating, but there is no variation in strength of aramid/PA6 hybrid substrate sample. However, maximum elongation % was remained consistent before and after PEDOT coating. Overall, the PEDOT-coated high performance fibers have retained mechanical properties due to their resistance against different chemicals.

3.6 | Thermogravimetric analysis

Polyamide and aramid fibers have extraordinary thermal stability in their parent form. The effect of PEDOT coating on their thermal properties was studied by TGA. In our previous studies, it was investigated that thermal properties of viscose fibers changed after PEDOT coating whereas, polyester (untwisted) fibers retained their thermal stabilities. Also, it was found that higher amount of PEDOT can be deposited on viscose fibers as compared to the polyester.[31]

The TGA thermographs of polyester (twisted), PA6, aramid, and hybrid yarns of aramid/PA6, before and after PEDOT coating along with pure PEDOT polymer, are shown in Figures 9 and 10. It can be seen that polyester (twisted) fibers have almost same thermal stability before and after coating as it was investigated earlier. It indicates a very thin layer or small quantity of PEDOT deposition of substrate fibers. Similarly, the decomposition temperature of PA6 was almost

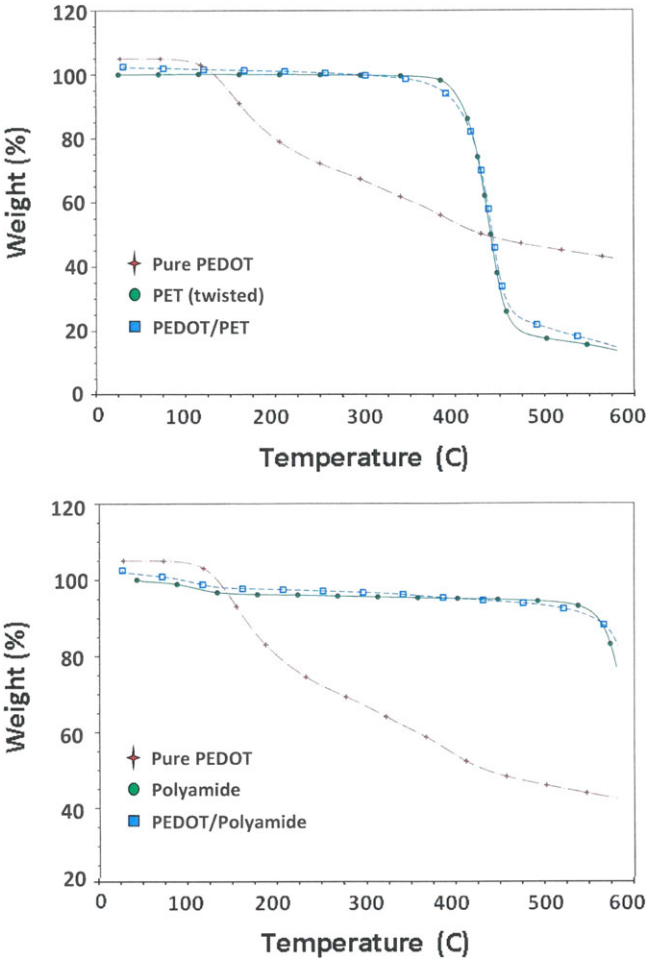

FIGURE 9 TGA thermographs of polyester, polyamide fibers before and after PEDO coating [Colour figure can be viewed at wileyonlinelibrary.com]

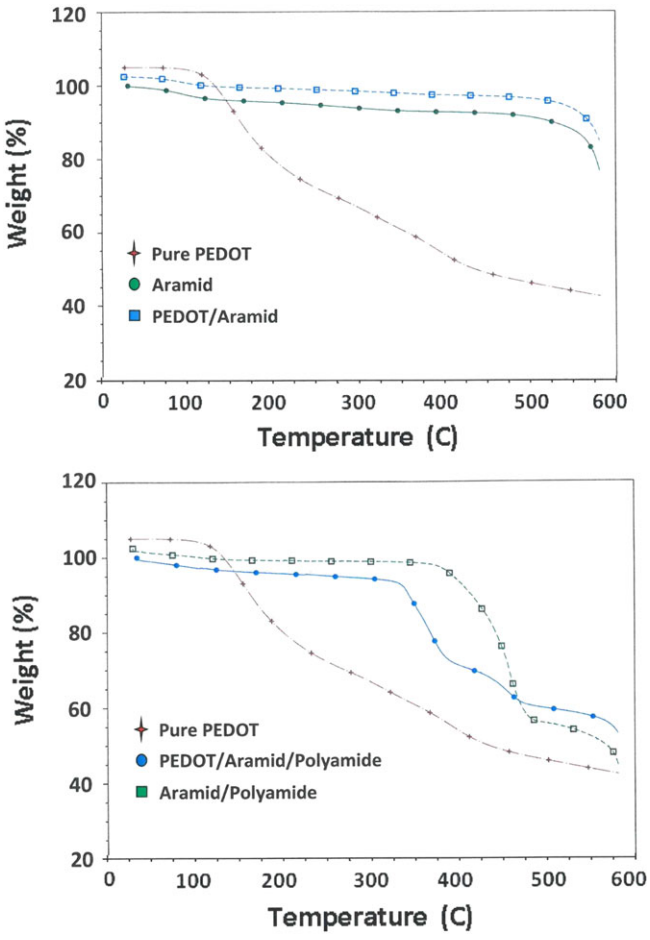

FIGURE 10 TGA thermographs of aramid and polyamide/aramid fibers before and after PEDO coating [Colour figure can be viewed at wileyonlinelibrary.com]

same before and after PEDOT coating. On the other hand side, TGA thermographs of aramid fibers showed different behavior. The PEDOT-coated aramid fibers have relatively better thermal stability as compared to the neat aramid fibers. It might be because of the protective coating of PEDOT polymer on substrate fibers. Conversely, the thermal properties of PA6/aramid hybrid fibers were different before and after PEDOT coating. The thermal decomposition of pure hybrid substrate sample was started at more than 400°C, whereas, after PEDOT coating, it was reduced to 350°C. There might be different reasons for this variation such as removal of spin-finishes from the surface of the substrate fibers while treating with $FeCl_3$ solution or absorption of larger quantity of oxidant due to the packed structure of hybrid yarn which might be degraded at lower temperature and, hence, reduces overall thermal stability of coated fibers. From these thermographs, it might be possible to get the quantitative estimation of PEDOT deposition on different substrate fibers.

4 | CONCLUSIONS

It was concluded that high-performance textile fibers such as, PA6, aramid, aramid/PA6 hybrid, and polyester fibers can successfully be functionalized by VPP of PEDOT. The deposition of dark-blue layer of PEDOT polymer on all substrate surfaces was the indication of successful coating and impregnation of PEDOT which was further analyzed with SEM images. The comparison between twisted and untwisted polyester yarns gave some interesting results, and it was revealed that the fiber structure might also have strong impact on distribution of PEDOT and conductivity values of coated fibers. The electrical resistance measurements and voltage-current *(V-I)* characteristics of PEDOT-coated fibers show that all fibers exhibit good electrical properties, out of which polyester (untwisted) fibers have best electrical conductivity values whereas, hybrid yarns of aramid/PA6 show minimum conductivity. The time range characteristics reveal that there is no considerable decrease in electrical resistance values, and, hence, no heat was generated with constant flow of current. The mechanical testing shows that all high-performance fibers almost retained their parent mechanical strength and % elongation even after PEDOT deposition, which was not the case for our previously studied viscose fibers. The TGA analysis indicates that PEDOT-coated polyester, PA6, and Twaron have almost same thermal stability before and after PEDOT coating. However, the thermal stability of hybrid fibers of aramid/PA6 was slightly reduced after PEDOT coating.

Based on the results obtained, it should be possible to functionalize the high-performance fibers by PEDOT coating and the obtained fibers could have acceptable electrical properties along with retained mechanical properties, which are the requirements to use them as sensors, actuators, and power transmitters in smart textile applications. Also, the active coating of PEDOT on high-performance fibers might reduce the problems associated with static electricity and, hence, increase their application areas.

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